



**Sudan University of Sciences & Technology**

**College of Graduate Studies**

**Bio Medical Engineering**



## **Fabrication and Characterization Carbon Nano Powder using Flame Synthesis**

تصنيع وتوصيف بكرة الكربون النانوية باستخدام طريقة تخليق اللهب.

A project submitted in partial fulfillment for the requirements of degree of M.Sc. in BME

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## الآية

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

قال تعالى:

اقْرَأْ بِاسْمِ رَبِّكَ الَّذِي خَلَقَ ﴿١﴾ خَلَقَ الْإِنْسَانَ مِنْ عَلَقٍ ﴿٢﴾ اقْرَأْ وَرَبُّكَ  
الْأَكْرَمُ ﴿٣﴾ الَّذِي عَلَّمَ بِالْقَلَمِ ﴿٤﴾ عَلَّمَ الْإِنْسَانَ مَا لَمْ يَعْلَمْ ﴿٥﴾ كَلَّا إِنَّ  
الْإِنْسَانَ لَيْطَغَى ﴿٦﴾ أَنْ رَأَهُ اسْتَعْتَى ﴿٧﴾ إِنَّ إِلَىٰ رَبِّكَ الرُّجْعَى ﴿٨﴾

سورة العلق الآيات 1-8

## *Dedication*

*I dedicate my dissertation work with special feeling  
of gratitude to my loving parents,,,*

*To who encouraged and helped me to complete the  
modest research with large*

*Respect to my teachers,,,*

## *Acknowledgment*

I would like to express my sincere thanks and gratitude to my teacher for letting me work on this project, **Dr. Fragoon Mohamed Ahmed**.

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## **Abstract**

This study is aim to fabricate carbon nano powder using flame synthesis method. With observance of growth conditions like temperature, catalyst and substrate, after that purify sample using centrifuge in order to remove impurities. Then sample was characterized using A Fourier Transform Infra-Red spectrometer(FTIR), in order to know functional groups. Else Characterize by Scanning Electronic Microscope (SEM), obtained the spectrum and image by scan surface of sample, image was appeared as form of carbon nano powder with different scales range which have same properties and behavior of Carbon Nanotube (CNTs) Martials.

## المستخلص

تهدف هذه الدراسة إلى تصنيع بديره الكربون النانويه باستخدام طريقه تخليق اللهب مع مراعاة ظروف النمو مثل درجة الحرارة، المحفز والتركيز، تم تنقيه العينة تنقيه باستخدام الطرد المركزي من أجل إزالة الشوائب. تم اختبار العينه وتعريضها للاشعه تحت الحمراء بواسطة جهاز (FTIR) لمعرفة المجموعات الوظيفيه ونسبها للعناصر الموجوده في العينه ومن ثم استخدم جهاز المسح الالكتروني الضوئي (SEM) لدراسه شكل العينه ومعرفة الحجم ونسبه التركيز لكل عنصر , ظهرت العينه في شكل مسحوق بديره الكربون النانويه والذي تبين انه يسلك نفس سلوك المواد النانويه .

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### List of abbreviations:

<b>Abbreviation</b>	<b>Definition</b>
<b>CNTs</b>	Carbon nano tubes.
<b>HREM</b>	High Resolution Electron Microscopy.
<b>SWCNTs</b>	Single-wall carbon nanotubes.
<b>MWCNTs</b>	Multiple walled carbon nanotubes.
<b>DWNT</b>	Double-walled carbon nanotubes DWNT.
<b>DC</b>	Direct current.
<b>Fe</b>	Iron.
<b>Co</b>	Cobalt.
<b>Ni</b>	Nickel.
<b>Y</b>	Yttrium.
<b>Mo</b>	Molybdenum.
<b>CVD</b>	Chemical Vapor Deposition.
<b>N<sub>2</sub></b>	Nitrogen.
<b>Ar</b>	Argon.
<b>K</b>	Kelvin.
<b>Co</b>	Cobalt.
<b>Cu</b>	Copper.
<b>Zn</b>	Zinc.

<b>CH<sub>4</sub></b>	Methane.
<b>C<sub>2</sub>H<sub>4</sub></b>	Ethylene.
<b>C<sub>2</sub>H<sub>2</sub></b>	Acetylene.
<b>O<sub>2</sub></b>	Oxidizer.
<b>CO<sub>2</sub></b>	Carbon oxidizer.
<b>H<sub>2</sub>O</b>	Water.
<b>H<sub>2</sub></b>	Hydrogen.
<b>C-C</b>	Single carbon bond.
<b>Tpa</b>	Terapascal.
<b>Gpa</b>	Gigapascal.
<b>W</b>	Watt.
<b>C°</b>	Celsius.
<b>H<sub>2</sub>O<sub>2</sub></b>	Hydrogen peroxide.
<b>KMnO<sub>4</sub></b>	Potassium permanganate.
<b>EM</b>	Electron Microscopy.
<b>TEM</b>	Transmission Electron Microscopy.
<b>SEM</b>	Scanning Electron Microscopy.
<b>Nm</b>	Nano meter.
<b>Mm</b>	Millimetres.
<b>μm</b>	Micrometres.



<b>FEG</b>	Field emission guns.
<b>3D</b>	Three dimension.
<b>eV</b>	Electron volt.
<b>JPEG</b>	Joint photographic explorer group.
<b>TIFF</b>	Tagged image file format.
<b>XRD</b>	X-ray diffraction.
<b>FTIR</b>	Fourier transform infrared spectroscopy.
<b>DNA</b>	Deoxyribonucleic acid.
<b>NDEO</b>	Nano scale-dispersed eye ointment
<b>C<sub>20</sub>H<sub>32</sub>O<sub>4</sub></b>	Acid resin.

# **CHAPTER ONE**

## **INTRODUCTION**

## **CHAPTER ONE**

### **INTRODUCTION**

#### **1.1 History of Nanotechnology**

American physicist Richard Feynman is considered the father of nanotechnology. He introduced the ideas and concepts behind nanotech in a 1959 talk titled "There's Plenty of Room at the Bottom." Feynman did not use the term "nanotechnology," but described a process in which scientists would be able to manipulate and control individual atoms and molecules (Anna Pratima, 2015).

Modern nanotechnology truly began in 1981, when the scanning tunneling microscope allowed scientists and engineers to see and manipulate individual atoms. IBM scientists Gerd Binnig and Heinrich Rohrer won the 1986 Nobel Prize in Physics for inventing the scanning tunneling microscope. Binnig and Rohrer were study Nanotechnology Center in Zurich, Switzerland, continues to build conducting research and developing new applications for nanotechnology.

The iconic example of the development of nanotechnology was an effort led by Don Eigler at IBM to spell out "IBM" using 35 individual atoms of xenon (jamis, david 2016).

By the end of the 20th century, many companies and governments were investing in nanotechnology. Major nanotech discoveries, such as carbon nanotubes, were made throughout the 1990s. By the early 2000s, nanomaterial were being used in consumer products from sports equipment to digital cameras.

Modern nanotechnology may be quite new, but nanometer-scale materials have been used for centuries (Harris, peter 2014).

As early as the 4th century, Roman artists had discovered that adding gold and silver to glass created a startling effect the glass appeared slate green when lit from the outside, but glowed red when lit from within. Nanoparticles of gold and silver were suspended in the glass solution, coloring it.

Artists from China, western Asia, and Europe were also using nanoparticles of silver and copper, this time in pottery glazes. This gave a distinctive "luster" to ceramics such as tiles and bowls (Vivekpatel, et al 2015).

Modern microscopy revealed the technology of "Damascus steel," a metal used in South Asia and the Middle East. Swords made with Damascus steel are legendary for their strength, durability, and ability to maintain a very sharp edge.

Study of nanotechnology was developed every day to get benefit of its unique properties and use it in health, education, environment...etc. (M. Wilson etal, 2016).

## **1.2 Research problem**

Carbon nano powder have unique properties like electrical, mechanical, biocompatibility, high elasticity, ultra weight and other properties which allow to use it in several aspects of life, specially their applications in medical field so we want try to know how we get benefit then apply in future work.

## **1.3 Research Objectives**

### **1.3.1 Objectives:**

- The main objective of this study is focusing on fabricate carbon nano powder, using flame synthesis method.
- To purify carbon nano powder after fabrication using centrifuge in order to remove impurities.
- To characterize sample using Fourier transform infrared spectroscopy (FTIR) and scanning electronic microscope (SEM.).

#### **1.4 Thesis layout:**

This thesis consists of six chapters, chapter one discuss the problem and research objectives chapter two reviews previous study of nanotechnology and carbon nanotube, chapter three include theoretical background of CNTs (discover, synthesis method, types and properties characterization of CNTs, purification methods and mainly applications in life).

Methodology, material process and techniques were discussed in chapter four, chapter five contain result and discussion, chapter six contain conclusion and recommendations.

**CHAPTER TWO**  
**LITREATURE REVIEW**

## CHAPTER TWO

### LITREATURE REVIEOW

#### 2.Literature Review:

At the beginning all attempt to prepare carbon was came to nothing and all carbon structure was prepared by laser vaporization of graphite not from synthetic organic chemistry, (Harry Kroto and s 1950).

Faraday (1955) and Gustav Mie (1957) begun the history of nanoparticle using roman glassmakers, glasses containing nanosized metal particles Lyncurgus cup.

In diffused light In focused light Explained by Michael and Photographic films using silver halide photochemistry (silver nanoparticles) by Richard Feynman(1958).H. Davy

Richard Feynman is considered the father of nanotechnology. He introduced the ideas and concepts behind nanotech in a 1959 talk titled "There's Plenty of Room at the Bottom." Feynman did not use the term "nanotechnology," but described a process in which scientists would be able to manipulate and control individual atoms and molecules.

C. Maxwell, G.Eastman predicted the existence of electron beam lithography, scanning tunneling microscope and building circuits on the scale of nanometer for powerful computers in diffused (1961). Through the decade nanoscale Materials Inc., neutralizer for chemical hazards of dry powder formulation, (Reactive Nanoparticle (RNPTM)) to bind with a variety of chemical warfare agents and toxic chemicals, and chemically convert them to safer by-products, NanoScale Materials Inc (1963)

In august 1985the two scientists were begun series experiments by vaporisation of graphite they was immediately struck by result in the distribution of gas-phase carbon clusters detected by mass spectrometry  $C_{60}$  was dominant species.

Modern nanotechnology truly began in 1981, when the scanning tunneling microscope allowed scientists and engineers to see and manipulate individual atoms. IBM scientists



Gerd Binnig and Heinrich Rohrer won the 1986 Nobel Prize in Physics for inventing the scanning tunneling microscope. Binnig and Rohrer were study Nanotechnology Center in Zurich, Switzerland, continues to build conducting research and developing new applications for nanotechnology.

The first use of the term 'nanotechnology' was by Taniguchi (1974) at the International Conference on Precision Engineering (ICPE). His definition referred to 'production technology to get extra high accuracy and ultrafine dimensions, that is, the preciseness and fineness on the order of 1 nm (nanometer),  $10^{-9}$  m in length.' Ideas of Nano technological strategy, which were put forward by Feynman, were developed by

E. Drexler in his book 'Vehicles of creation: the arrival of the nanotechnology era' (1986).

By rolling a graphene sheet into a cylinder and capping both end of the cylinder with a half of fullere molecule a carbon nanotube is formed. Harry Kroto discovered  $C_{60}$  molecule in (Kroto, et al. 1988) while experimenting a laser ablation system for the vaporization of graphite by laser beams and depositing them on a copper collector and it was the beginning of a new area in carbon material science.

$C_{60}$  was produced in bulk by used high power laser to vaporise graphite which produced red solution which has been dried to produce plate like 'fullerite' contain 90%  $C_{60}$  and 10%  $C_{70}$  .( Kratschmer and Huffman, 1990).

By the end of the 20th century, many companies and governments were investing in nanotechnology. Major nanotech discoveries, such as carbon nanotubes, were made throughout the 1990s. By the early 2000s, nanomaterial were being used in consumer products from sports equipment to digital cameras.

Iijima experimented this technique in order to observe fullerene and by passing large current between two graphite rods, he vaporized them and condensed them on Cu tip. When he looked at the result through an electron microscope, he noticed something unexpected; he

discovered carbon nanotubes in the NEC laboratory in Japan the carbon nanotubes CNTs was discovered by Iijima then he decided to use TEM to see more details about CNTs which it accompanied by other materials include nanoparticles. (Sumio Iijima, 1991)

Scientists were reported synthesis of single wall of carbon nanotube (SWCNTs) and the multiple wall of carbon nanotube (MWCNTs) was discovered by used catalyst. (Iijima, et al 1993)

Although Feynman and Drexler certainly popularized nanotechnology, their influence did not directly lead to the design of Nano scale materials. Rapid progress in nanotechnology could only take place after the arrival of sophisticated instrumentation, capable of viewing and manipulating materials on the Nano scale.

The Nobel Prize in 1994 in Physics were awarded to Gerd Binnig and Heinrich Rohrer to honor their design of the scanning tunneling microscope (STM). They shared the Prize with Ernst Ruska, the inventor of the first electron microscope, another essential tool for the modern nanomaterial's scientist. In fact, the resolution of modern electron microscopes are now high enough to provide images of individual atoms, and are often or oxidation state of the surface atoms

DR Salvetat and other scientist stated that Some of carbon nanotube's excellent physical properties are high aspect ratio, high Young modulus, high tensile strength, high thermal and electrical conductivity (Salvetat, et al. 1995). CNTs which synthesised by laser vaporisation of graphite resulted high yield of SWCNTs (Smallery's group, 1996).

High yields of SWCNTs was obtained by using a very simple CO<sub>2</sub> laser system operating in a continuous wave, (Braid, 1998).

In this approach, a feedstock also at: Eloret Corporation. Such as CO or a hydrocarbon is heated from 800–1000°C with a transition metal catalyst to promote nanotube growth (Kong, et al. 1999).

First policy conference on advanced nanotechnology was held. First center for nano mechanical systems was established, Feynman Prize in Nanotechnology was awarded for designing stable protein structures and for constructing a novel enzyme with an altered function.

(braidy,2000)

He also pointed out the role of some laser parameters (such as laser power, repetition rate, pulse duration & laser wavelength) on the synthesis & characteristics of SWCNTs. (braidy, 2002).

Chemical vapour deposition (CVD) has been widely used to grow CNTs in recent years 3D Nano systems like robotics, 3D networking and active nano products that change their state during use were prepared. (M Meyyappan; et al. 2003).

Scientists were work for develop and detect another method for prepare CNTs, which are arc discharge, laser vaporization and chemical vapor deposition (CVD) methods. (Baddour and Briens, 2005).

CVD was observed as suitable for product CNTs, and Catalyst material is very important for CNT growth, which are appropriate for CNT growth but especially Fe, Co, Ni are the best ones according to many researchers studied previously.(Rashidi, 2007).Carbon nanotube was fabricated by used -reinforced Aluminum strips using rolling technique. the Al-CNT mixtures was blended in either a mixer-shaker at a rotary speed of 46 rpm, or under argon in a planetary mill at a rotary speed of 300 rpm properties, (2008, Christoph T. Wirth) . Purification of carbon nanotubes (CNTs) is a very actively discussed topic in contemporary CNT literature. To a large extent, impurities embedded in CNTs influence the physical and chemical characteristics of the CNTs. Different purification methods yield different CNT characteristics and may be suitable for the production of different types of CNTs.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) widely used in material science, metallurgy science and life science researches. TEM is an imaging technique where a beam of electrons is focused onto a specimen causing an enlarged version to appear on a fluorescent screen or layer of photographic film. SEM is a technique of electron microscope to produce high resolution images of a sample surface. This article describes the basic principle of TEM and SEM, and their applications. Nature and Science. (2006).

Developments in the purification Methods of CNTs were reviewed, and the production methods are briefly discussed and summarized. This is followed by a detailed description of the three major purification methods. Chemical, physical, and multi-step purification. (PeiseanGoh, et al 2008)

Single wall carbon nanotube (SWNT) was synthesized in flames form using different burner configurations and conditions the experiment have common morphological trends but some properties was not appear clearly when they characterized it (M Meyyappan, 2010-2011)

Because of their high young modulus and low weight they are useful as reinforcing agents in composite materials and in a variety of applications such as sensors, field emission devices, flat panel displays, energy storage, electrochemical devices and electronic devices (2013)

XRD provides information on structures, phases, preferred crystal orientations (texture), and other structural parameters, such as average grain size, crystallinity, Strain, and crystal defects. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of

Atoms within the lattice. Consequently, the X-ray diffraction pattern is the fingerprint of periodic atomic arrangements in a given material. This review summarizes the scientific trends associated with the rapid development of the technique of X-ray diffraction

Over the past five years pertaining to the fields of pharmaceuticals, forensic science, geological applications, microelectronics, and glass manufacturing, as well as in corrosion analysis. Taylor & Francis (2014).

Controllable growth of CNTs were applied true and proper. Though growth conditions play a significant role in properties of CNTs and their mechanisms to be fully established and still being researched heavily, they are good thermal conductors along their axis but act as insulators in the lateral direction. Major manufacturing techniques employed for fabrication of CNTs are Arc discharge, Laser Ablation and Chemical vapor deposition. Carbon Nanotubes are extending our ability to fabricate devices such as molecular

Probes, pipes, wires, bearings, springs, gears and pumps (rajesh et al, 2014)

(SWCNTs & MWCNTs) have already proven to serve as safer and more effective alternatives to previous drug delivery, among the various methods, (Kalpna Varshney, 2015).

In same year Dr. Rafiq Zakaria Campus, Rauza Bagh, Aurangabad- 431001, *Maharashtra, India* discuss an application of Nanotechnology in various fields such as health and medicine, electronics, energy and environment, is discussed in detail. Applications of nano particles in drug delivery, protein and peptide delivery, cancer are explained. Applications of various nano systems in cancer therapy such as carbon nano tube, dendrimers, nano crystal, nano wire, nano shells etc. are given. The advancement in nano technology helps in the treatment of neuro degenerative disorders such as Parkinson's disease and Alzheimer's disease. Applications of nano technology in tuberculosis treatment, the clinical application of nanotechnology in operative dentistry, in ophthalmology, in surgery, visualization, tissue engineering, antibiotic resistance, immune response are discussed in this article. Nano pharmaceuticals can be used to detect diseases at much earlier stages

Nano carrier-drug conjugates are more effective and selective; they can reduce the toxicity and other adverse side effects in normal tissues by accumulating drugs in target sites. So

scientist seen that they needed to develop and apply safe nanomaterial in drug delivery in the future. (MaharshiDayanand, 2016).

