Nanotechnology and the Most Important Characterization Techniques for Nanomaterial's: A Review

Ruqayah A. Ulwali[1*], Heba Kh. Abbas[1], Noor Yasoob[1], Hanan Abed Alwally[2]

Abstract

Due to the importance of nanotechnology because of its features and applications in various fields, it has become the focus of attention of the world and researchers. In this study, the concept of nanotechnology and nanomaterials was identified, the most important methods of preparing them, as well as the preparation techniques and the most important devices used in their characterization.

Key words: Manufacture, Nanometers, Micro-electronic Devices.

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Introduction

During the past few years, a new term has come to the limelight, and this term has become a major focus of the world's attention, which is nanotechnology. Nanotechnology or nanoproducts service is derived from the name of nanometer as a unit of measurement equal to one billionth of a meter or one part of a thousand millionth of a meter (Abass et al., 2020), Figure (1) represents the scale of objects in nano, micro, millimeter and meter (Leydecker, 2008). Nanotechnology is an application of various physical, chemical, biological, engineering, medical and pharmaceutical sciences; and harnessing them to design and manufacture tools and equipment at a scale not exceeding 100 nanometers through the assembly of basic components (atoms) of materials; since all materials are composed of atoms compacted in a specific order, replacing an atom of an element with atom of another element produces various other materials. Sometimes these materials appear with new properties that we did not know before, which leads to the opening of new areas for its use and harnessing it for the benefit of mankind (Singh et al., 2017, Gupta et al., 2016).

In nanotechnology, like other technologies, a number of special terms are used, including nanoscience, meaning that science that is concerned with the study and characterization of nanomaterials and specifies their chemical, physical and mechanical properties while studying the related phenomena arising from their miniaturization.

It goes without saying that reducing the sizes and scales of materials to the nanometer level is not an end in itself, but rather a sophisticated scientific philosophy and a qualitative and scientific reversal of the classics and constants of physical and chemical theories in order to produce a new class of materials known as nanomaterials to match their

Corresponding author: Ruqayah A. Ulwali
Address: [1]Physics Department, College of Science for Women, University of Baghdad, Baghdad, Iraq; [2]Department of Computer Science, College of Science, Mustansiriya University, Baghdad, Iraq.
[1*]E-mail: ruqayah.a@csw.uobaghdad.edu.iq

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distinctive properties with the requirements of advanced technology for the intended application purpose, based on this, the applications of nanotechnology are not limited to one branch of science, engineering or medicine, but its applications extend to include all branches and applications (Shah et al., 2015, Barabadi et al., 2014).

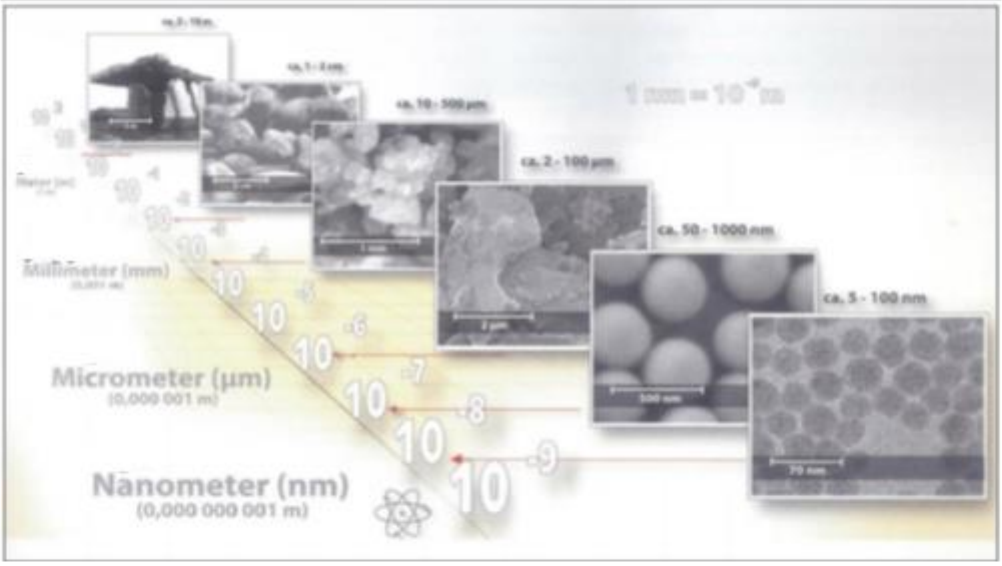

Figure 1. Scale objects in nano, micro, millimeter and meter (Leydecker, 2008)

Nanomaterials

Nanomaterials as that distinct class of advanced materials that can be produced so that the dimensions of its dimensions or the dimensions of its internal granules range from (1-100) nanometers. And that it has highly distinctive characteristics and characteristics that cannot be found clustered in traditional materials. Nanomaterials are the building materials of the twenty-first century, which is considered a benchmark for the advancement of the civilization of nations(Singh et al., 2017, Gupta et al., 2016). There are two main reasons behind changing the properties of materials when they are at the nanoscale:

A- An increase in the ratio of the surface area to the total volume of the material, as the surface area of the material increased, the size decreased and thus the free electrons responsible for the reactions would increase (Kattan, 2011). Figure (2) shows the change in the color of gold according to the size of its particles.