Also in 2016 CNTs exist as single (SWNTs), and multi-walled (MWNTs) structures. Scientist present several interesting properties, such as high aspect-ratio, ultra-light weight, strength, high thermal conductivity and electronic properties ranging from metallic to semiconducting. The production of carbon nanotubes can be done by plasma based synthesis method or arc discharge evaporation method, laser ablation method, thermal synthesis process, chemical vapor deposition and by plasma-enhanced chemical vapor deposition. The CNTs are valuable in the field of drug delivery, blood cancer, breast cancer, brain cancer, liver cancer, cervical cancer, gene therapy, immune therapy, biomedical imaging, biosensors and tissue engineering. This review leads to a useful knowledge related to general overview, types, preparation methods and applications of CNTs.

Central composite design (CCD) was used for controlling the size of clusters and achieving uniform distribution of CNTs particles. (NargesMohammadian, 2017)

Significant progress has been made not only in improving the yields of carbon nanotubes, but also in gaining a profound fundamental understanding of the growth processes. Flames are emerging as a powerful tool for the synthesis of carbon nanotubes and carbon nanofibers. The flame volume provides a carbon-rich chemically reactive environment capable of generating nanostructures during short residence times in a continuous single-step process. The present work provides a concise review of the advances made over the past two decades in the areas of flame synthesis of carbon nanotubes and carbon nanofibers. An overview of existing flame methods to synthesize carbon nanotubes is first provided. Various catalytic materials, fuel types, and flame configurations have been employed in an attempt to achieve controlled synthesis of carbon nanotubes and carbon

Nanofibers. Diffusion and premixed flames in counter-flow and co-flow geometries are also discussed. Various hydrocarbon fuels, oxygen enrichment, and dilution with inert gases are then examined in detail. The ability to synthesize and control carbon

Nanotubes and carbon nanofibers is essential for the fabrication of Nano mechanical and electrical devices. A fundamental understanding of the growth mechanism and development of control methods is critically important to address these issues. The

Purpose of the present review is to clarify the growth mechanisms and achieve controlled flame synthesis of carbon nanotubes and carbon nanofibers.(Junjie Chen\*, Xuhui Gao,2017)

Properties can be manipulated by controlling the diameter, chirality, wall nature, and length of CNTs which can lead to use as applications in important field of life, like biomedical, electrical application (Shabeer Ahmad, 2018).Carbon Nanotubes was synthesised via Chemical Vapour Deposition (CVD) from liquefied petroleum gas (LPG) without Hydrogen and the characterization of CNTs was confirmed the purity of the product which about 91.2% (w/w). (Nguyen Duc Vu Quyen, 2019).

Carbon nanotubes (CNTs) were synthesized by the flame fragment deposition (FFD) technique using Iraqi liquefied petroleum gas (LPG) as a source of carbon in a, then it purified it by H<sub>2</sub>O<sub>2</sub>/acetone ,TEM reflected carbon CNTs, which extends their properties for new advanced applications (Asmaa H, 2020).

**CHAPTER THREE**  
**THEORTICAL BACK GROUND**



## CHAPTER THREE

### THEORETICAL BACK GROUND

#### 3.1 Introduction of nanotechnology:

Nanotechnology is the study of extremely small structures. The word “Nano” means very small or miniature size, Nanotechnology is the treatment of individual atoms, molecules, or compounds into structures to produce materials and devices with special properties. Nanotechnology involve understand and control matter and nanometer scale deals with dimensions between approximately in the size of 0.1 to 100 nm; however it is also inherent that these materials should display different properties such as electrical conductance chemical reactivity, magnetism, optical effects and physical strength, from bulk materials as a result of their small size, and thus can be used for a broad range of applications and the creation of various types of nano materials and nano devices. Nanotechnology can increase the surface of area materials and this allow more atoms to interact with other materials that achieve nano material scale to be more durable and conductive. (M,Wilson et al 2016).

Sometimes a distinction is made between nanotechnology and nanoscience, the latter focusing on the observation and study of phenomena at the nanometre scale, and ways of manipulating matter at that scale, at which many properties of matter differ from those familiar at larger scales. The distinction is not of great importance, however: the nanotechnologist will perforce have to observe, study and manipulate matter in the course of his or her work.

‘Nano science’ suggests a solid body of theory, upon which a technology could be built; such theory is still inchoate, however, and the nanotechnologist is as likely to contribute to it as the Nano-scientist. (Jeremy J. Ramsden , 2018)

### **3.2 Classifications of nanomaterial:**

There are different type of nanomaterial and different way for classify them.

#### **3.2.1 Natural nanomaterial:**

As the name suggest are those that naturally occur in the world it include particles made up by volcanic, smoke, and even some like molecule in our bodies such as hemoglobin in our blood.

#### **3.2.2 Nanoparticles:**

Nanoparticles can include carbon, like fullerenes, as well as nanometer-scale versions of many other elements, such as gold, silicon, and titanium. Quantum dots, a type of nanoparticle, are semiconductors made of different elements, including cadmium and sulfur. Quantum dots have unusual fluorescent capabilities. Scientists and engineers have experimented with using quantum dots in everything from photovoltaic cells (used for solar power) to fabric dye.

#### **3.2.3 Fullerenes:**

Fullerenes are allotropes of carbon. Allotropes are different molecular forms of the same element. The most familiar carbon allotropes are probably diamond and graphite, a type of coal. Fullerenes are atom-thick sheets of another carbon allotrope, graphene, rolled into spheres or tubes.

The most familiar type of spherical fullerene is probably the buckminsterfullerene, nicknamed the buckyball. Buckyballs are nanometer-sized carbon molecules shaped like soccer balls tightly bonded hexagons and pentagons.

Buckyballs are very stable and able to withstand extreme temperatures and pressure. For this reason, buckyballs are able to exist in extremely harsh environments, such as outer space. In fact, buckyballs are the largest molecules ever discovered in space.

### **3.2.4 Man-made nanomaterial:**

Are those that occur from object or process that created by people for example include exhaust from fossil fuel burning engines and some forms of pollution.

The properties of nanoparticles have been important in the study of nanomedicine(Briens,(2015).

There are four main types of intentionally produced **man-made nanomaterials:**

Carbon-based, metal-based, dendrimers, and nanocomposites.

#### **Carbon-based nanomaterials:**

Carbon-based nanomaterials are intentionally produced fullerenes. These include carbon nanotubes and buckyballs.

Carbon nanotubes are often produced using a process called carbon assisted vapor deposition. In this process, scientists establish a substrate, or base material, where the nanotubes grow. Silicon is a common substrate. Then, a catalyst helps the chemical reaction that grows the nanotubes. Iron is a common catalyst. Finally, the process requires a heated gas, blown over the substrate and catalyst. The gas contains the carbon that grows into nanotubes, we will discussed synthesis of CNTs later by details.

#### **Metal-based nanomaterials:**

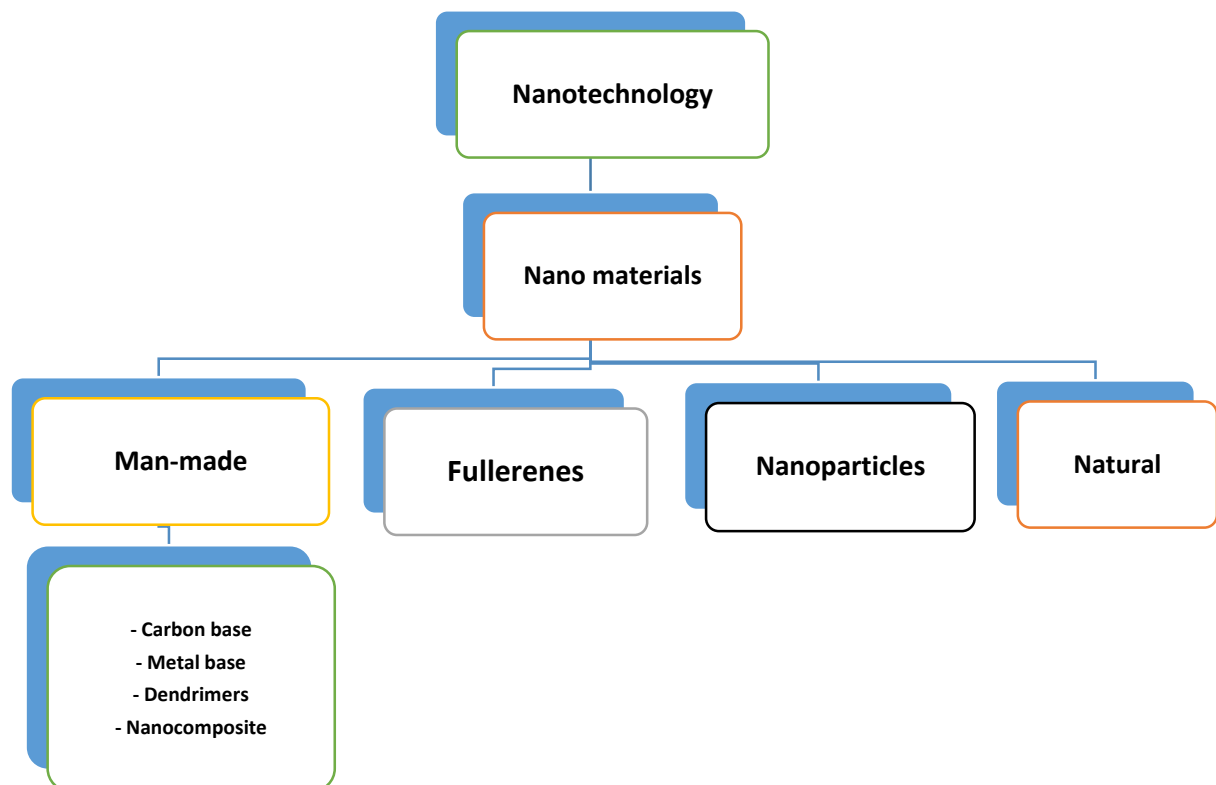
Metal-based nanomaterials include gold nanoparticles and quantum dots. Quantum dots are synthesized using different methods. In one method, small crystals of two different elements are formed under high temperatures. By controlling the temperature and other conditions, the size of the nanometer-scale crystals can be carefully controlled. The size is what determines the fluorescent color. These nanocrystals are quantum dots tiny semiconductors suspended in a solution.

### **Dendrimers:**

Dendrimers are complex nanoparticles built from linked, branched units. Each dendrimer has three sections: a core, an inner shell, and an outer shell. In addition, each dendrimer has branched ends. Each part of a dendrimer its core, inner shell, outer shell, and branched ends can be designed to perform a specific chemical function. Dendrimers can be fabricated either from the core outward (divergent method) or from the outer shell inward (convergent method).

### **Nanocomposites:**

Composites combine nanoparticles with other nanoparticles or with larger, bulk-type materials. Nanoparticles, such as Nano sized clays, are already being added to products ranging from auto parts to packaging materials, to enhance mechanical, thermal, barrier, and flame-retardant properties. (Anna Pratima, 2015).



**Fig (1.1) Classifications of Nano materials.**

### **3.3 General application of nanotechnology:**

#### **Energy**

Nanotechnology is helping inform the development of alternative energy sources, such as solar and wind power. Solar cells, for instance, turn sunlight into electric currents. Nanotechnology could change the way solar cells are used, making them more efficient and affordable.

Solar cells, also called photovoltaic cells, are usually assembled as a series of large, flat panels. These solar panels are big and bulky. They are also expensive and often difficult to install. Using nanotechnology, scientists and engineers have been able to experiment with print-like development processes, which reduces manufacturing costs. Some experimental solar panels have been made in flexible rolls rather than rigid panels. In the future, panels might even be "painted" with photovoltaic technology.

The bulky, heavy blades on wind turbines may also benefit from nanotech. An epoxy containing carbon nanotubes is being used to make turbine blades that are longer, stronger, and lighter. Other nanotech innovations may include a coating to reduce ice build-up.

Nanotech is already helping increase the energy-efficiency of products. One of the United Kingdom's biggest bus operators, for instance, has been using a nano-fuel additive for close to a decade. Engineers mix a tiny amount of the additive with diesel fuel, and the cerium-oxide nanoparticles help the fuel burn more cleanly and efficiently. Use of the additive has achieved a 5% annual reduction in fuel consumption and emissions.

## **Water**

Access to clean water has become a problem in many parts of the world. Nano materials may be a tiny solution to this large problem.

Nano materials can strip water of toxic metals and organic molecules. For example, researchers have discovered that nanometer-scale specks of rust are magnetic, which can help remove dangerous chemicals from water. Other engineers are developing nanostructured filters that can remove virus cells from water.

Researchers are also experimenting with using nanotechnology to safely, affordably, and efficiently turn saltwater into freshwater, a process called desalination.

In one experiment, nano-sized electrodes are being used to reduce the cost and energy requirements of removing salts from water.

## **Nano medicine**

Nanotechnology can help medical tools and procedures be more personalized, portable, cheaper, safer, and easier to administer. Silver nanoparticles incorporated into bandages, for example, smother and kill harmful microbes. This can be especially useful in healing burns.

Nanotech is also furthering advances in disease treatments. Researchers are developing ways to use nanoparticles to deliver medications directly to specific cells. This is especially promising for the treatment of cancer, because chemotherapy and radiation treatments can damage healthy as well as diseased tissue.

Dendrimers, nanomaterials with multiple branches, may improve the speed and efficiency of drug delivery. Researchers have experimented with dendrimers that deliver drugs that slow the spread of cerebral palsy in rabbits, for example.

The list goes on. Fullerenes can be manipulated to have anti-inflammatory properties to slow or even stop allergic reactions. Nanomaterial may reduce bleeding and speed coagulation.

Diagnostic testing and imaging can be improved by arranging nanoparticles to detect and attach themselves to specific proteins or diseased cells.

### **Electronics**

Nanotechnology has revolutionized the realm of electronics. It provides faster and more portable systems that can manage and store larger and larger amounts of data.

It improved display screens on electronic devices. This involves reducing power consumption while decreasing the weight and thickness of the screens.

Nanotechnology has allowed glass to be more consumer-friendly. One glass uses nanomaterials to clean itself, for example. As ultraviolet light hits the glass, nanoparticles become energized and begin to break down and loosen organic molecules dirt on the glass. Rain cleanly washes the dirt away. Similar technology could be applied to touch-screen devices to resist sweat (Rajaashree 2009).

### **Food**

The food industry is using nanomaterials in both the packaging and agricultural sectors. Clay nano composites provide an impenetrable barrier to gases such as oxygen or carbon dioxide in lightweight bottles, cartons, and packaging films. Silver nanoparticles, embedded in the plastic of storage containers, kill bacteria.

Engineers and chemists use nanotechnology to adapt the texture and flavor of foods. Nanomaterials' greater surface area may improve the "spreadability" of foods such as mayonnaise, for instance.

Nanotech engineers have isolated and studied the way our taste buds perceive flavor. By targeting individual cells on a taste bud, nanomaterials can enhance the sweetness or saltiness of a particular food. A chemical nicknamed "bitter blocker," for instance, can trick the tongue into not tasting the naturally bitter taste of many foods.

## **Clothing**

Scientists and engineers are using nanotechnology to enhance clothing. By coating fabrics with a thin layer of zinc oxide nanoparticles, for instance, manufacturers can create clothes that give better protection from ultraviolet radiation, like that from the sun. Some clothes have nanoparticles in the form of little hairs or whiskers that help repel water and other materials, making fabric more stain-resistant.

Some researchers are experimenting with nanotechnology for "personal climate control." Nano fiber jackets allow the wearer to control the jacket's warmth using a small set of batteries.

## **Cosmetics**

Many cosmetic products contain nanoparticles. Nanometer-scale materials in these products provide greater clarity, coverage, cleansing, or absorption. For instance, the nanoparticles used in sunscreen (titanium dioxide and zinc oxide) provide reliable, extensive protection from harmful UV radiation. These nanomaterials offer better light reflection for a longer time period.

Nanotechnology may also provide better "delivery systems" for cosmetic ingredients. Nanomaterials may be able to penetrate a skin's cell membranes to augment the cell's features, such as elasticity or moisture.

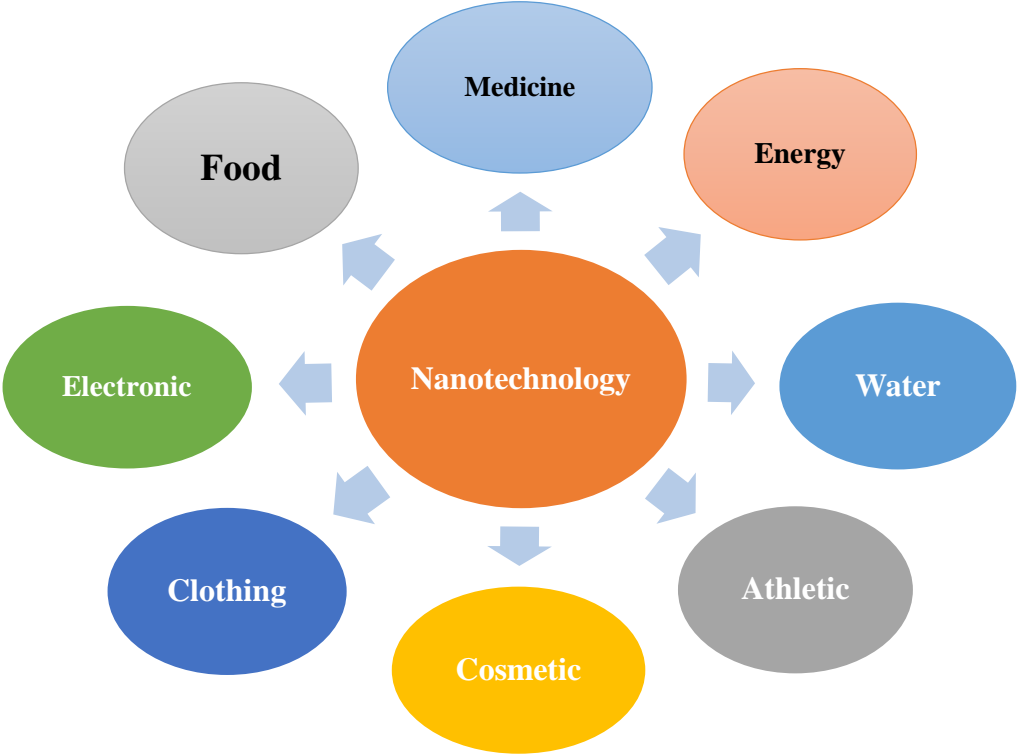
## **Athletics**

Nanotech is revolutionizing the sports world. Nanometer-scale additives can make sporting equipment lightweight, stiff, and durable.

Carbon nanotubes, for example, are used to make bicycle frames and tennis rackets lighter, thinner, and more resilient. Nanotubes give golf clubs and hockey sticks a more powerful and accurate drive.



Carbon nanotubes embedded in epoxy coatings make kayaks faster and more stable in the water. Similar epoxy keeps tennis balls bouncy (Briens, (2015)).



**Fig (2.1) General application of nanotechnology.**

In this section we will concern of carbon nanotubes ‘‘CNTs’’ which is a branch of nanotechnology we will discuss (Synthesis, Characterization, Application of CNTs).

### **3.4 Carbon Nanotube (CNTs):**

Carbon nanotubes (CNTs) are known as nano-architected allotropes of carbon, having graphene sheets that are wrapped forming a cylindrical shape. Rolling of graphene sheets in different ways makes CNTs either metals or narrow-band semiconductors. Over the years, researchers have devoted much attention to understanding the intriguing properties of CNTs. The discovery of carbon nanotubes by Iijima in 1991 using High Resolution Electron Microscopy (HREM) has stimulated intense experimental and theoretical studies on carbon nanotubes. Carbon nanotubes are allotropes of carbon that have a nanostructure, which can have a length-to-diameter ratio more than 1,000,000. Theoretical studies have predicted exciting electronic properties for the nanotubes. The potential application of carbon nanotubes to the synthesis of nanowires has been demonstrated.

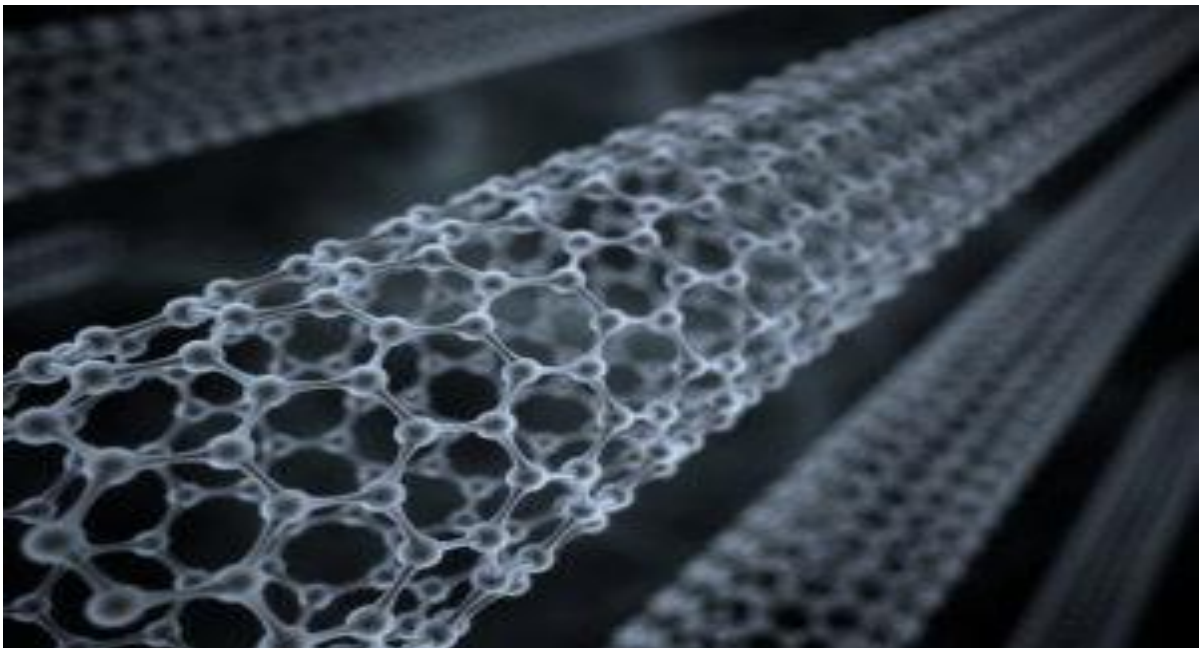
They exhibit some unusual properties like a high degree of stiffness, a large length-to-diameter ratio, and exceptional resilience, and for this reason, they are used in a variety of applications. These properties can be manipulated by controlling the diameter, chirality, wall nature, and length of CNTs which are in turn (Iijima, 1991).

### **3.5 Types of carbon nanotubes (CNTs):**

A Carbon Nanotube is a tube-shaped material, made of carbon, having a diameter measuring on the nanometres scale. The graphite layer appears somewhat like a rolled-up chicken wire with a continuous unbroken hexagonal mesh and carbon molecules at the apexes of the hexagons known as graphene. Carbon Nanotubes have many structures, differing in length, thickness, and in the type of helicity and number of layers a graphene sheet can be rolled more than one way, producing different types of carbon Nanotubes. And thus Carbon Nanotubes can be categorized by their structures.

#### **3.5.1 Single-wall carbon nanotubes (SWCNTs):**

SWCNTs consist of a single cylindrical carbon layer with a diameter in the range of 0.4-2 nm, depending on the temperature at which they have been synthesized. It was found that the higher the growth temperature larger is the diameter of CNTs (E.N.Ganesh, 2015).

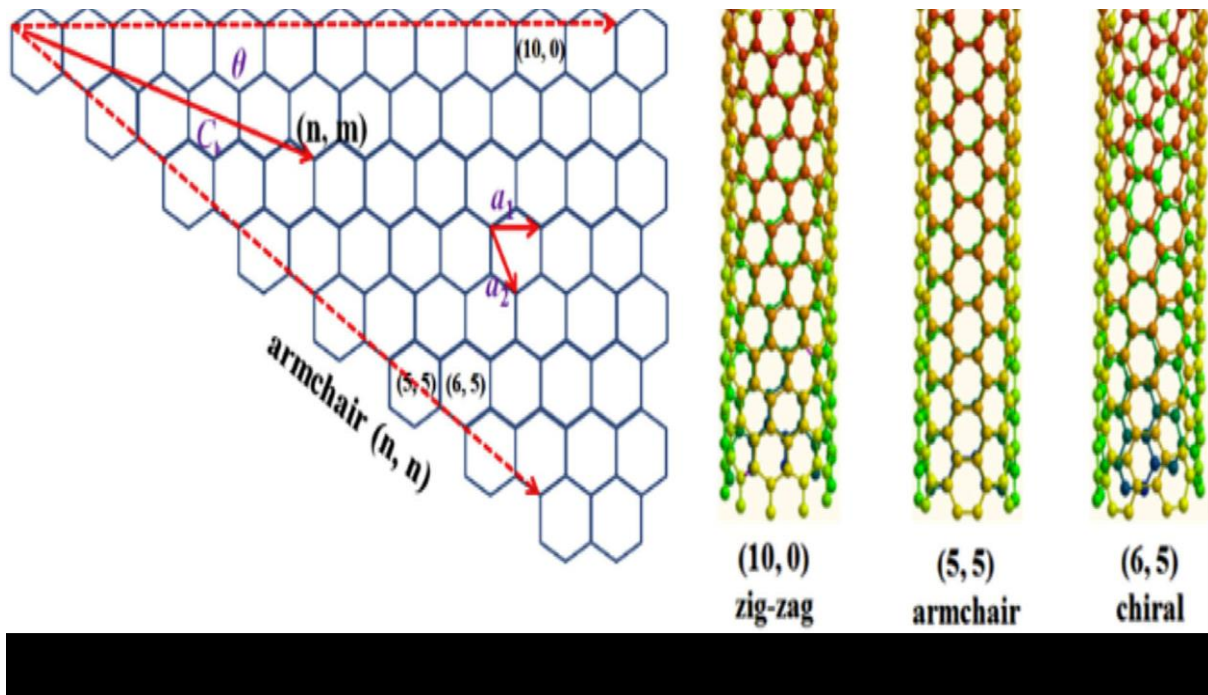


**Fig (3.1) Single Walled Carbon Nanotubes Structure.**

SWCNTs exist in three unique geometries, which are: arm chair, zig- zag and chiral.

The structure of a SWNT can be conceptualized by wrapping a one-atom-thick layer of graphite called graphene into a seamless cylinder. The way the graphene sheet is wrapped is represented by a pair of indices  $(n, m)$  called the chiral vector. The integer's  $n$  and  $m$  denote the number of unit vectors along two directions in the honeycomb crystal lattice of graphene. If  $m = 0$ , the Nanotubes are called zigzag, which is named for the pattern of hexagons as we move on circumference of the tube. If  $n = m$ , the nanotubes are called "armchair", which describes one of the two conformers of cyclohexene a hexagon of carbon atoms. Otherwise, they are called "chiral", in which the  $m$  value lies between zigzag and armchair structures. The word chiral means handedness and it indicates that the tubes may twist in either direction. The mechanical, electrical, optical, and other properties of CNTs are determined by the chirality. It has been reported that the electrical properties of CNTs are influenced by the chiral vector and corresponding pairs of integers. Depending on rolling up of the graphene sheet.

SWCNTs have an ultra-high surface area as large as 1300 m<sup>2</sup>/g, which renders sufficient space for drug loading and bio conjugation. SWCNTs are known to be more efficient than MWCNTs. This is due to the reason that SWCNTs have ultra-high surface area (Zainab Najaf et al 2019).



**Fig (3.2) Geometries of SWCNTs.**

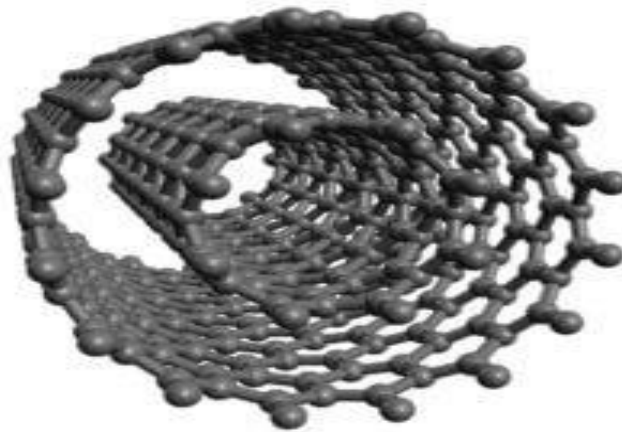
### 3.5.2 Multiple walled carbon nanotubes (MWCNTs):

MWCNTs consist of several coaxial cylinders, each made of a single graphene sheet surrounding a hollow core. The outer diameter of MWCNTs ranges from 2-100 nm, while the inner diameter is in the range of 1-3 nm, and their length is one to several micrometres.

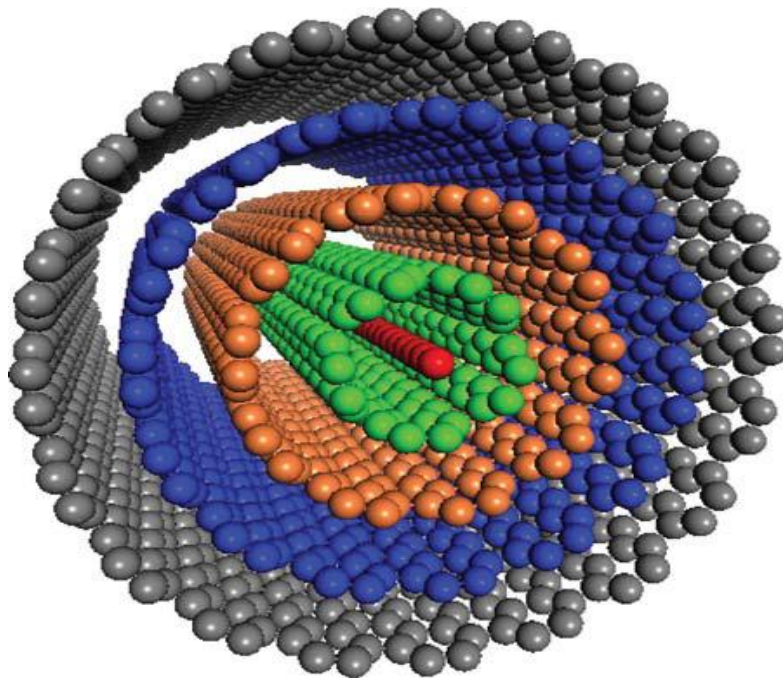
The interlayer space is approximately equal to the space between the graphene sheets in graphite. Decoration of multiwall carbon nanotubes (MWCNTs) consists of depositing nanoparticles on its walls or ends, bonded by physical interaction. It can be used in potential applications in catalysis, biosensors, biomedical, magnetic data storage, and electronic devices (E.N.Ganesh, 2015).

The special place of double-walled carbon nanotubes (DWNT) must be emphasized here because their morphology and properties are similar to SWNT but their resistance to chemicals is significantly improved. This is especially important when functionalization is required (this means grafting of chemical functions at the surface of the nanotubes) to add

new properties to the CNT. In the case of SWNT, covalent Functionalization will break some C=C double bonds, leaving "holes" in the structure on the nanotube and thus modifying both its mechanical and electrical properties. In the case of DWNT, only the outer wall is modified. DWNT synthesis on the gram-scale was first proposed in 2003 by the CVD technique, from the selective reduction of oxide solutions in methane and hydrogen. (Rajaashree 2009).



**Fig(3.3) Double Walled Carbon Nanotubes Structure (DWCNTs).**



**Fig(3.4) Multiple Walled Carbon Nanotubes Structure (MWCNTs).**

<b>SWNT</b>	<b>MWNT</b>
Single layer of graphene.	Multiple layer of grapheme
Catalyst is required for synthesis.	Can be produced without catalyst
A chance of defect is more during Functionalization.	A chance of defect is less but once occurred it's difficult to Improve.
It can be easily twisted and are more pliable.	It can not be easily twisted.
Easy assessment and characterization	Structure is complicated
Poor purity	High purity
Difficult bulk synthesis due to the requirement of appropriate growth and atmospheric condition.	Easy bulk synthesis.

**Table (3.1) comparison between SWCNTs and MWCNTs**



### **3.5.3 Others types of CNTs:**

#### **Nanotours:**

A nanotorus is theoretically described as carbon Nano tube bent into a torus (doughnut shape). Nanotori are predicted to have many unique properties, such as magnetic moments 1000 times larger than previously expected for certain specific radii. Properties such as magnetic moment, thermal stability etc. varies widely depending on radius of the torus and radius of the tube. Nano-torus particles are promising in nano-photonics applications.

#### **Polymerized SWNT:**

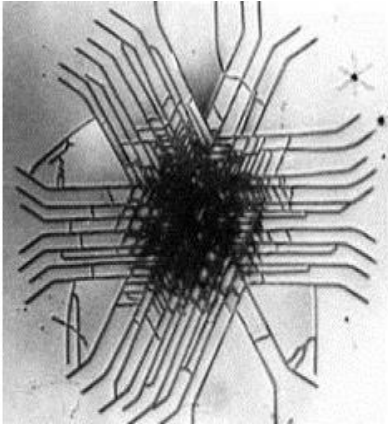
These are the solid-state manifestation of fullerenes and related compounds and materials. Many single walled nanotubes intertwine to form polymerized SWNTs, which are comparable to diamond in terms of hardness. (Rajesh Purohit et al, 2014).

#### **Nanobuds:**

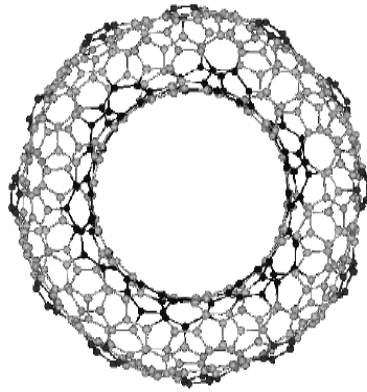
Carbon Nanobuds are a newly discovered material combining two previously discovered allotropes of carbon: carbon nanotubes and fullerenes. In this new material fullerene-like "buds" are covalently bonded to the outer sidewalls of the underlying carbon nanotube.

#### **Nano horn:**

They were first reported by Harris et al and Iijima, Single-walled carbon nano horns (SWCNHs) are horn-shaped single walled tubules with a conical tip t no catalyst is required for synthesis so high purity materials can be produced. Their high surface area and excellent electronic properties have led to use as electrode material for energy storage. Currently, SWCNHs have been widely studied for various applications, such as gas storage, adsorption, and catalyst support. (Kalpna Varshney, 2014)



**a- Polymers**



**b- nanotorus**



**c- nanobuds**



**d- carbon nano horns**

**Fig (3.5) other types of CNTs.**

### **3.5.4 Carbon nano powder :**

Carbon (C) Nanoparticles, nanodots or nanopowder are black spherical high surface area graphitic carbon . ... Surface functionalized nanoparticles allow for the particles to be preferentially adsorbed at the surface interface using chemically bound polymers.

Nanopowders can be defined as powdered materials with individual particles in nanometer scale or materials with crystalline in nanometer scale. Nanoparticles (NPs) are made up of a large amount of atoms or molecules bonded with each other with a total size varying from 1 nm to around 100 nm. Due to their very small sizes, NPs possess an extraordinarily high surface area-to-volume ratio, which changes their physical-chemical properties compared to their macroscale counterparts.