B- The quantitative restriction or confinement or the so-called quantum effect, means the restriction of the random movement of the electron to restrict or limit the electron within the specified energy range. We call it discrete(Son et al., 2009).

Figure 2. Shows the change in the color of gold according to the size of its grains (Aldrich, 2015).

Classification of Nanomaterials

1) Zero-dimensional Nanomaterials (0D)

They are materials in which electrons are bound in three dimensions, such as nanoparticles and quantum dots (Tiwari et al., 2012).

2) One-dimensional Nanomaterials (1D)

Materials in which electrons are restricted in motion in one dimension. Examples of these materials are chips or thin film such as nanomaterials employed in surface coating works, as well as manufacturing various semiconductor materials such as silicon and employing them in the manufacture of Solar cells (Tiwari et al., 2012).

3) Two-dimensional Nanomaterials (2D)

Materials in which electrons are restricted in motion in the two dimensions. Nanotubes or nanocylinders are important models for this class of materials and it would not be strange for carbon nanotubes to be filtered because they are used as supporting and strengthening materials for metal molds to raise their hardness values and improve their mechanical properties. And, in particular, raising its breakdown strength, as it combines other unique properties such as the superior ability to conduct thermal and electrical conductivity, in addition to its distinct chemical properties, it is expected to use nanotubes and nanowires in the manufacture of solar cell components, electronic chips, sensors and micro-electronic devices.

In addition to that, three-dimensional nanomaterials (3D) are, materials in which electrons are free to move in three dimensions are called (bulk), and examples of them are nanopowders, nanocapsules, fullerenes and polycrystalline materials where in nanostructural elements of 0D, 1D and 2D are closely related with each other and form interfaces(Hornyak et al., 2018). Figure (3) illustrates the classification of nanomaterials.

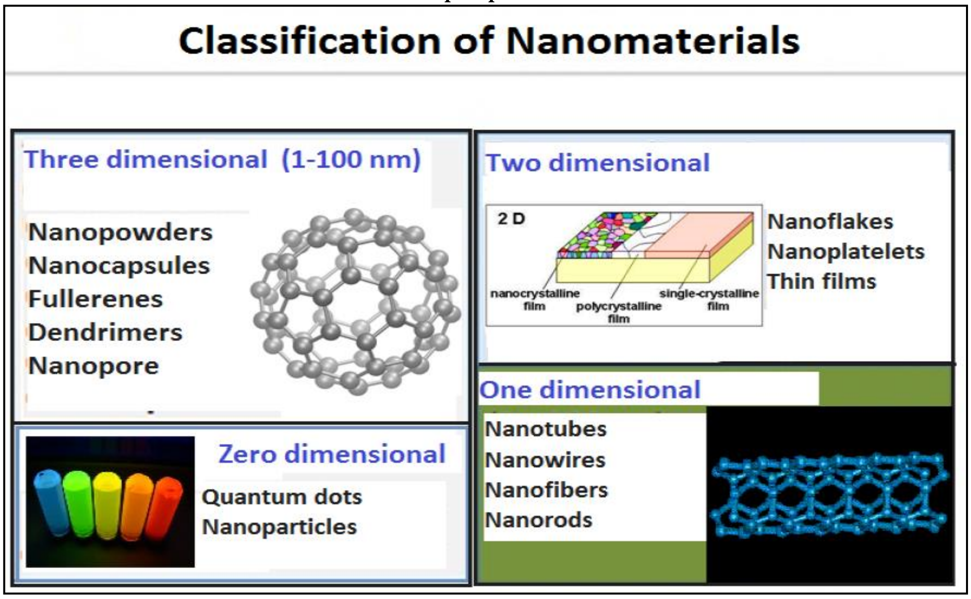

Figure 3. Classification of Nanomaterials (Mishra, 2013)

Techniques to Reach Nanoscale

Most famous nanomaterial manufacturing technology, Top-down approach and Bottom-up approach:

1) Top-down Approach

It begins with cutting the material (bulk), which means a large size, until it reaches pieces within the nanoscale, where nanoparticles are manufactured from larger particles using sculpting, grinding, fragmentation, or other techniques as in the microelectronic industries(Li et al., 2011, Khan et al., 2019).

2) Bottom–up Approach

Nanomaterials are prepared by building them, based on atoms or individual molecules, by direct control, and it is a far-reaching technology where they are arranged with each other until they reach the desired size and shape, and this process is similar to the process of building a wall (Xu et al., 2006, Iravani, 2011).

And by studying the top-down method, it led to the division of parts smaller and smaller until they reach a size of approximately 100 nanometers, then the bottom-up method that started with atom or part of the material, and the acquisition of the resulting material new properties not present in the material with its natural size, fewer defects and more homogeneous chemical composition(Vieu et al., 2000). Figure (4) illustrates the techniques for obtaining nanomaterials.