As the size decreases to nanoscale, the properties of atoms present on the outer boundary of particles become dominant. The optical properties change significantly in comparison with those of the bulk due to their varied size-dependent interactions with light. For example, plasmons are generated by the coherent oscillations of conduction-electrons in response to incident light. Once such oscillations are confined to a metal–dielectric interface, or light interacts with NPs whose sizes are smaller than the excitation wavelength, a local charge oscillation nearby the interface or around the particle is produced in resonance with the light frequency, which is well known as localized surface plasmon resonance . This capability of restricting the light at a nanoscale dimension provides NPs with tremendous unique features, including large electromagnetic field enhancements, high photothermal conversion efficiency, and rich spectral responses .

Furthermore, due to quantum-sized confinement, the electrons in the conduction band and the holes in the valence band lose certain freedoms. For instance, they are able to be confined into 2D (quantum well), 1D (quantum wire), and 0D (quantum dot), which are termed low-dimension structures . The energy levels of these systems become discrete and quantized, as

well as the electron-hole transitions . This causes the fundamental differences of the electrical properties between micro- or bulk materials and nano\_structured materials.

Nanoscale materials also alter the mechanical properties. For instance, nano\_crystalline materials may exhibit distinguished mechanical characters, including increased hardness and toughness, enhanced thermal expansion coefficient and diffusivity, reduced elastic modulus and ductility, and superior soft magnetic properties in comparison with conventional polycrystalline materials .

Nano materials not only provide us with opportunities to interpret the nature of solid interfaces and to expand our understanding of the structure–property relationship down to the nanoscale regime, but also introduce attractive potentials for scientific and technology applications. NPs are successfully gaining interest due to their worth in a wide range of applications, such as superlenses, surface-enhanced spectroscopy and microscopy, nanoscale lasing, quantum computing, plasmon-assisted photolithography, photocatalysis, light harvesting, biochemical sensing, and organic solar cell The enthusiasm for utilization of nanomaterials is sweeping the planet, generating several billion dollars of investment annually in research and development . Brad Herring (2018)

### **3.6 Carbon Nanomaterial GROWTH METHODS:**

There are various techniques used for growth of CNTs. Three popular methods are arc discharge, chemical vapor deposition (CVD), laser ablation the common characteristic of these techniques is to provide energy to a carbon source for the creation of Carbon atoms that generate CNTs. The energy source is current in an arc discharge, the high-intensity light from a laser in the laser ablation & heat from a furnace in CVD.

#### **3.6.1 Arc Discharge Method:**

The arc discharge method is a well-known method for the formation of CNTs. In this method, a buffer gas such as helium is introduced in a chamber containing a cathode, a graphite anode, and vaporized carbon

Molecules. The chamber also contains a small amount of metal catalysts such as nickel, cobalt or iron. Under applied pressure, the chamber is heated to 4000 K, and a direct current (DC) current is passed through the sample. In the progression of this technique, nearly half of the vaporized carbon solidifies on the tip of cathode in the form of a “hard cylindrical deposit”. Condensation of the remaining carbon occurs, forming “cathode soot” on the cathode, and “chamber soot”, which is present all over the

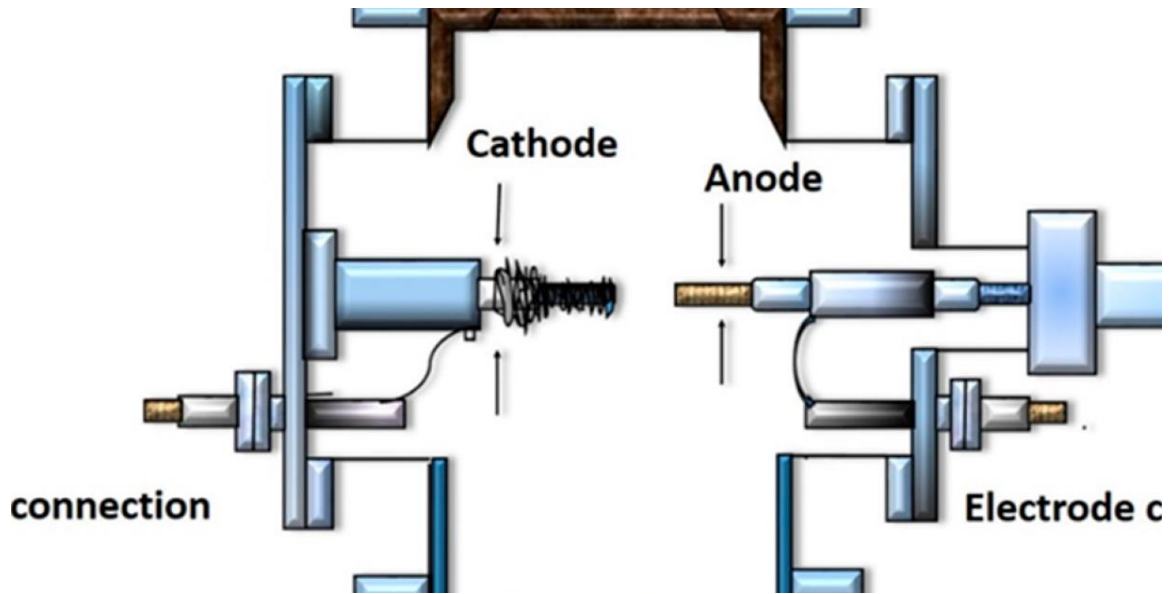
Walls of the chamber. The chamber and the cathode soot yield either SWCNTs or MWCNTs.

The selection of the inert gas and the added metallic catalyst decides whether the resultant CNTs are SWCNTs or MWCNTs. For the production of CNTs, catalyst such as Fe, Co, Ni, Y, or Mo. are necessary for growth in the arc discharge,

The advantage of this method is the high yield of nanotubes.

However, the little control over the orientation of the nanotubes is the key disadvantage that ultimately affect their activity.

The products need to be purified later on. Methods such as centrifugation, oxidation, filtration, and acidic treatment are being used for the purification (Ahmad Fauzi Ismail, et al, 2013 ).



**Fig(3.6). Schematic diagram showing Arc discharge method.**

### **3.6.2 Laser Ablation Method:**

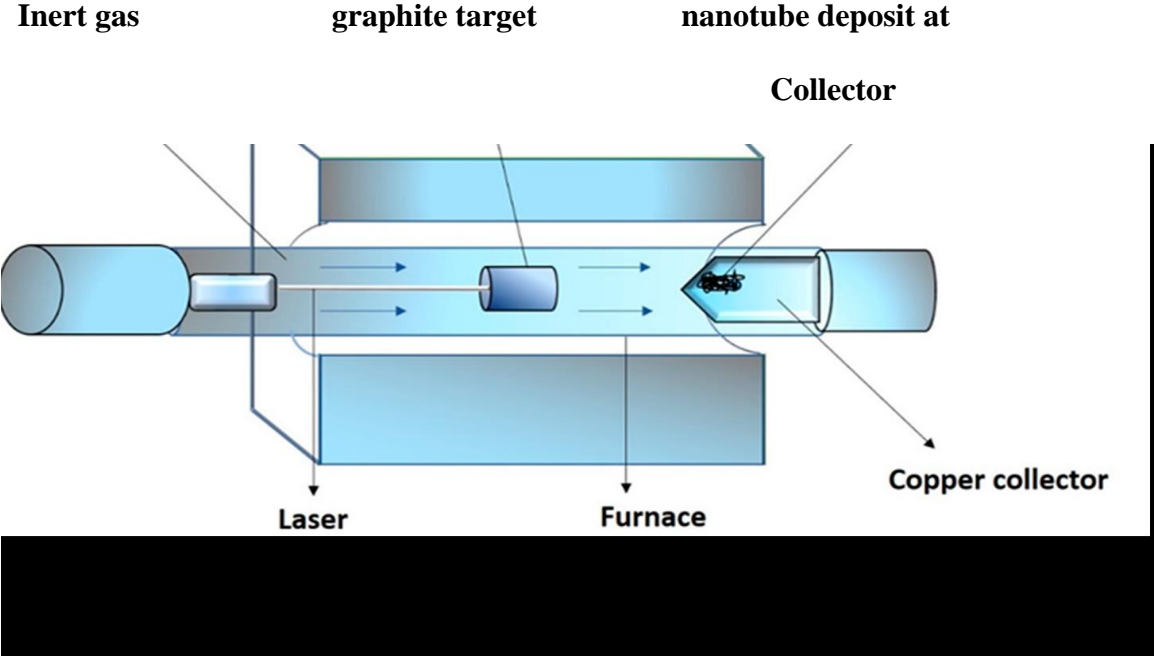
The laser ablation technique and the arc discharge method are similar in principles and mechanisms; however, they are different by the input energy sources. In the laser ablation method, the required energy is provided by a laser. A tube made up of a quartz-containing graphite block is heated in a furnace at 1200°C using a high-power laser in the presence of metal particles as catalysts. This method utilizes an intense laser pulse or a continuous laser to ablate a Co-Ni/graphite composite target. The target is placed in a tube furnace. When this target vaporizes, and catalyst vapors occur, then cloud condenses, and the small amount of carbon species come together to form a larger one. The vaporized catalysts prevent the closing of these carbon molecules into cage structure, so the growth process is finished a stream of argon is away the vaporized carbon, which condenses downstream onto the cooler walls of the quartz, SWCNTs and metallic particles are present in this condensation. According to studies, the laser power can influence the diameter of the CNTs. The diameter of the tube becomes thinner as the laser pulse power is increased. Other studies reported that ultrafast laser pulses are of great potential, and are capable to produce larger quantities of SWCNTs. The SWCNTs produced by this method are of high purity and quality.

The main advantages of this technique include relatively low metallic impurities, and a relatively high yield, due to the vaporization tendency of the metallic atoms from the end of the tube, once it is closed.

The key drawback of this technique is that the synthesized nanotubes may not be regularly straight, and have some degree of branching.

The length of MWNT produced through laser ablation is much shorter than that produced by arc discharge method. Therefore, this method does not seem adequate for the synthesis of MWCNTs.

Furthermore, this procedure involves graphite rods of high purity, and requires high powers of lasers, and the amount of CNTs produced are not as great as in the arc-discharge technique (Eatemadi, 2014).



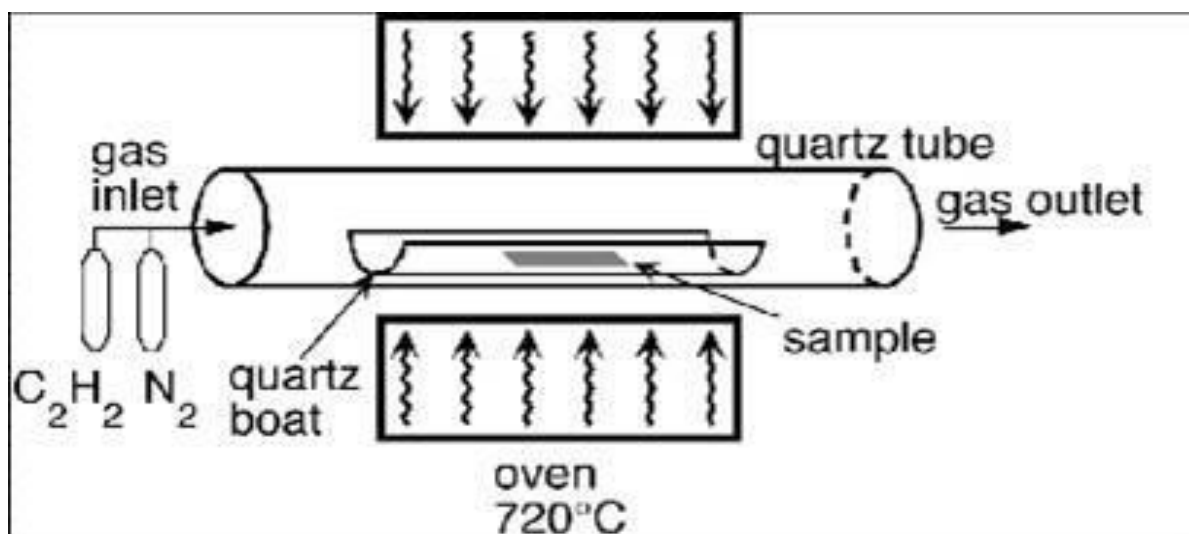
**Fig (3.7): Schematic structure showing the laser ablation method.**



### **3.6.3 Chemical Vapor Deposition (CVD) Method:**

Chemical vapour deposition (CVD) is the best technique to synthesize CNTs. In this method, the decomposition of the carbon precursor & CNT formation take place on the surface of catalyst particles. Temperatures and their size can be controlled by varying the size of catalyst particles. CVD is used for the large scale production of CNTs. It is achieved by taking a carbon source in the gas phase & using an energy source, such a resistively heated coil, to impart energy to a gaseous carbon molecule. Commonly used carbon Sources include methane, carbon monoxide, acetylene etc. This will result in the formation of CNTs, if the proper parameters are maintained. The CNT synthesis using CVD is essentially a two steps process. A catalyst preparation step is the first step Followed by actual synthesis of nanotube. The catalyst is generally prepared by sputtering, physical vapour deposition (PVD), dip coating etc. The next step is heating up the substrate in a carbon rich gaseous environment. Temperature for the synthesis of nanotubes in this technique is generally 500 to 1000 C.

When compared with the previous two methods, CVD is a simple and economic technique for synthesizing CNTs at relatively low temperature and ambient pressure, but at the cost of crystallinity. It is a versatile process as it harnesses a variety of hydrocarbons in any state (solid, liquid or gas), enables the use of various substrates and allows CNT growths in a variety of forms, such as powder, thin or thick films, aligned or entangled, straight or coiled, or even a desired architecture of nanotubes at predefined sites on a patterned substrate. It also offers better control over growth parameters.



**Fig (3.8): Schematic diagram of thermal CVD method.**

Figure shows a schematic diagram of the setup used for CNT growth by CVD in its simplest form. The process involves passing a hydrocarbon vapor (typically for 15-60 minutes) through a tube furnace in which a catalyst material is present at sufficiently high temperature ( $600-1200^\circ C$ ) to decompose the hydrocarbon. CNTs grow over the catalyst and are collected when the system is cooled to room temperature. When a liquid hydrocarbon (benzene, alcohol, etc.) is being employed, it is heated in a flask and an inert gas is purged through it to carry the vapor into the reaction furnace. The design of the CVD reactor depends on whether the carbon precursors are liquid or gas. Liquid carbon precursors often use a bubbler to vaporize the reactants, and a carrier gas (reactive gases such as  $H_2$  or inert gas such as  $N_2$  or Ar) to transport the vaporized reactants into the CVD reactor. The three main parameters which affect CNT growth in CVD are the hydrocarbon, catalyst and growth temperature. Generally, low-temperature CVD ( $600-900^\circ C$ ) yields MWNTs, whereas a higher temperature ( $900-1200^\circ C$ ) reaction favors SWNT growth, indicating that SWNTs have a higher energy of formation, probably owing to their small diameters, which results in high curvature and high

strain energy. The catalyst particle size has been found to dictate the nanotube diameter. Hence, metal nanoparticles of controlled size can be used to grow CNTs of controlled diameter. Thin films of catalyst coated onto various substrates have also been proved successful in achieving uniform CNT deposits. In addition, the material, morphology and textural properties of the substrate greatly affect the yield and quality of the resulting CNTs (vivikpatel, 2014).

#### **3.6.4 Flame Synthesis Method:**

The potential of the flames for synthesis of elongated carbon nano forms was demonstrated long before carbon nanotubes were discovered by Iijima in the process of vaporization of graphite. The formation of elongated filamentous carbon on the surface of a solid support inserted in a diffusion flame was reported in very early flame studies.

Flames offer the potential for the fabrication of CNTs in large amounts at considerably lower cost, as compared to the existing methods. It is capable of producing nanotubes of carbon on required surfaces, and specifically in a controllable way. The catalytic precursors, in the flame synthesis method are usually introduced into the flame system, where they undergo nucleation and finally condense into solid spherical metallic nanoparticles. Flame parameters can also be applied to obtain suitable flame conditions, which would enable the fabrication of ideal sizes of catalyst particles for the growth and inception of CNTs. The catalyst properties and alteration of the flame parameters can remarkably influence the heat and carbon source, the activation and deactivation of catalyst particles, and the formation of catalysts, as well as the morphology of the final synthesized products (Yuan et al, 2018).

### **Growth controlling parameters:**

There are the three major factors that determine the optimum region for carbon nanotube growth inside a flame which are gas phase composition, temperature and the catalyst. Careful control of these variables can result in a high yield rate of pure carbon nanotubes when compared to other synthesis methods.

### **Gas phase composition inside a flame**

Carbon nanotubes are formed when carbon in gaseous form is deposited in form of the structured solid on to a catalyst particle. The concentration of gaseous precursors and the resulting deposition rate play an important role in determining the structure of the nanotube that is dependent on the concentration of gaseous precursor. These gaseous precursors are formed through the complex phenomena that occur inside a flame.

### **Temperature**

Temperature is one of the important parameter that governs the growth of carbon nanotubes inside a flame. Evolution of temperature field and gas phase chemistry occurs simultaneously inside a flame, because of the coupled energy and mass transfer phenomena. Flame environment provides an inherent source of heat and thus high temperature which makes it one of the ideal candidates for CNT growth. However, the temperature field inside the flame shows a large variation ranging from ~2000 K near the flame front to the cooler regions of ~ 800 K. Thus, appropriate regions of the flame need to be probed for the growth of carbon nanotubes.

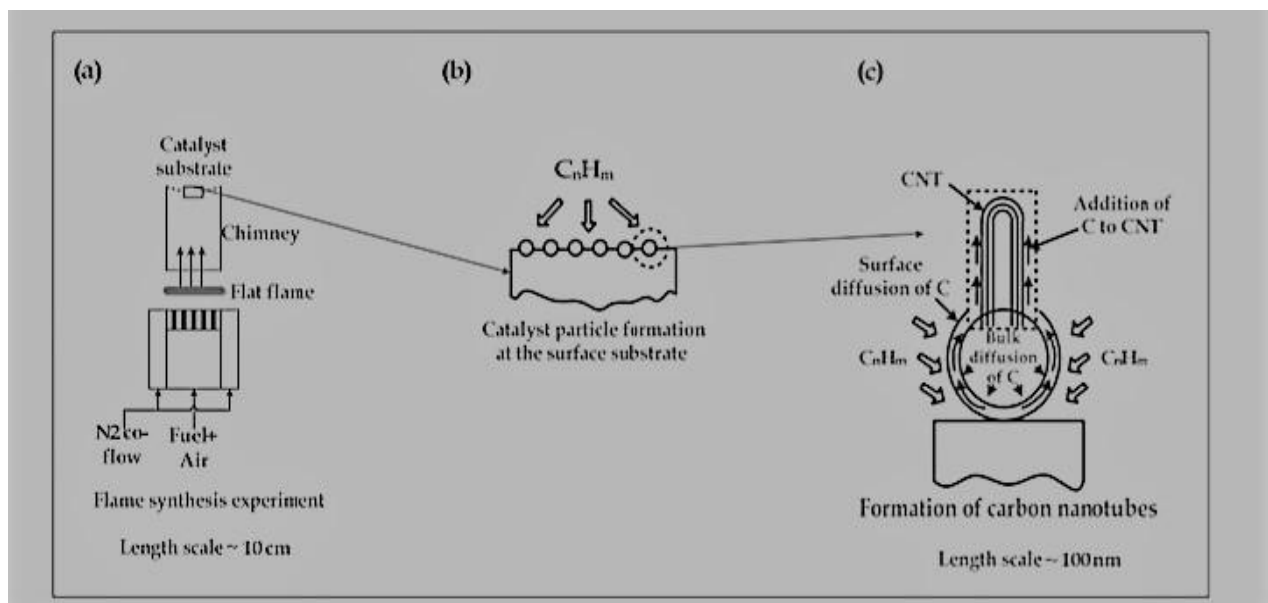
### **Catalyst**

Mainly, transition metals such as Iron (Fe), Nickel (Ni), Cobalt (Co) have been employed for CNT synthesis in a flame. Alloys of these metals with metals like Copper (Cu) and Zinc (Zn) have also been used. Properties of these catalyst materials are unique which allow to produce both types of carbon nanotubes (SWCNTs, MWCNTs).

**Growth of CNTs in flames:**

In a flame, fuel (generally hydrocarbons such as methane (CH<sub>4</sub>), ethylene (C<sub>2</sub>H<sub>4</sub>), and Acetylene (C<sub>2</sub>H<sub>2</sub>) etc.) reacts with oxidizer (O<sub>2</sub> from air) to produce gaseous mixture that includes carbon dioxide (CO<sub>2</sub>), water vapor (H<sub>2</sub>O), carbon monoxide (CO), hydrogen (H<sub>2</sub>), saturated and unsaturated hydrocarbons (C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub> etc.) and radicals. Hydrocarbons and carbon monoxide constitute the gaseous precursor mixture that is the source of solid carbon deposited on catalyst particles to form carbon nanostructures. Metal catalysts, inserted in the flame either in the form of a substrate coating or as aerosol particles, provide the necessary reaction sites for deposition of solid carbon. The structure of the formed carbon nanotube (MWNTs and/or SWNT) depends on the catalyst particle size and carbon deposition rate. Post flame gas phase chemistry, temperature at the surface of the catalyst particle and the structure and type of catalyst particle are the key controlling parameters for growth of nanotubes in the flame synthesis process as mentioned above.

The figure below describe Formation of carbon nanotubes in a flame environment The growth process can be broken down into following steps, (a) establishment of the flame that acts as the source for gaseous precursors and heat (b) simultaneous formation of catalyst particles and growth of carbon nanotube occurring at nano-scale, and (c) Diffusion of gaseous carbon species to the catalyst particle followed by the deposition of carbon is usually assumed to occur at the leading face of the particle ( jay p.gore , anube sane, 2016).



**Fig (3.9): flame synthesis method.**

### 3.7 Properties of carbon nanotube:

#### 3.7.1 Mechanical Properties

Carbon nanotubes are the strongest and stiffest materials yet discovered in terms of tensile strength and elastic modulus respectively. This strength results from the covalent  $sp^2$  bonds formed between the individual carbon atoms. Because of C-C bonds, CNTs are expected to be extremely strong along their axes and have a very large Young's modulus in their axial direction. The Young modulus value of a SWNT is estimated as high as 1Tpa to 1.8 Tpa.

A single perfect nanotube is about 10 to 100 times stronger than steel per unit weight. The Young's modulus of the best nanotubes can be as high as 1000 GPa which is approximately 5x higher than steel. The tensile strength, or breaking strain of nanotubes can be up to 63 GPa, around 50x higher than steel. These properties, coupled with the lightness of carbon nanotubes.

Materials	Young's module(GPa)	Tensile strength(GPa)
SWCNT	1054	150
MWCNT	1200	150
Steel	208	0.4
Wood	16	0.008

**Table (3.2) Comparison of Mechanical Properties of CNTs with other string material**

### **3.7.2 Thermal Properties:**

All nanotubes are expected to be very good thermal conductors along the tube, exhibiting a property known as "ballistic conduction," but good insulators laterally to the tube axis. It is predicted that carbon nanotubes will be able to transmit up to 6000 W at room temperature; compare this to copper, a metal well-known for its good thermal conductivity, which transmits 385 W.

The temperature stability of carbon nanotubes is estimated to be up to 2800 °C in vacuum and about 750 °C in air. Thermal expansion of CNTs will be largely isotropic, which is different than conventional graphite fibers, which are strongly anisotropic. This may be beneficial for carbon-carbon composites. It is expected that low-defect CNTs will have very low coefficients of thermal expansion (H. Dai et al, 2014).

### **3.7.3 Electrical Properties**

The electronic properties of carbon nanotubes are also extraordinary. Especially notable is the fact that nanotubes can be metallic or semiconducting depending on their structure. Thus, some nanotubes have conductivities higher than that of copper, while others behave more like silicon. There is great interest in the possibility of constructing Nano scale electronic devices from nanotubes, and some progress is being made in this area.

### **3.7.4 Kinetic properties**

Multi-walled nanotubes, multiple concentric nanotubes precisely nested within one another, exhibit a striking telescoping property whereby an inner nanotube core may slide, almost without friction, within its outer nanotube shell thus creating an atomically perfect linear or rotational bearing.



### **3.7.5 Defects**

As with any material, the existence of defects affects the material properties. Defects can occur in the form of atomic vacancies. A single monoatomic vacancy induces magnetic properties. High levels of such defects can lower the tensile strength. Another form of defect that may occur in carbon nanotubes is known as the Stone Wales defect, which creates a pentagon and heptagon pair by rearrangement of the bonds, which reduces the tensile strength. The nanotube's electrical properties are also affected by the presence of defects. A common result is the lowered conductivity through the defective region of the tube. The tube's thermal properties are also heavily affected by the defects

### **3.7.6 Optical Properties:**

Optical properties of SWNT are related to their quasi one- dimensional nature. Theoretical studies have revealed that the optical activity of chiral nanotubes disappears if the nanotubes become larger therefore, it is expected that other physical properties are influenced by these parameters too. Use of the optical activity might result in optical devices in which CNTs play an important role (vivikpatel, 2014).

### **3.8 Purification of carbon nanotube:**

Purification of CNTs generally refers to the separation of CNTs from other entities, such as carbon nanoparticles, amorphous carbon, residual catalyst, and other unwanted species. A variety of techniques have been used to synthesize CNTs including electric-arc discharge, laser vaporization, and chemical vapour deposition (CVD). Most approaches in general, produce powders containing not only CNTs but also other carbonaceous particles such as amorphous carbon, fullerenes, nano crystalline graphite, and metals that were introduced as catalysts during the synthesis.

Generally, to provide the active catalyst with the desired stability, powdered catalysts are used for CNT growth which are supported on hard-to-reduce oxides (i.e., alumina, silica, and titanium) to minimize the metal particle size.

These supported catalysts have been successfully used in the large-scale production of CNTs. As the supports are very difficult to be removed, aggressive treatments are required to produce pristine CNTs but may cause damages to the CNTs' yield and structure. This problem sometimes hinders an accurate analysis of the CNT characteristics and limits the best performance of CNTs for new applications. However, most of the advanced technological applications of CNTs mainly depend on the purity of the materials. Overall, as-prepared materials contain a variety of impurities, such as amorphous carbon, metallic catalyst, and multi-walled graphite leading to an impediment to more detailed characterization of the properties of CNTs and their future applications in industries where some applications require localized and aligned distributions of nanotubes, while some require pristine-structured nanotubes.

The types of impurities and their amount present in CNTs depend strongly on the synthesis methods, types of catalyst and carbon source, the stability and morphology of the CNTs.

Purification procedures are chemical and physical methods, both of methods are depending on nanotube morphologies (single-walled or multi-walled), growth processes, and metal catalysts (A. Fonseca, 2017).

### **3.8.1 Chemical methods:**

The chemical methods separate the synthesis products based on their reactivity which normally introduce unavoidable defects along the tubes and the pentagonal structure at the tube ends, causing remarkable damages to the structure and morphology of the CNTs. However, with proper control of the reaction conditions, purification of CNTs through the removal of metal catalyst particles may result in higher purity as well as the tips opening. Acids and oxidizing agents, alkali treatment and annealing in inert gases are some examples of chemical purification methods.

Oxidants that are commonly used in the treatment include nitric acid, sulphuric acid potassium permanganate ( $\text{KMnO}_4$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), the extent of oxidation depends on the redox potential of the system and also on the structural features of the nanotubes.

### **3.8.2 Physical methods:**

On the other hand, the physical methods, such as ultra-sonication, filtration, centrifugation, and size-exclusive chromatography, separate the impurities based on their size. These processes are relatively mild and do not cause severe damages to the tubes, but they are normally more complex and less effective. In general, physical methods are applied to separate and remove the undesirable impurities such as nano capsules, aggregate and amorphous carbon. The physical is more complex and less effective compared to the chemical methods, thus leading to a lower purity of CNTs.

### **3.6.3 Multi-Step Purification**

Purification of CNTs can be carried out by combining the chemical treatment and physical separation in a multi-step procedure in order to effectively remove the amorphous carbon, metal particles, and multi-shell carbon nano capsules. Multi-step purification is necessary particularly when a single treatment is not sufficient to simultaneously remove all the impurities that are present in the CNTs (Tee Madzlan Aziz, 2018).

### **3.9 Characterization of carbon nanotubes:**

Carbon nanotubes are have very small dimension which make their characterization complicated and require sophisticated instruments, general CNTs characterize by tow techniques, electron microscopic, Diffraction, spectroscopy.

Spectroscopy, it often used to more deeply investigate the morphology and the structure, such as X-ray scattering, often used to more deeply investigate the morphology and the structure.

#### **Electron Microscopy (EM)**

Electron microscopy is an essential tool for characterizing any nanomaterial because it allows direct observation of size, shape, structure. The local structure of the CNTs can be investigated at the nanometre and sub nanometre level by Transmission Electron Microscopy (TEM), and the scanning electron microscopy (SEM).

E M was developed due to the limitations of Light Microscopy which is limited by the physics of light to 500 or 1,000 times magnification and a resolution of 0.2 micrometres. In the early of the 1930 decade, this theoretical limit had been reached and there was

A scientific desire to see the fine details of the interior structures of organic cells. E M is a scientific technique used to examine objects on a very fine scale. This thin specimen is irradiated with an electron beam of uniform current density.

This required 10,000 times magnification which was not possible to be obtained using Light Microscopes.

Electron Microscopes operate exactly as their optical counterpart except that they use a focused beam of electrons instead of light to "image" the specimen and gain information about its structure and composition.

### **3.9.1 Scanning electron microscopy (SEM)**

Scanning Electron Microscope functions exactly as their optical counterparts except that they use a focused beam of electrons instead of light to “image” the specimen and gain information as to its structure and composition. Given sufficient light, the unaided human eye can distinguish two points 0.2 mm apart. If the points are closer together, they will appear as a single point. This distance is called the resolving power or resolution of the eye. Similarly, light microscopes use visible light (400- 700nm) and transparent lenses to see objects as small as about one micrometres (one millionth of a meter), such as a red blood cell (7  $\mu\text{m}$ ) or a human hair (100  $\mu\text{m}$ ). Light microscope has a magnification of about 1000x and enables the eye to resolve objects separated by 200 nm. Electron Microscopes were developed due to the limitations of light microscopes, which are limited by the Physics of light.

Electron Microscopes are capable of much higher magnifications and have a greater resolving power than a light microscope, allowing it to see much smaller objects at sub cellular, molecular and atomic level. The smallest the wavelength of the illuminating sources is the best resolution of the microscope.(Hongbao Ma, 2016)

#### **3.9.1.1 Components of SEM**

##### **Electron Column**

The electron column is where the electron beam is generated under vacuum, focused to a small diameter, and scanned across the surface of a specimen by electromagnetic deflection coils. The lower portion of the column is called the Specimen chamber.

##### **Electron gun:**

An electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost.

Other types of electron emitters include lanthanum hexaboride cathodes, and field emission guns (FEG), which may be of the cold-cathode type using tungsten single crystal emitters or the thermally assisted Schottky type, using emitters of zirconium oxide.

### **Condenser Lenses:**

After the beam passes the anode it is influenced by two condenser lenses that cause the beam to converge and pass through a focal point. In conjunction with the selected accelerating voltage the condenser lenses are primarily responsible for determining the intensity of the electron beam when it strikes the specimen.

### **Apertures:**

The function of these apertures is to reduce and exclude extraneous electrons in the lenses. The final lens aperture located below the scanning coils determines the diameter or spot size of the beam at the specimen. The spot size on the specimen will in part determine the resolution and depth of field. Decreasing the spot size will allow for an increase in resolution and depth of field with a loss of brightness.

### **Scanning System:**

Images are formed by rastering the electron beam across the specimen using deflection coils inside the objective lens. The stigmator or astigmatism corrector is located in the objective lens and uses a magnetic field in order to reduce aberrations of the electron beam. The electron beam should have a circular cross section when it strikes the specimen however it is usually elliptical thus the stigmator acts to control this problem.

### **Specimen Chamber:**

The lower portion of the column is specimen stage and controls are located. Specimens are mounted and secured onto the stage which is controlled by a goniometer. The secondary electrons from the specimen are attracted to the detector by a positive charge. Manual stage controls are found on the front side of the specimen chamber for x-y-z movement.

### **Electron Detectors:**

Detectors collect the signal generated from interaction of beam with specimen. Electronic detectors convert the signal into digital images and most often collected signal are Secondary electrons by secondary electron detector.

#### **3.9.1.2 How Scanning Electron Microscope (SEM) works:**

- The electron gun produces an electron beam when tungsten wire is heated by current and accelerated by the anode.
- The beam travels in the vacuum column through electromagnetic fields and lenses, which focus the beam down toward the sample.
- A mechanism of deflection coils enables to guide the beam so that it scans the surface of the sample in a raster pattern.
- When the incident beam touches the surface of the sample and produces signals
- The emitted signals are trapped by electrical detectors, convert into digital images and displayed on a screen as digital image.
- Provides information sample's elemental composition, structural variation and morphology.
- In the SEM, use much lower accelerating voltages to prevent beam penetration into the sample since the requirement is generation of the secondary electrons from the true surface structure of a sample. Therefore, it is common to use low KV, in the range 1-5kV for biological samples, even though the SEMs are capable of up to 30 kV(Dr. M. Kannan ,2017).

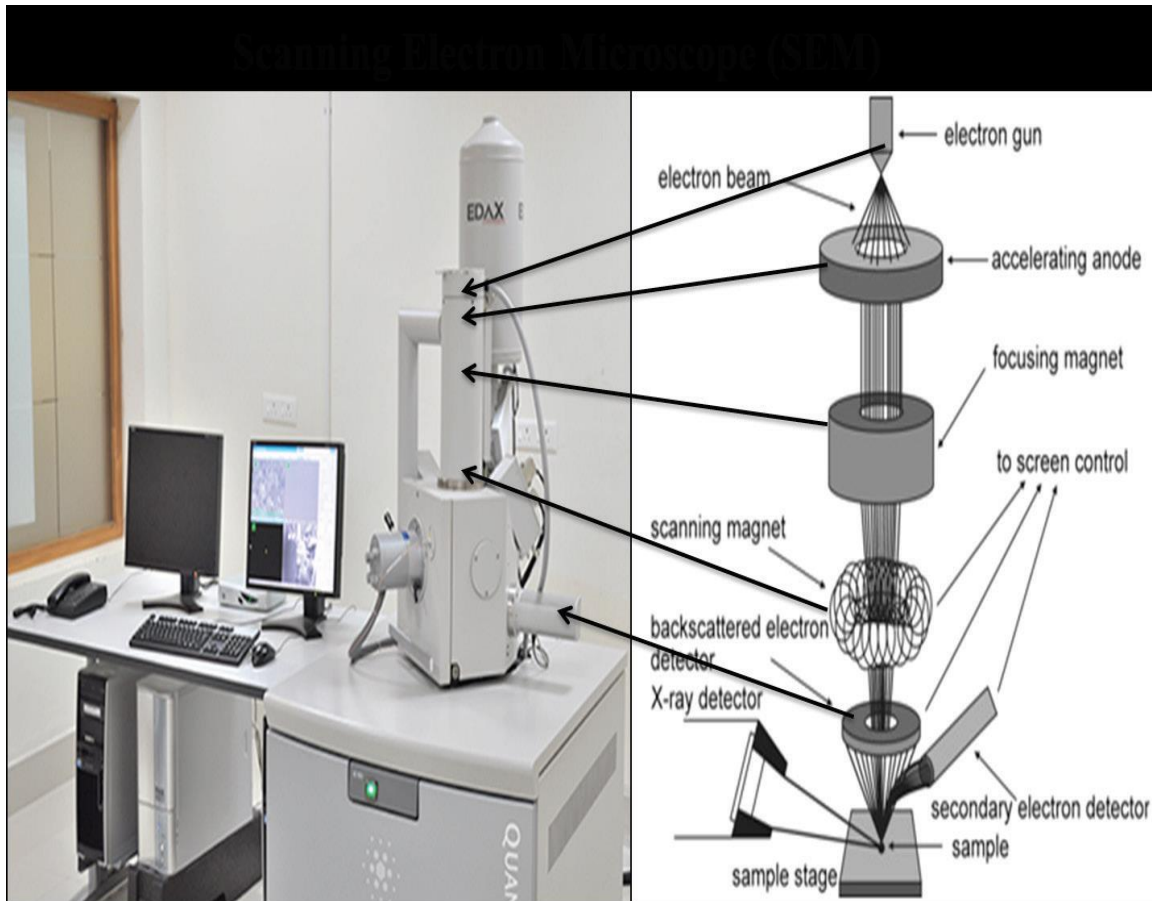


### **3.9.1.3 Advantages of SEM**

- It gives detailed 3D and topographical imaging and the versatile
- Information garnered from different detectors.
- This instrument works very fast.
- Modern SEMs allow for the generation of data in digital form.
- Most SEM samples require minimal preparation actions.
- SEMs carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.