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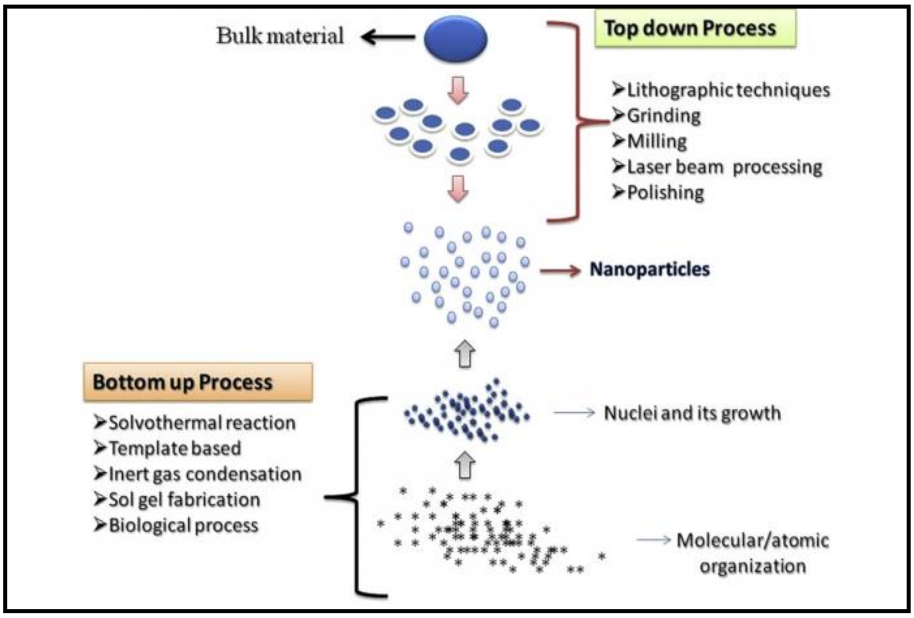

Figure 4. Represents the techniques for obtaining nanomaterials (Baker et al., 2013)

Methods for Obtaining the Nanoscale

Nanomaterials are prepared using physical, chemical and biological methods. The physical method involves using some expensive equipment, high temperature and pressure, and a lot of space to create the devices. The chemical method involves the use of toxic chemicals that can be dangerous to the environment and to the person handling it. Studies state that some of the toxic chemicals that we use in physical and chemical methods may present the problem in nanoparticles, which may be dangerous in the field of their application in the medical field. Thus, the need arose for an environmentally friendly and inexpensive way to synthesize nanoparticles and use fewer chemicals, which is the biological method (Li et al., 2011). Here are some of the preparation methods:

1) Precipitation Method

This method is done by dissolving the basic formation materials in deionized water by stirring, and then adding the reducing agent in the form of droplets to the liquid, after a period of time a precipitate forms at the bottom of the beaker, then the precipitate is separated from the solution and dried for the purpose of conducting the necessary tests for the resulting nanomaterial. This strategy includes very important points because it is a straightforward, low-priced, single-stage process and particle size and composition can be easily controlled in this method, and there are also different possibilities for modifying particle surface condition and general homogeneity (Iqbal et al., 2017). Figure (5) represents the precipitation method to form the nanomaterial.

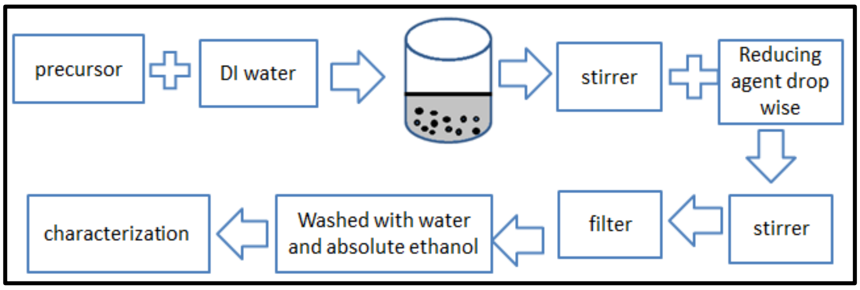

Figure 5. Represents the method of sedimentation to form the nanomaterial (Iqbal et al., 2017).

2) Solvothermal Method

In this method the chemical reaction takes place in a fixed vessel such as a pressurized gas vessel or autoclave, in which the solvents are transferred to temperatures much higher than their boiling point. Initially, the basic formation materials and the reducing agent are dissolved in the beaker and then transferred to the autoclave. The material is characterized after formation. This method is considered to be a versatile method, convenient and straightforward, but considerations of temperature, pressure and volume are very important(Demazeau, 2010). Figure (6): represents the solvothermal method for the formation of nanomaterials.