### **3.9.1.4 Disadvantages of SEM**

- SEMs are limited to solid samples.
- SEMs are expensive and large.
- Special training is required to operate an SEM.
- The preparation of samples can result in artifacts.



**Fig (3.10): Schematic diagram of SEM.**

### **3.10 Transmission electron microscopy (TEM):**

Transmission electron microscopy (TEM) is the original form of electron microscopy and analogues to the optical microscope. It can achieve a resolution of ~0.1 nm, thousand times better resolution, cannot be reached by the light microscope. The beam of electrons passes through the specimen and analyzes the internal structure of the specimen in the form of images. The electron has the poor penetrating capability and gets absorbed in the thick specimen. Therefore, the thickness of the specimen should not be more than few hundred Angstroms (one angstrom =  $10^{-10}$  m) However sometimes, slightly thickens samples are used in high voltage electron microscope.

The amount and scale of the information which can be extracted by TEM depends critically on four parameters; the resolving power of the microscope (usually smaller than 0.3 nm); the energy spread of the electron beam (often several eV); the thickness of the specimen (almost always significantly less than 1  $\mu\text{m}$ ), and; the composition and stability of the specimen. The first and second of these depend largely on the depth of your pocket – the more you spend, the better the microscope parameters. The third is usually determined by your experimental skill while the last depends on luck or your choice of experimental system.

### **3.9.2.1 Components of TEM**

#### **Electron gun**

The source of electrons, the cathode, is a heated a sharply pointed rod shaped Lanthanum hexaboride. The filament is surrounded by a control grid called as Wehnelt cylinder, with a central aperture arranged on the axis of the column; the Apex of the cathode is arranged to lie at or just above or below this aperture.

The cathode and control grid are at a negative potential equal to the desired accelerating Voltage and are insulated from the rest of the instrument. The final electrode of The electron gun is the anode, which takes the form of a disk with an axial hole. Electrons leave the cathode and accelerate toward the anode. The control and

Alignment of the electron gun is critical in ensuring satisfactory operation.

### **Condenser lenses system**

The intensity and angular aperture of the beam are controlled by the condenser lens system between the gun and the specimen. A single lens may be used to converge the beam onto the object, but, more commonly, a double condenser is employed. In this the first lens is strong and produces a reduced image of the source, which is then imaged by the second lens onto the object. The use of a small spot size minimizes disturbances in the specimen due to heating and irradiation.

### **The image-producing system:**

#### **Objective lenses and projector lenses**

The specimen grid is carried in a small holder in a movable specimen stage. The objective lens is usually of short focal length (1–5 mm) and produces a real intermediate image that is further magnified by the projector lens or lenses. A single projector lens may provide a range of magnification of 5:1, and by the use of interchangeable pole pieces in the projector a wider range of magnifications may be obtained. Modern instruments employ two projector lenses (one called the intermediate lens) to permit a greater range of magnification and to provide a greater overall magnification without a commensurate increase in the physical length of the column of the microscope.

For image stability and brightness, the microscope is often operated to give a final magnification of 1,000–250,000x on the screen. If a higher final magnification is required, it may be obtained by photographic or digital enlargement. The quality of the final image in the electron microscope depends largely upon the accuracy of the various mechanical and electrical adjustments with which the various lenses are aligned to one another and to the illuminating system. The lenses require

Power supplies of a high degree of stability; for the highest standard of resolution, Electronic stabilization to better than one part in a million is necessary. The control of a modern electron microscope is carried out by a computer, and dedicated Software is readily available.

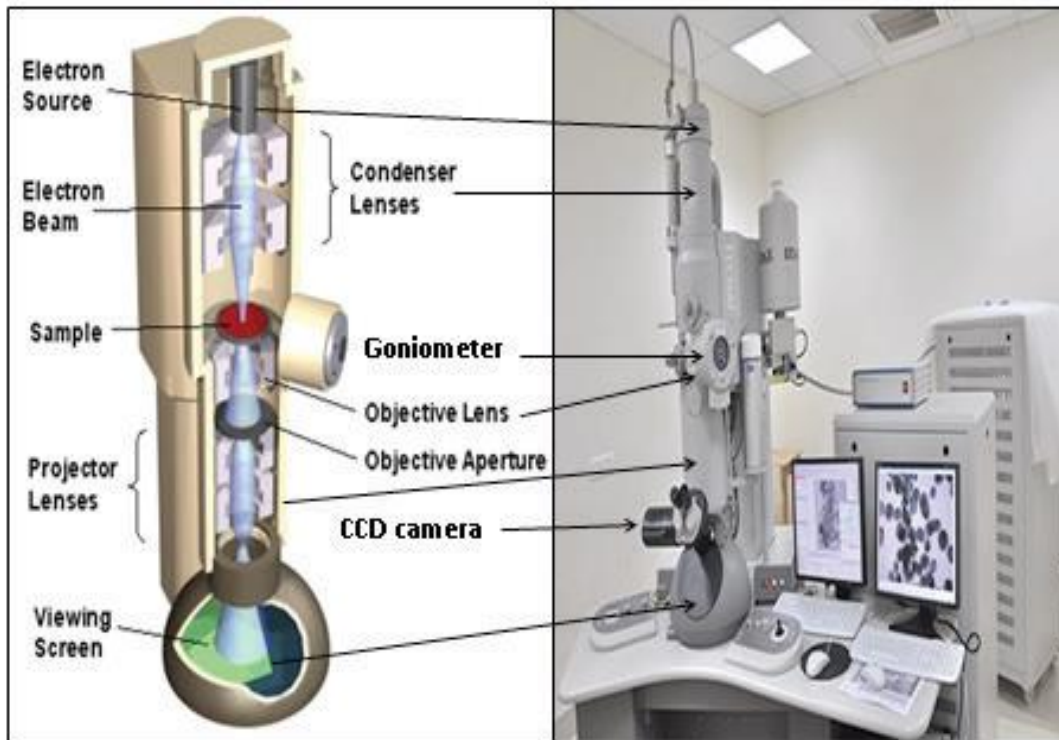
**Image translation system (Fluorescent screen and Digital photographic unit):**

TEM provides information in the form of variations of electron intensity in the Image. The electron image is monochromatic and must be made visible to the eye Either by allowing the electrons to fall on a fluorescent screen fitted at the base of the microscope column or by capturing the image digitally for display on a computer monitor. Computerized images are stored in a format such as TIFF or JPEG.

The information that TEM or SEM examination can yield are the following:

- The topographical information: the surface features of an object or "how it looks", its texture.
- The morphological information: the shape and size of the particles making up the object.
- The composition information: the elements and compounds that the object is composed of and the relative amounts of them (Howe, J.M. 2017).

## Transmission Electron Microscopy (TEM)



**Fig (3.11) Schematic Diagram of TEM.**

### **3.9.2.2 Advantages of TEM**

- TEMs offer the most powerful magnification, potentially over one million times or more
- TEMs have a wide-range of applications and can be utilized in a variety of different scientific, educational and industrial fields.
- TEMs provide information on element and compound structure.
- Images are high-quality and detailed.
- TEMs are able to yield information of surface features, shape, size and structure.
- They are easy to operate with proper training.

### **3.8.2.3 Disadvantages of TEM**

- TEMs are large and very expensive.
- Laborious sample preparation.
- Potential artifacts from sample preparation.
- Operation and analysis requires special training.

### **3.9.3 X-ray diffraction (XRD):**

X-ray diffraction (XRD) is a powerful non-destructive technique for characterizing crystalline materials. It provides information on structures, phases, preferred crystal orientations (texture), and other structural parameters, such as average grain size, crystallinity, strain, and crystal defects. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of atoms within the lattice. Consequently, the X-ray diffraction pattern is the fingerprint of periodic atomic arrangements in a given material.

X-ray diffraction is a high-tech, non-destructive technique for analysing a wide range of materials including fluids, metals, minerals, polymers, catalysts, plastics, pharmaceuticals, thin-film coatings, ceramics, solar cells, and semiconductors.

The technique finds innumerable practical applications in various industries, including microelectronics, power generation, aerospace, and many more. XRD analysis can easily detect the existence of defects in a particular crystal, its resistance level to stress, its texture, its size and degree of crystallinity, and virtually any other variable relating to the sample's basic structure. (Aboul-Enein, et al(2015))

#### **3.9.3.1 There are several advantages of XRD techniques:**

- Non-destructive, fast, and easy sample preparation.
- High-accuracy and can be done in situ.
- Allows characterizing single crystal, poly, and amorphous materials.
- Standards are available for thousands of material systems.





**Fig (3.12): X-ray diffraction (XRD)**

### **3.10 Fourier transform infrared spectroscopy (FTIR):**

A Fourier Transform Infra-Red spectrometer or FTIR analysis is an analytical technique used to identify organic, polymeric and in some case inorganic materials.

It based on the interaction of the IR radiation with matter this techniques may use for identification and characterization of chemical structures, most important feature of this method are: non-destructive , real time measurement and relatively easy to use.

The FTIR analysis method uses infrared light to scan test samples and observe chemical properties, it sends infrared radiation of about 10,000 to 100cm through a sample, with some radiation absorbed and some passed through. The absorbed radiation is converted into rotational or vibrational energy by the sample molecules, the resulting signal at the detector present as spectrum typically from 4000 to 400 cm representing molecular finger print of the sample, each molecule or chemical structure will produce unique spectral fingerprint making FTIR analysis a great tool for chemical identification.

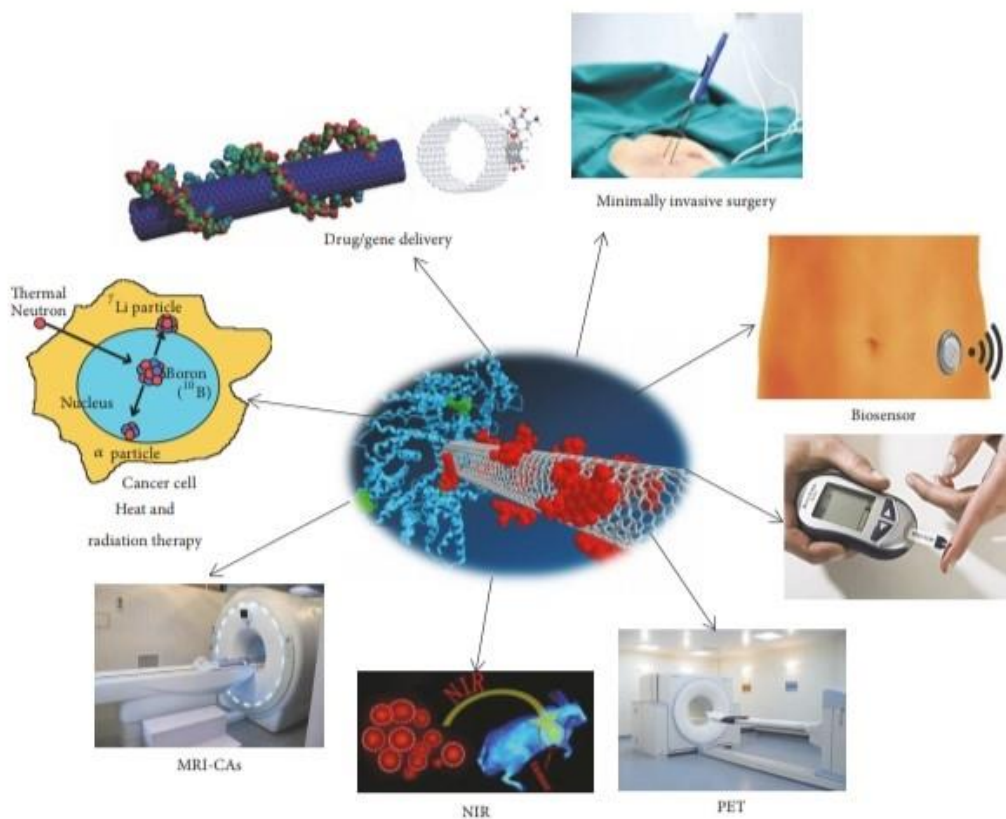
FTIR result figure with peaks every beak indicates of concentration of certain (RolantEbaMedjo, 2016).



**Fig (3.13) Fourier transform infrared spectroscopy (FTIR).**

### 3.10 Applications of carbon nanotubes

CNTs are difficult to evenly disperse in a liquid matrix such as epoxies and other polymers. This complicates efforts to utilize the nanotubes' outstanding physical properties in the manufacture of composite materials, as well as in other practical applications which require preparation of uniform mixtures of CNTs with many different organic, inorganic, and polymeric materials. To make nanotubes more easily dispersible in liquids, it is necessary to physically or chemically attach certain molecules, or functional groups, to their smooth sidewalls without significantly changing the nanotubes' desirable properties. Functionalization methods such as chopping, oxidation, and "wrapping" of the CNTs in certain polymers can create more active bonding sites on the surface of the nanotubes.



**Fig (3.14) Schematic diagram for application of CNTs.**

### 3.10.1 Biomedicine use of CNTs

#### 3.10.1.1 Drug delivery:

CNTs can be functionalized by attaching biological molecules, such as lipids, proteins, biotins, etc. to them. Then they can usefully mimic certain biological functions, such as protein adsorption, and bind to DNA and drug molecules. Specific drug delivery is an essential method used in medicine to deliver pharmaceuticals to the specific place in the body where it is needed. The method shows great promise in cancer therapy since one of the biggest challenges in treating cancer is the severe side effects caused by the chemotherapy. The harsh medication used to treat cancer attacks not only the cancer cells, but also the healthy cells of the body, and this is what causes the side effects of the treatment.(Anna Pratima, 2015).

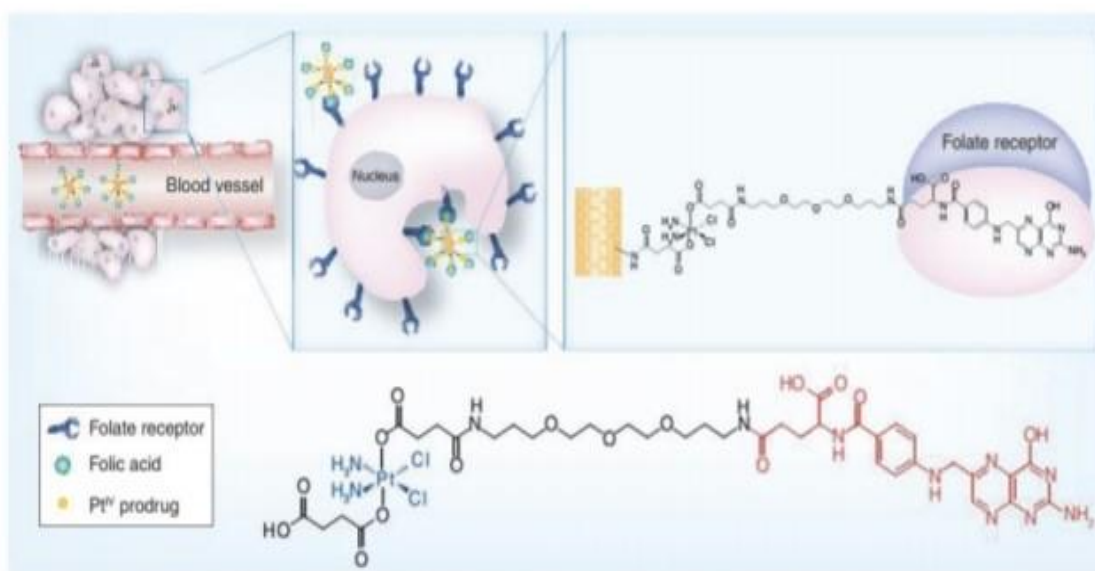


Fig (3.15) CNTs as Drug delivery.