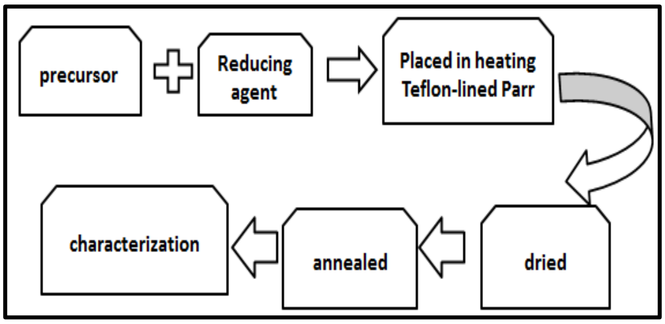

Figure 6. Represents the thermal solvent method for the formation of nanomaterials (Iqbal et al., 2017)

3) Hydrothermal Method

This method is mainly used to create a monocrystalline crystal in the aqueous form, also using an autoclave. This method is useful as it is able to manufacture large crystals of high quality but are unstable near the melting point, and this process also requires expensive and unsuitable equipment to monitor the crystals (Mohan et al., 2020). Figure (7): represents the formation of nanomaterials by the hydrothermal method.

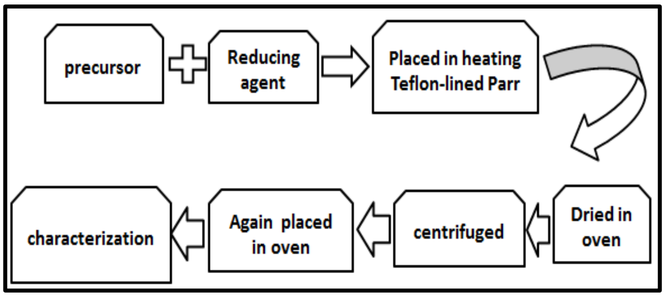

Figure 7. Represents the formation of nanomaterials by the hydrothermal method(Iqbal et al., 2017)

4) Sol Gel Method

This method is used to prepare materials in a variety of shapes, such as powders and thin films. It is considered one of the easy and cheap methods, by adding the solution to the sediment and after a period of time the mixture becomes in the form of a transparent solution and then turns into a gel. This method has benefits from it, the nanoparticles are formed at room temperature and it was found to be an effective and moderate way for the large-scale industrial production of micro-nanoparticles without any expensive chemicals or molds, but it is difficult to control the measurement of the oxides of grafted nanoparticles, which is the disadvantage of this method (Silva et al., 2020). Figure (8): represents the formation of nanomaterials by the sol- gel method.

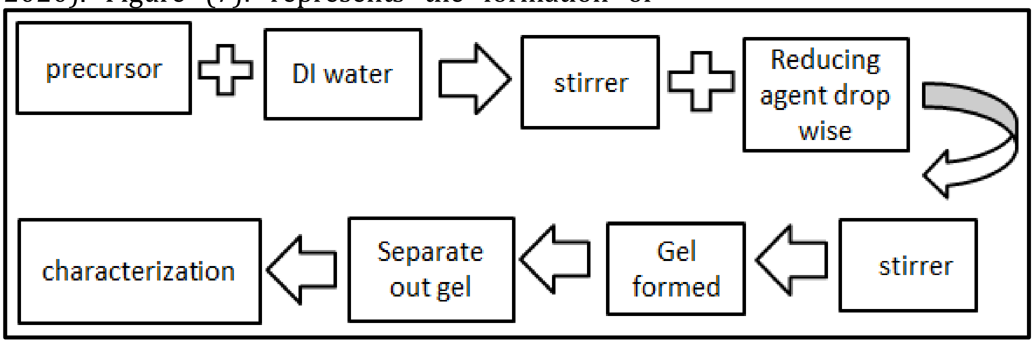

Figure 8. Represents the formation of nanomaterials by the sol-gel method (Iqbal et al., 2017)

5) Green Synthesis

It is a botanical synthesis of metallic nanoparticles, simple, easy to use, fast and environmentally friendly among all preparation methods. Biomolecules present in plant extracts can increase the generation rate of nanoparticles or product stability. Initially, the plant extract is collected from the leaves or from the juice of the plant. Then, by using the extract with the basic formation materials, the nanoparticles can be formed. In addition, the substances present in the plant extract play an important role in the surface composition and volume, as they can act as a covering and surrounding agent at the same time (Gowramma et al., 2015, Makarov et al., 2014). Figure (9): represents the formation of nanomaterials by the green synthesis method.

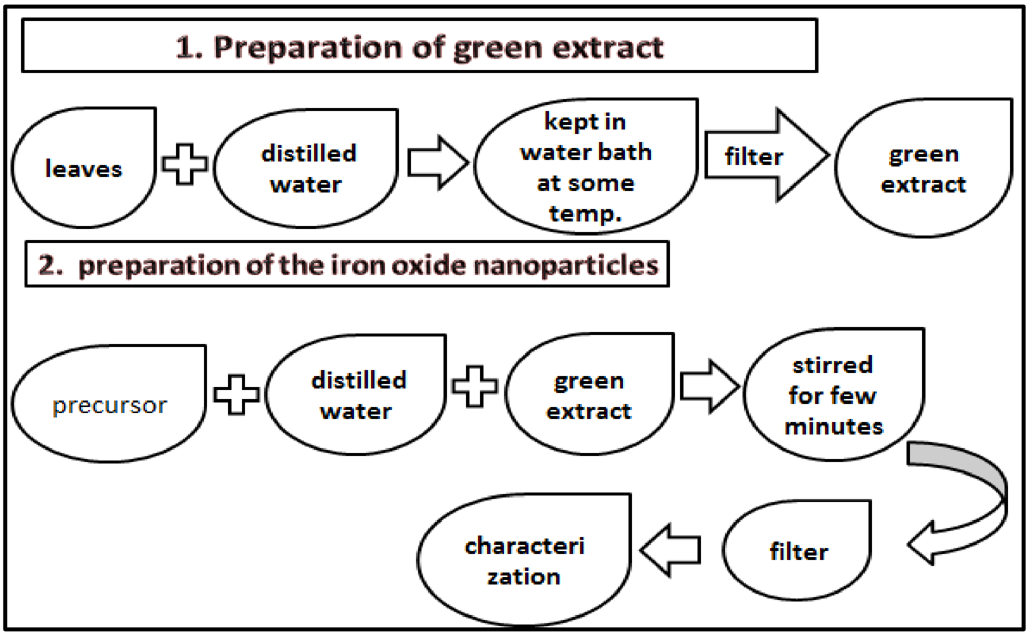

Figure 9. Represents the formation of nanomaterials by the green synthesis method (Iqbal et al., 2017)

6) Laser Ablation Method

Laser ablation is the interaction of laser light with the surface of a solid target, causing the solid target and a small amount of ocean (liquid) to be vaporized. The reaction products are usually nanoparticles consisting of atoms of both the target and the liquid, which form suspended in the liquid. Then this method is called laser ablation in the liquid, this method is distinguished over other physical methods chemical vapor deposition, vapor phase transfer, and laser ablation in a vacuum, etc. crystallized nanoparticles can be obtained in one step without the need for heat treatment and in pure colloidal liquids, chemicals such as surfactants can be added to liquids in order to control the size and state of nanoparticle aggregation, so it is considered a clean and green method to prepare minerals and metal oxides for nanoparticles (Simakin et al., 2004). Figure (10): represents the formation of nanomaterials by the method of laser ablation in a liquid.

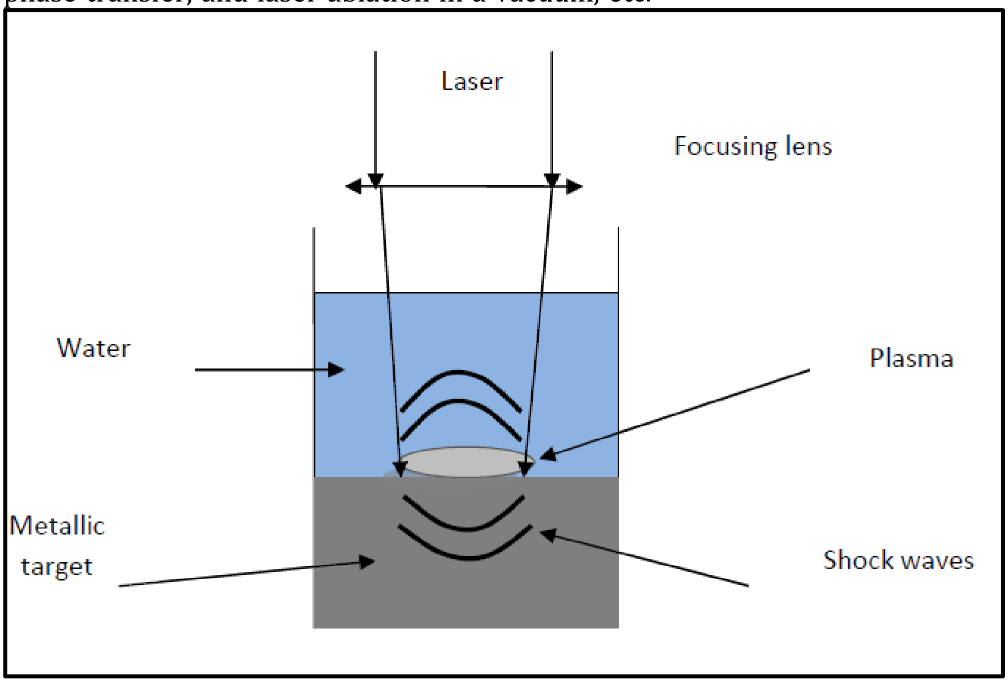

Figure 10. Represents the formation of nanomaterials by the method of laser ablation in a liquid (Zhu et al., 2001).