### **3.10.1.2 Applications in Ophthalmology**

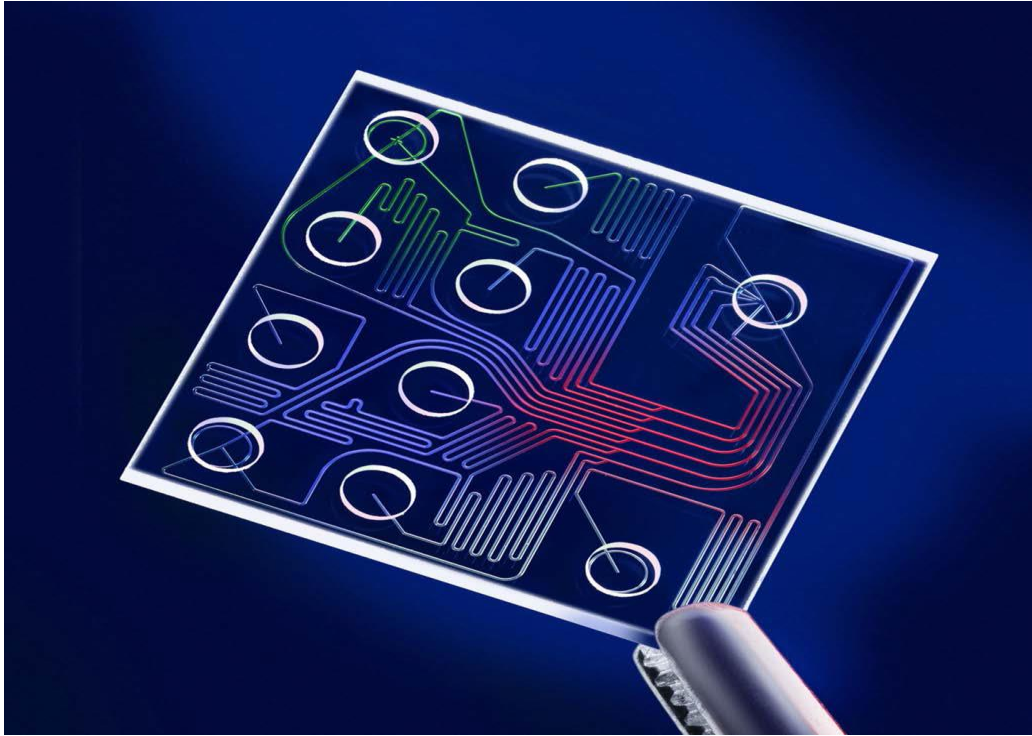
The aim of nano medicine is to monitor, control, construct, repair, defend, and improve human biological systems at the molecular level, with the help of nano devices and nanostructures that operate massively in parallel at the unit cell level, in order to achieve medical benefit. Principles of nanotechnology are applied to nano medicine such as biomimicry and pseudo intelligence. Some applications of nanotechnology to ophthalmology include treatment of oxidative stress; measurement of intraocular pressure; theragnostics; use of nano particles for treatment of choroidal new vessels, to prevent scars after glaucoma surgery, and for treatment of retinal degenerative disease using gene therapy; prosthetics; and regenerative nano medicine. The current therapeutic challenges in drug delivery, postoperative scarring will be revolutionized with the help of nanotechnology and will help in various unsolved problems such as sight-restoring therapy for patients with retinal degenerative disease. Treatments for ophthalmic diseases are expected from this emerging field. A novel nanoscale-dispersed eye ointment (NDEO) for the treatment of severe evaporative dry eye has been successfully developed.

(Zhang W et al, 2014)

### **3.10.1.3 Lab-on-a-chip**

A number of researchers are currently working to develop what they call a “lab-on-a-chip.” These small devices will be able to take one of your body fluids (like blood or saliva) and analyze it to determine whether you are likely to develop specific diseases or health conditions, without ever visiting a doctor. One way this might work is that every day at breakfast you would breathe into a straw and the chip would see if your susceptibility for any problems or exposures to dangerous chemicals has increased. Such daily monitoring might help to find future conditions very early and perhaps make treatments more successful. Some of these devices are currently available, but scientists are working to make even smaller

designs that can screen for many more diseases with a faster response time. ( Ali Jackson ,2018)



**Fig (3.16): Lab-on-a-chip**

#### **3.10.1.4 Artificial implants**

Normally body shows rejection reaction for implants with the post administration pain but, miniature sized nanotubes and nano horns get attached with other proteins and amino acids avoiding rejection. Also, they can be used as implants in the form of artificial joints without host rejection reaction. Moreover, due to their high tensile strength, carbon nanotubes filled with calcium and arranged/grouped in the structure of bone can act as bone substitute.

#### **3.10.1.5 Genetic Engineering**

In genetic engineering, CNTs used to manipulate genes and atoms in the development of bio imaging genomes, proteomics and tissue engineering. The unwound DNA (single stranded) winds around SWNT by connecting its specific nucleotides and causes change in its electrostatic property. This creates its potential application in diagnostics (polymerase chain reaction) and in therapeutics. Wrapping of carbon nanotubes by single-stranded DNA was found to be sequence-dependent, and hence can be used in DNA analysis. Nanotubes due to their unique cylindrical structure and properties are used as carrier for genes (gene therapy) to treat cancer and genetic disorders.

#### **3.10.1.6 Biosensors**

CNTs act as sensing materials in pressure, flow, thermal, gas, optical, mass, position, stress, strain, chemical, and biological sensors. Some applications of carbon nanotube based sensors are given below. Biomedical industry CNT-incorporated sensors are expected to bring about revolutionary changes in various fields and especially in the biomedical industry sector. An example is the glucose sensing application, where regular self-tests of glucose by diabetic patients are required to measure and control their sugar levels. Another example is monitoring of the exposure to hazardous radiation like in nuclear plants/reactors or in chemical laboratories or industries. The main purpose in all these cases is to detect the exposure in different stages so that appropriate treatment may be administered. CNT-based nano sensors



are highly suitable as implantable sensors. Implanted sensors can be used for monitoring pulse, temperature, blood glucose, and also for diagnosing diseases. One such example is the use of nanotubes to track glucose levels in the blood, which would allow diabetics to check their sugar levels without the need for taking samples by pricking their fingers. (Deng P, Xu Z, Li J, 2013).

### **3.10.1.7 Surgery**

The technique developed by Rice University, two pieces of chicken meat is fused by a flesh welder, by placing two pieces of chicken touching each other. In this technique, green liquid containing gold-coated nano shells is allowed to dribble along the seam and two sides are weld together. This method can be used arteries which have been cut during organ transplant. The flesh welder can be used to weld the artery perfectly.

### **3.10.1.8 Visualization**

Drug distribution and its metabolism can be determined by tracking movement. Cells were dyed by scientists to track their movement throughout the body. These dyes excited by light of a certain wavelength to glow. Luminescent tags were used to dye various numbers of cells. These tags are quantum dots attached to proteins which penetrate cell membranes. The dots were of various sizes and bio-inert material. As a result, sizes are selected so that the frequency of light used to make a group of quantum dots fluoresce, and used to make another group incandesce. Thus both groups can be lit with a single light source.

### **3.10.1.9 Tissue engineering**

In tissue engineering, nanotechnology can be applied to reproduce or repair damaged tissues. By using suitable nanomaterial-based scaffolds and growth factors, artificially stimulated cell proliferation, in organ transplants or artificial implants therapy nano technology can be useful, which can lead to life extension.

#### **3.10.1.10 Antibiotic resistance**

Antibiotic resistance can be decreased by use of nano particles in combination therapy. Zinc Oxide nano particles can decrease the antibiotic resistance and enhance the antibacterial activity of Ciprofloxacin against microorganism, by interfering with various proteins that are interacting in the antibiotic resistance or pharmacologic mechanisms of drugs.

#### **3.10.1.11 Immune response**

The nano device bucky balls have been used to alter the allergy/ immune response. They prevent most cells from releasing histamine into the blood and tissues, as these bind to free radicals better than any anti-oxidant available, such as vitamin E.

#### **3.10.1.12 Nano pharmaceuticals**

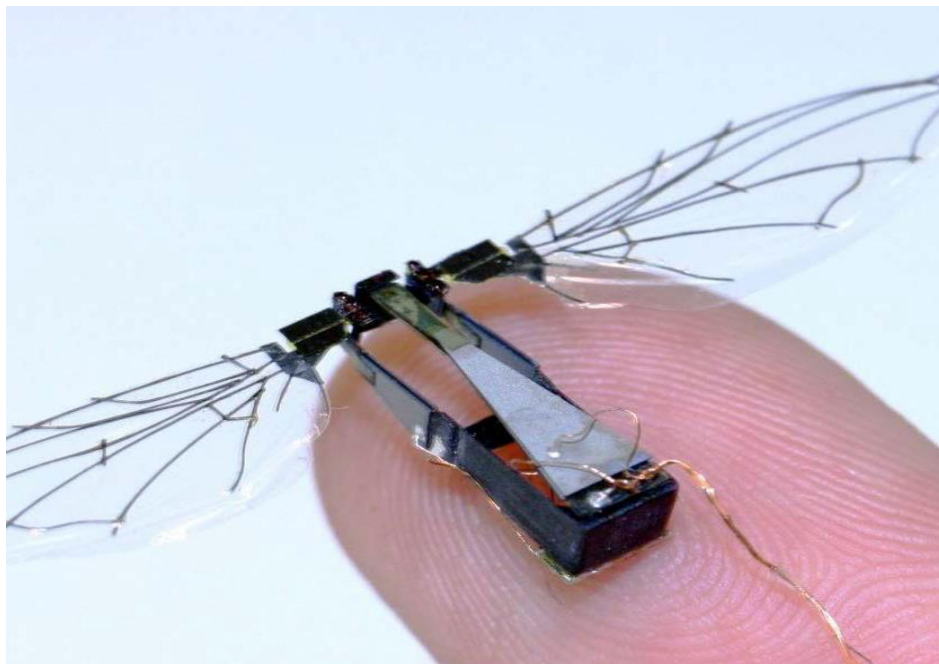
Nano pharmaceuticals can be used to detect diseases at much earlier stages and the diagnostic applications could build upon conventional procedures using nanoparticles. Nano pharmaceuticals are an emerging field where the sizes of the drug particle or a therapeutic delivery system work at the nanoscale. Delivering the appropriate dose of a particular active agent to specific disease site still remains difficult in the pharmaceutical industry. Nano pharmaceuticals have enormous potential in addressing this failure of traditional therapeutics which offers site-specific targeting of active agents. Nano pharmaceuticals can reduce toxic systemic side effects thereby resulting in better patient compliance.

Pharmaceutical industry faces enormous pressure to deliver high-quality products to patients while maintaining profitability. Therefore pharmaceutical companies are using nanotechnology to enhance the drug formulation and drug target discovery. Nano pharmaceutical makes the drug discovery process cost effective, resulting in the improved Research and Development success rate, thereby reducing the time for both drug discovery and diagnostics.

### **3.10.2 Another applications:**

#### **3.10.2.1 Mini-drones**

There is currently a major push in nanotechnology to create electronic devices that are even smaller and more powerful than those we have today. One of the proposed benefits of such scaling down is the possibility of building incredibly tiny drones that could fly almost undetected through the air and collect data (including images) of people and places without their knowledge. This research is motivated by a desire to decrease the chances and impacts of terrorist attacks, but surveillance technologies are currently being employed by many people outside of the military as well.



**Fig (3.17): Mini-drones**

### 3.10.2.2 Nano silver socks

Nano silver socks are one of the most widespread nanotechnology products available. They are just like normal socks except that they have silver nanoparticles embedded in their threads. The nanoparticles act as an antibiotic agent, killing bacteria and fungus on the wearer's feet. This can help keep foot odor from occurring as well as provide protection against infection for people with circulation issues or compromised immune systems. Researchers have shown that the silver nanoparticles can wash out of the socks over time, allowing the particles to enter the wastewater stream. Scientists are still trying to determine the impact of nanoparticles on the wastewater treatment system and what happens after they leave the wastewater system. ( Ali Jackson ,2018)



**Fig (3.18): Nano silver socks**

### **3.10.2.3 Water Treatment**

Different heavy and toxic metals can be removed from water by using CNTs as adsorbents.

### **3.10.2.4 Energy storage**

Carbon Nano fluids are extensively used in the solar thermal conversion, due to their excellent performance as a solar absorber and a heat transfer fluid which can be used in Capacitor and battery.

### **3.10.2.5 Electronics**

#### **Electrodes**

Carbon nanotubes have been widely used as electrodes for chemical and biological applications and many other electrochemical studies, like transparent conductive electrodes (TCEs), which simultaneously conduct the current and transmit light mostly in the visible spectral range, are of great significance.

#### **Field Emission**

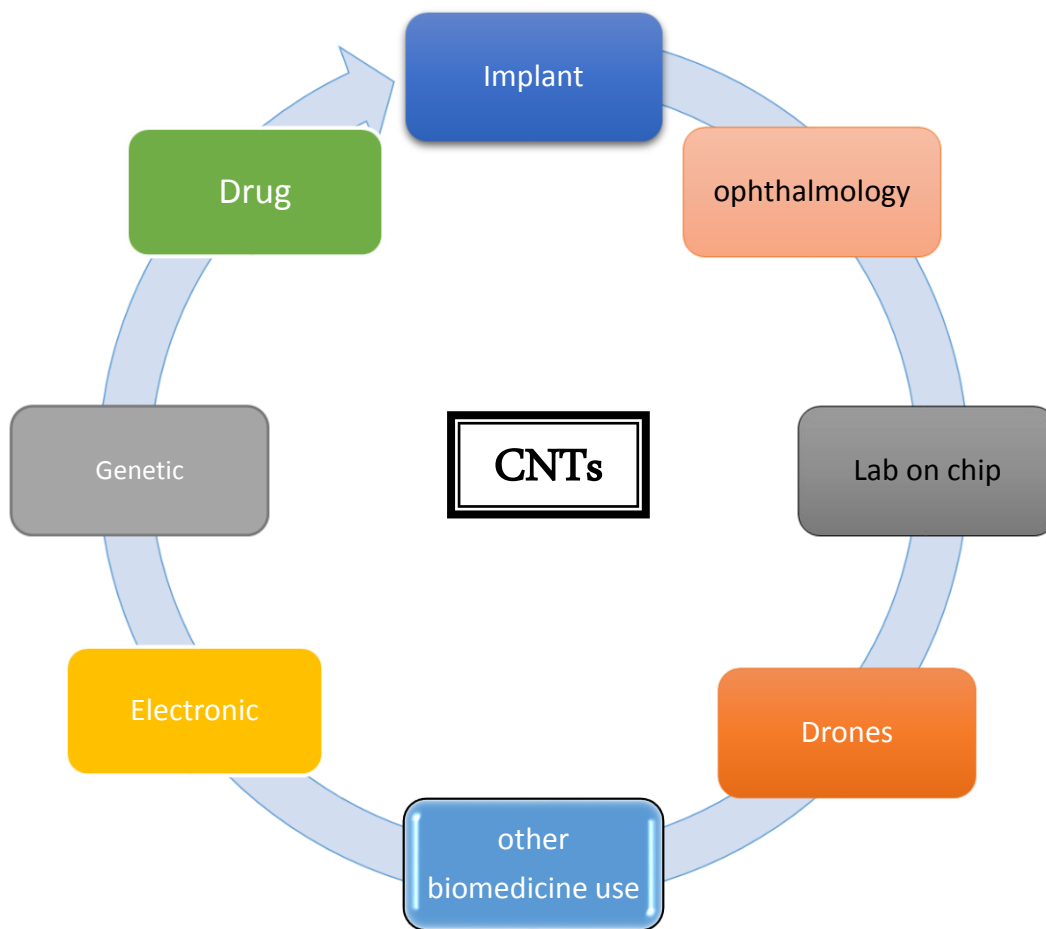
CNTs, due their high chemical stability, a sharp tip, better mechanical power, and good aspect ratio, are appropriate to be used as field emission electron sources

#### **Carbon Nanotube-Based Diodes, Field-Effect Transistors,**

The mutual properties of both metallic and semiconducting types of CNTs can form special building blocks for future electronics.

#### **Displays**

CNTs given high electrical conductivity, sharpness and more concentrate of electrical field the high field emission which make new class, high resolution and low cost. (Dionne, 2016)



**Fig (3.19) Application of CNTs**

### **3.11 Advantages of carbon nanotubes CNTs**

- Biocompatible, Non-biodegradable and non-immunogenic nature.
- Highly elastic nature and have the possibility of intracellular delivery.
- Ultra-light weight and do not break down during processing.
- It has an open end on both sides, which makes the inner surface accessible and subsequent incorporation of species within nanotubes is particularly easy.
- Nanotubes have longer inner volume relative to the diameter of nanotubes for entrapment.
- CNTs are able to enter cells by spontaneous mechanism due to its tubular and nano needle shape.
- It has distinct inner and outer surface, which can be differentially modified for chemical biochemical functionalization.

### **3.12 Limitation of CNTs**

- May exhibit minimum cytotoxicity.
- Lack of solubility in most solvents compatible with the biological milieu (aqueous based).
- The production of structurally and chemically reproducible batches of CNTs with non-identical characteristics.
- Difficulty in maintaining high quality and minimal impurities.

## **CHAPTER FOUR**

### **MATERIAL AND PROCESS**



# CHAPTER FOUR

## MATERIAL AND PROCESS

Flame method was chosen to fabricate carbon nanotube, it cost effective, simple and it is components available according to our situation several steps have been followed, first I synthesized material, purified it then characterized.

### 4.1 Synthesis:

Firstly, material which used as carbon source was selected.

#### 4.1.1 Carbon source:

**Frankincense** (also known as **olibanum**) is an aromatic resin used in incense and perfumes, obtained from trees of the genus *Boswellia* in the family Burseraceae. The word is from Old French franc encens ('high-quality incense')

There are several species of *Boswellia* that produce true frankincense *Boswellia sacra* (syn. *B. bhaw-dajiana*, syn. *B. carteri*), *B. frereana*, *B. serrata* (*B. thurifera*, Indian frankincense), and *B. papyrifera*. Resin from each is available in various grades, which depend on the time of harvesting. The resin is hand-sorted for quality.

Frankincense was used as source of carbon which contains:

- Acid resin (6%), soluble in alcohol ( $C_{20}H_{32}O_4$ ).
- Gum (similar to Arabic gum) 30-36%.
- 30-acetyl-beta-boswellic acid (*Boswellia sacra*)
- Incensole acetate.
- Natural material with flammable properties.

It have bought from the market then have used it by it is nature without any modifications.

It have been burned and burning time took for three to five minutes, in room.



**Fig (4.1): Natural Frankincense**

### 4.1.2 Substrate

Pure Iron pieces had been used as substrate. It have been divided into parts in order to cover frankincense during burning .



**Fig (4.2): Iron pieces**

### 4.1.3 Catalysis:

Methanol and ferric acid were mixed then added to surface of iron piece. To maintain stability for iron particle's and act as catalysis. During flame it will pick carbon particles to substrate surface.



**Fig (4.3):Methanol**



**fig (4.4): ferric acid**



**Fig (4.5): Mix of methanol and ferric acid**





**Fig (4.6):After adding the mixed material to substrate surface**

Frankincense were put in the middle between two block with 5 cm between them and covered by Substrates. When we burned particles all substrate surface had been covered, then we collect it as you can see in figures below.