Techniques Used to Characterize Nanoparticles

1) X-ray Diffraction Technique (XRD)

X-rays are electromagnetic rays that interact primarily with electrons in an atom. When X-ray photons collide with electrons, some of the incident beam photons deviate from their original direction. If the wavelength of the incident X-ray does not change (that is, X-ray photons have not lost any energy) the process is called flexible scattering, where by the momentum of movement is only transformed in the scattering process. These are the X-rays that we measure in diffraction experiments and that give us information about the distribution of electrons in materials. On the other hand, in the process of inflexible scattering or Compton scattering, the X-rays transfer some of their energy to the electrons, so the neutral X-rays have a wavelength different from that of the incident X-rays(Holcomb, 1967).

According to Braque's law, when the rays fall on the crystal, the waves are reflected in more than one layer separated by a distance of d, and even when the reflected interference of these waves is constructive, the interference remains between them a fixed phase, where the path of each wave is equal to a whole number n of the wavelength λ(Fultz and Howe, 2012), Figure (11) represents the reflection of X-rays from the crystalline planes, and the path difference between the two waves applies to it:

$$n\lambda=2d\sin(\theta) \quad (1)$$

This relationship connects each of:
d: the distance between layers of atoms.
λ: the length of the rays wavelength.
θ: the angle between the incident ray and the plane of the crystal layer.
n: is an integer that specifies the degree of diffraction.

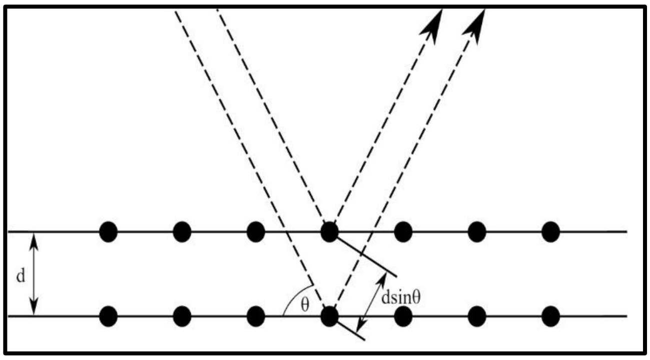

Figure 11. Crystalline levels and Braque's law (Yamaguchi et al., 2007)

The technique of X-ray diffraction is used to know the nature of the crystal structure, the main crystal phases, and the prevailing orientation of the materials prepared under certain conditions. As well as identifying some structural parameters such as grain size, lattice constants and the width of the curve at the middle of the peak (Abass et al., 2018).

2) Scanning Electron Microscopy (SEM)

It is a type of electron microscope that produces images of a sample by scanning the surface of the sample. When the electron beam scans the surface of the sample, it interacts with the surface and specifically extracts electrons from the surface of the sample. These extracted electrons are detected by the detector by attracting the scattered electrons and depending on the number of electrons that reach the detector, they record a certain degree of brightness on the screen. By using additional probes, the scattered electrons are detected by reflection from the surface of the back scattered sample, as well as the X-rays emitted from the sample (Poletti et al., 2003). Figure (12) represents a scanning electron microscope. A scanning microscope is used to:

* Topographic studies.
* Microstructure analysis.
* Elemental analysis if equipped with suitable detector (energy / dispersive wave rays). 48
* chemical composition.
* Initial mapping.

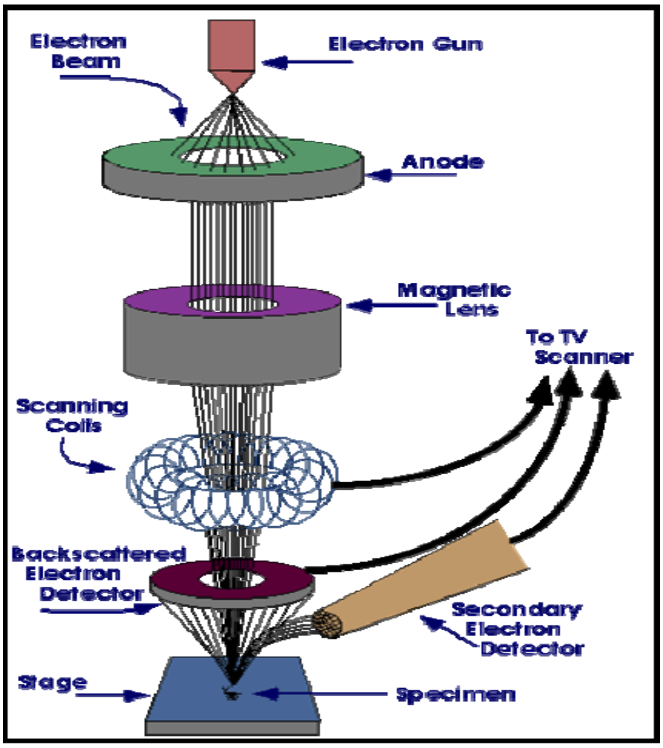

Figure 12. Represents a scanning electron microscope(Huggett and Shaw, 1997) (30)

3) Transmission Electron Microscopy (TEM)

A transmission electron microscope is a very powerful tool for materials science. A high-energy beam of electrons is illuminated through a very thin sample. Interactions between electrons and atoms can be used to observe features such as crystal structure, dislocations and grain boundaries, chemical analysis can also be performed. TEM can be used to study layer growth, composition, and defects in semiconductors. It works on the same basic principles as light microscopy, but it uses electrons instead of light. Since the wavelength of electrons is much smaller than the length of light, the optimum resolution that can be obtained for TEM images is better than for light microscopy (Das et al., 2013). Thus, TEM can reveal the finest details of the internal structure in some cases as small as individual atoms. Figure (13) represents the transmission electron microscope.

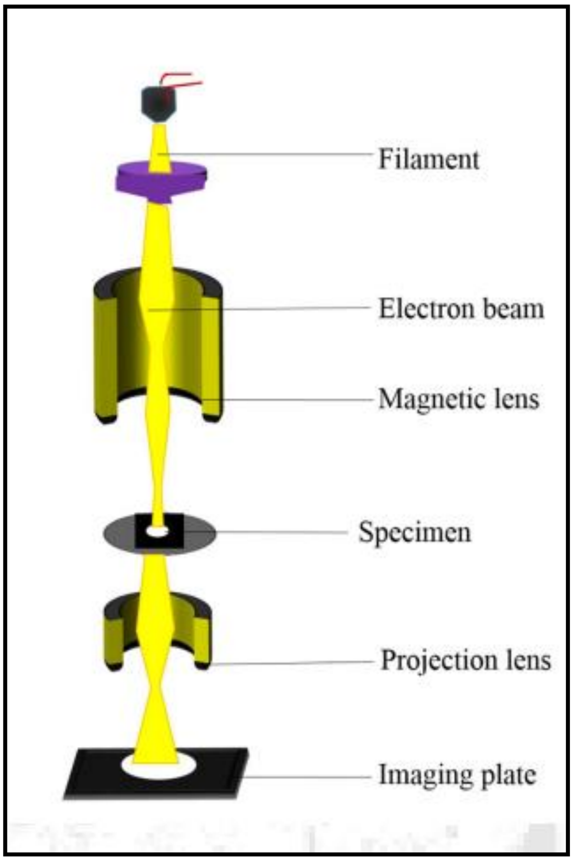

Figure 13. Represents the transmission electron microscope (McDonagh, 2015)

4) Atomic Force Microscope (AFM)

It is a device used in the field of nanotechnology to learn and map surfaces of nanoscale and micron dimensions. Also called a scanning force microscope; SFM. The device consists of a needle with micron dimensions that passes over the surface to be scanned, this needle is fixed to a horizontal holder while it is itself perpendicular to this holder and on the surface to be scanned, a laser beam is projected onto the holder, which rises and falls with the height and fall of the needle, and thus with a variety the surface topography is from high and low, and the laser ray reflex is captured on the holder on a receiver. Thus the topography of the scanned surface is determined and drawn according to the movement of the laser beam reflex (Last et al., 2010). Figure (14) represents an atomic force microscope.

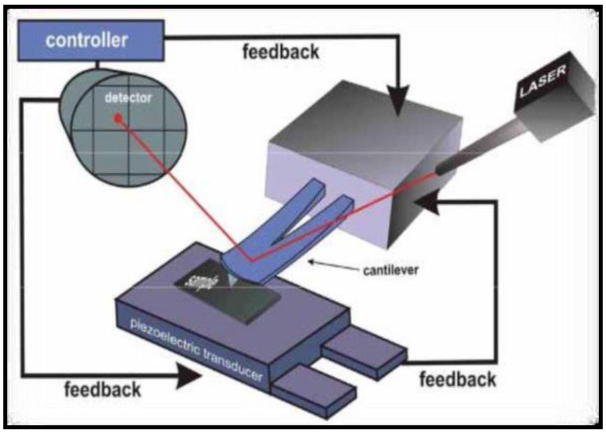

Figure 14. represents the atomic force microscope (Alexander et al., 1989)

5) Fourier-Transformed Infrared Spectroscopy (FTIR)

It is a technique used to obtain an infrared spectrum for the absorption or emission of a solid, liquid or gaseous substance. The Fourier Spectrometer simultaneously collects spectral high-resolution data over a wide spectral range. This provides a major advantage over a spectrophotometer, which measures intensity over a narrow range of wavelengths simultaneously. There are three infrared regions(Hassan and Gould, 1992) :

- Near infrared region υ (with wavelength number 4000-13000) cm-1
- The middle infrared region υ (with wavelength number 400-4000) cm-1
- Far-infrared region υ (with wavelength number 10-400) cm-1

By means of the vibrating frequencies detected by the device, it is possible to know the elements that are solidified in the sample. Figure (15) represents the vibrational movements of most of the particles.

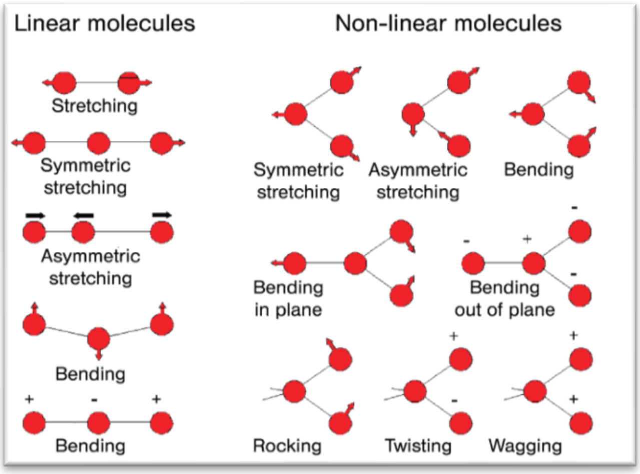

Figure 15. Represents the basic vibrations of most of the particles (Altindal et al., 2001)

6) Ultraviolet -Visible Spectroscopy (UV/Vis)

UV Visible spectroscopy is a type of spectroscopy which is classified under absorption spectroscopy, which occurs in the UV spectrum and in the visible spectrum. This means that this spectroscopy uses light in the visible fields and in the adjacent fields of the ultraviolet spectrum, and even parts of the near infrared (NIR) spectrum. The absorption or reflection in the visible field affects the sense of color appearing on chemicals, where electron transitions occur. As a result of the effect of electromagnetic radiation.

This spectroscopy is concerned with the electron transitions from steady state to excited state. It is thus complementary to fluorescence spectroscopy, which studies the fluorescence resulting from the transition from the excited state to the steady state. In visible and ultraviolet spectroscopy, the particles are exposed to electromagnetic radiation in the visible and ultraviolet fields, which leads to irritation and excitation of the valence electrons (such as the p or d electrons in the external orbits), that is, they gain energy and have an electron transfer within the energy levels of the molecule (Perkampus, 2013). Figure (16) is an illustrative diagram of the components of the dual beam spectrometer.

The energy of the photon absorbed for this transition to occur corresponds to the energy difference between the energy levels in which the transition takes place (Ali et al., 2020). Calculate that energy from the equation:

$$Eg = h\,C / \lambda \text{................} \quad (2)$$

Where it represents:

E: Energy

h: Planck's constant

ν :Frequency

c: the speed of light

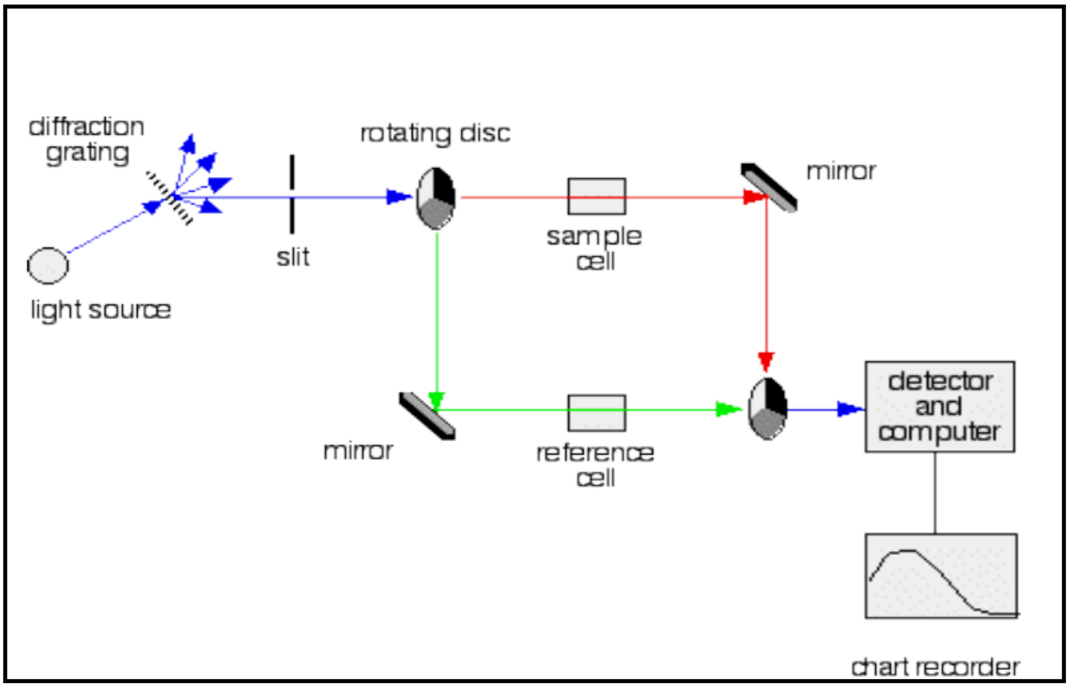

Figure 16. Illustration of the components of the dual beam spectrometer (Skoog et al., 2007)

Conclusion

At the end of this research we conclude that nanotechnology is one of the most important technologies today and in the future and has become at the forefront of the most important areas in all fields of science, because of its importance in improving products, treating diseases and serving humanity in all areas of life, as well as it gives great hope for revolutions. Scientific in physics, chemistry, biology, engineering and others.

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