**Fig (4.7) block**

**fig (4.8): Frankincense between block**



**Fig (4.9): flame of frankinosinse. Fig (4.10): substrate around flame.**



**Fig(4.11): after burn finish.**





**Fig (4.12): Samples after collected from substrate.**

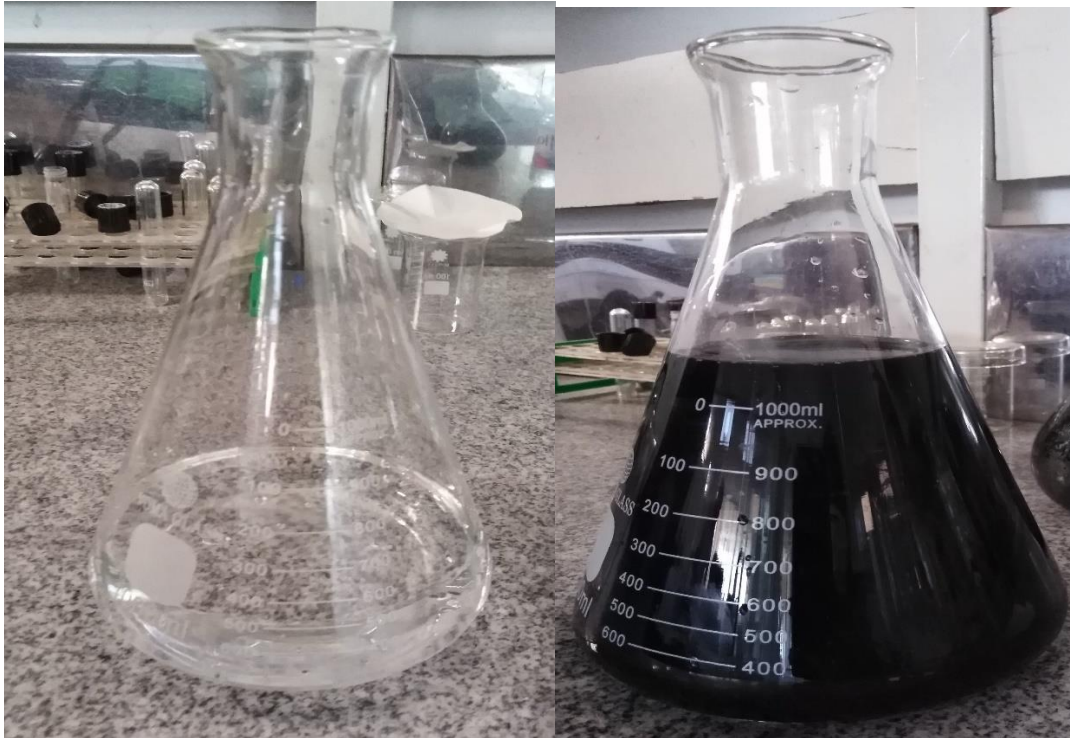
25ml of nitric acid, 75 ml of sulfuric acid were added to sample which about 1.5 Gm. then we mixed it together, after that we left it for two days in temperature room.



**Fig (4.13): Mixed sample**

## 4.2 Dilution:

After 48 hours left, distiller water was added to sample for dilution.



**Fig (4.14): Adding distiller water to sample.**

### 4.3 Purification:

Centrifuge was used for separate sample particles, we used centrifuge with 14800 velocity per second. Samples were put in tube for 20 minutes, as it showed in the following picture.



**Device Name: centrifuge.**

**Company: herolab**

**Model: microlen 16**

**Speed: 14800**

**Fig(4.15): Centrifuge device**



**Fig (4.16) sample inside tubes.**



**Fig (4.16) Tubes inside device**



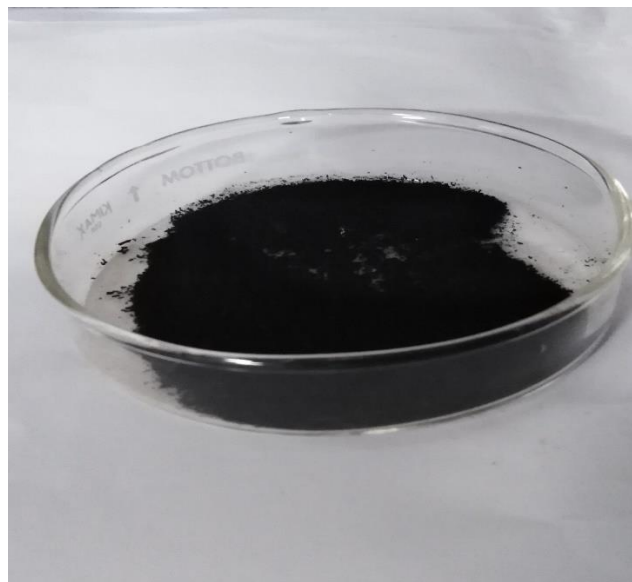
**Fig (4.17) Deposition of sample after centrifuge.**



I washed sample twice time using distiller water then left it until dry then we collected it as form of powder for characterize it.



**Fig(4.18): after dryness**



**Fig (4.19): Collected sample**

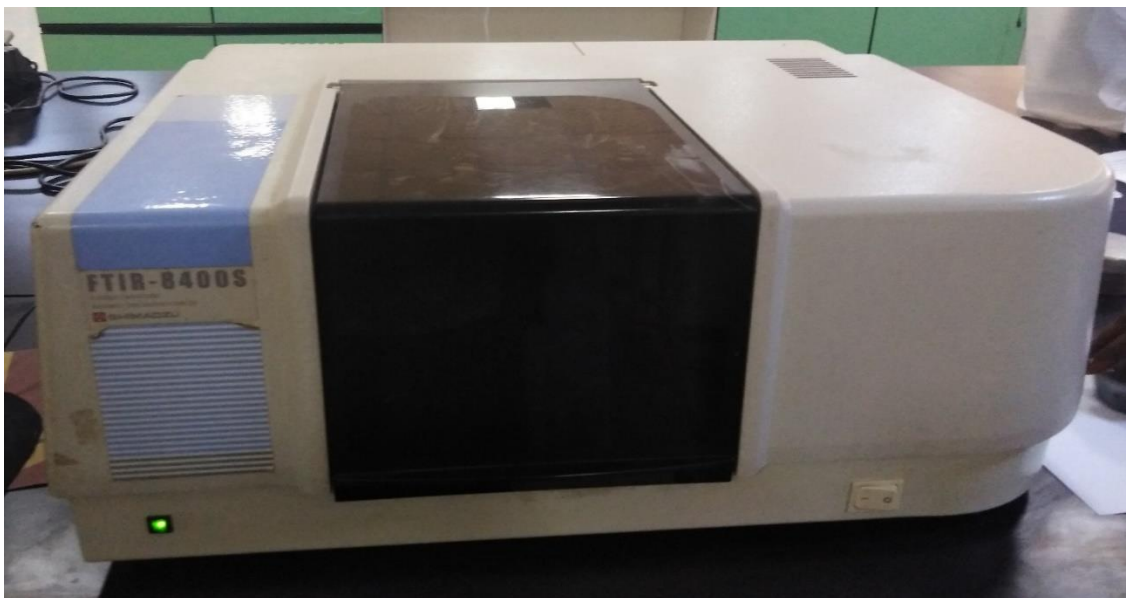
#### **4.4 Characterization devices:**

##### **Fourier transform infrared spectroscopies (FTIR):**

Test of Infrared and Fourier transform infrared spectroscopies (FTIR) with 8400S

Spectrophotometer model was done in central lab in Khartoum University, FTIR evaluate the impurities resulting from synthesis and to detect the presence of organic molecules capped on the nanotube surface. Functionalization by binding a series of functional groups onto the CNT sidewalls by means of chemical reactions or physical interactions is a common practice to enhance the solubility and increase the selectivity of CNTs and the majority of IR and FT-IR applications have been aimed at characterizing such surface modifications.

IR radiation with matter this techniques may use for identification and characterization of chemical structures, most important feature of this method are: non-destructive, real time measurement and relatively easy to use.



Model: FTIR 4800S

- **Fig (4.20) FTIR 8400S Spectrophotometer model**



## Scanning electron microscopy (SEM)

Scanning Electron Microscope functions exactly as their optical counterparts except that they use a focused beam of electrons instead of light to “image” the specimen and gain information as to its structure and composition. Given sufficient light, the unaided human eye can distinguish two points 0.2 mm apart. If the points are closer together, they will appear as a single point of image.

Light microscope has a magnification of about 1000x and enables the eye to resolve objects separated by 200 nm. Electron Microscopes were developed due to the limitations of light microscopes, which are limited by the Physics of light.



Device name: Scanning Electronic Microscope.

Model: Vega 3.

Origin: Techeiqu.

Agent: Medtic.

Serial number: 115-0082

**Fig (4.21)** Scanning Electronic Microscope (SEM) device

# **CHAPTER FIVE**

## **Results and discussion**

# CHAPTER FIVE

## Results and discussion

### 5.1 FTIR result:

FTIR analysis was done for carbon powder at Khartoum University in central lab. The analysis was performed in Bruker Tensor in the scanning range of 400 – 4000  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$ .

Sample was grinded, compressed and placed inside it infrared ray passed through sample, each curve indicates for concentration of specific functional group.

Every spectrum number indicate for specific functional group for instance Alcine group which contain double bond two atoms concentrate in (1600), Carbonile group COOH appeared in (1700), Triple bond showed in (2225), OH group in (3400).

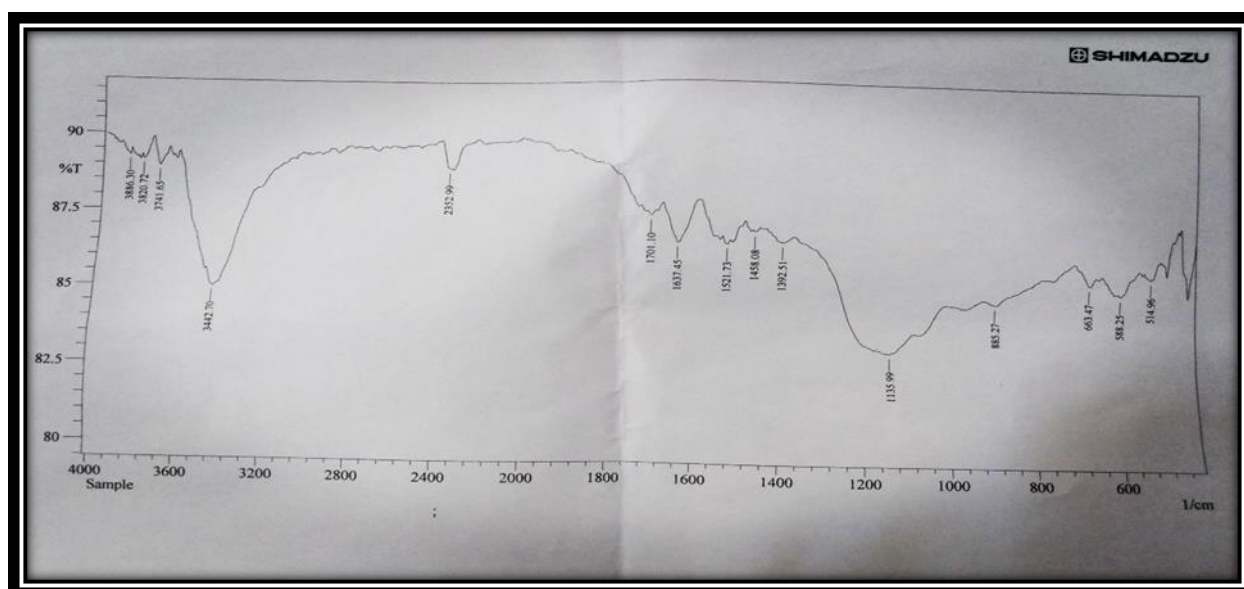


Fig (5.1) Spectrum of FTIR result

## 5.2 Scanning electron microscope (SEM) result:

Test of scanning electron microscope (SEM), was done in forensics in order to obtain image natural and burning frankonsice with their spectrum shown in figures below.

Spectrum result contain large amount of carbon (79.3) which lead to produce nano powder material and contain (29.2) of oxygen , and another elements.

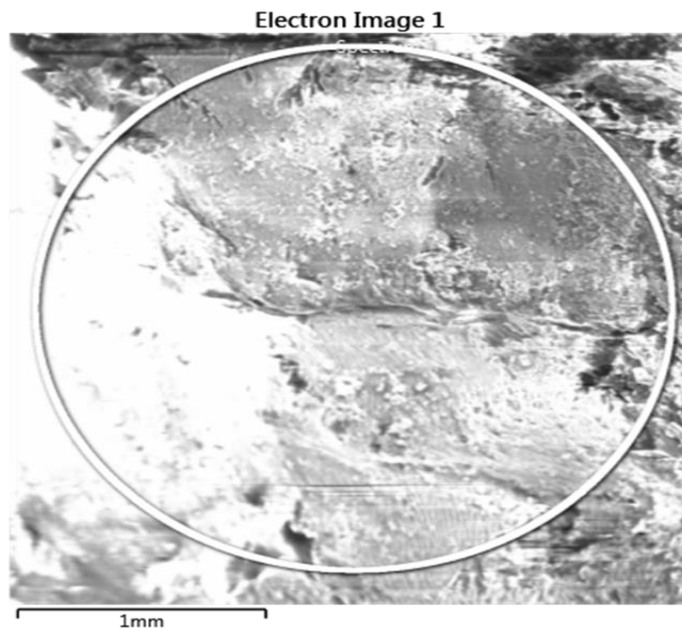


FIG (5.2) Image of standard frankoinseinsse.

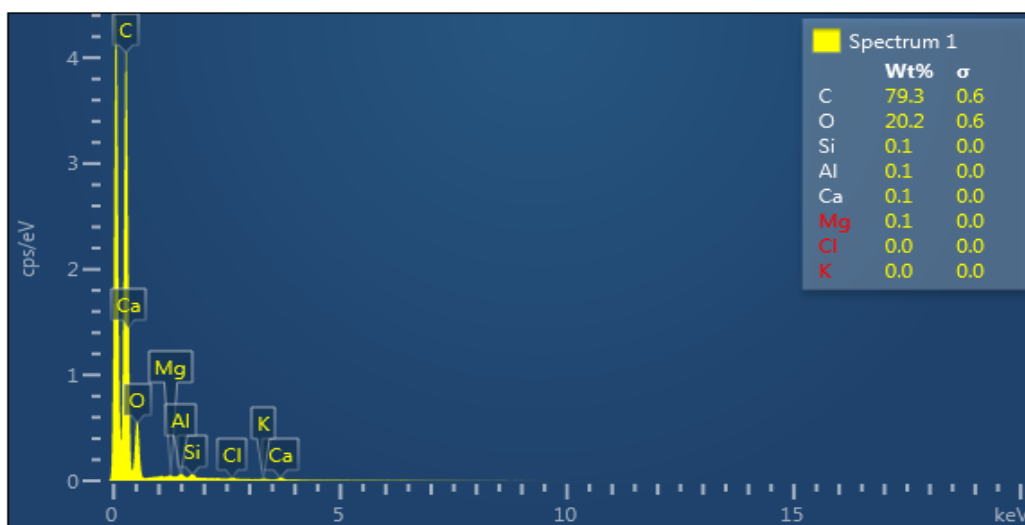
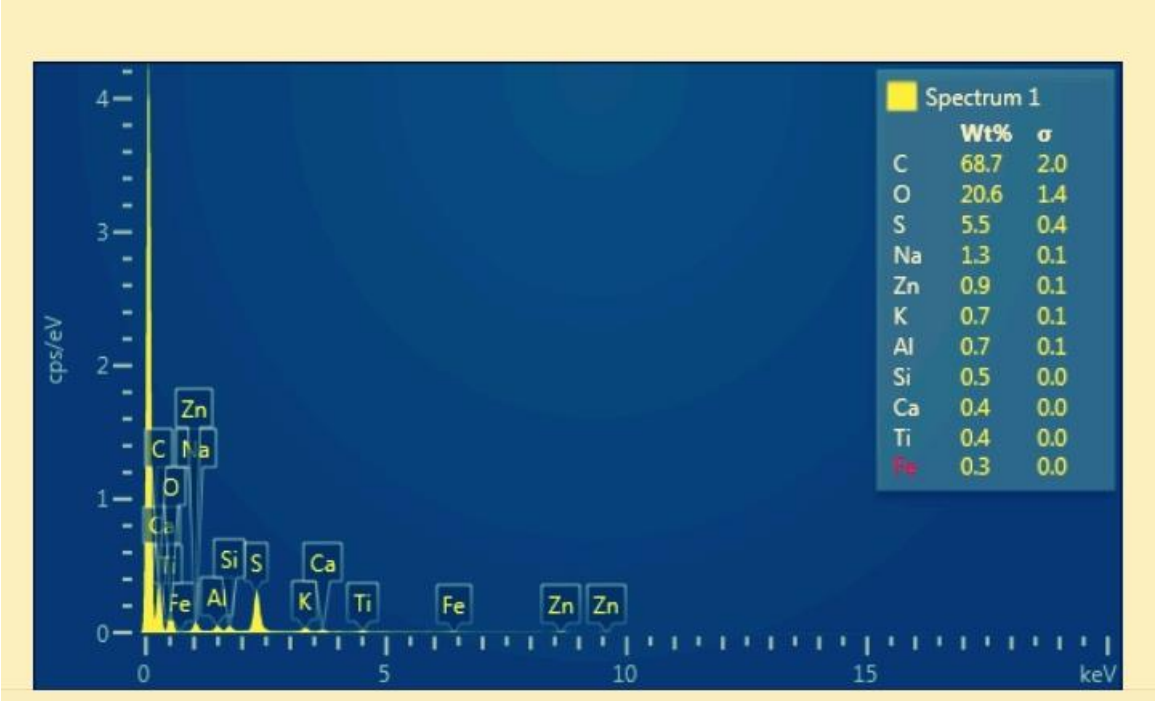


Fig (5.3): Spectrum of standard frankinosinsse.

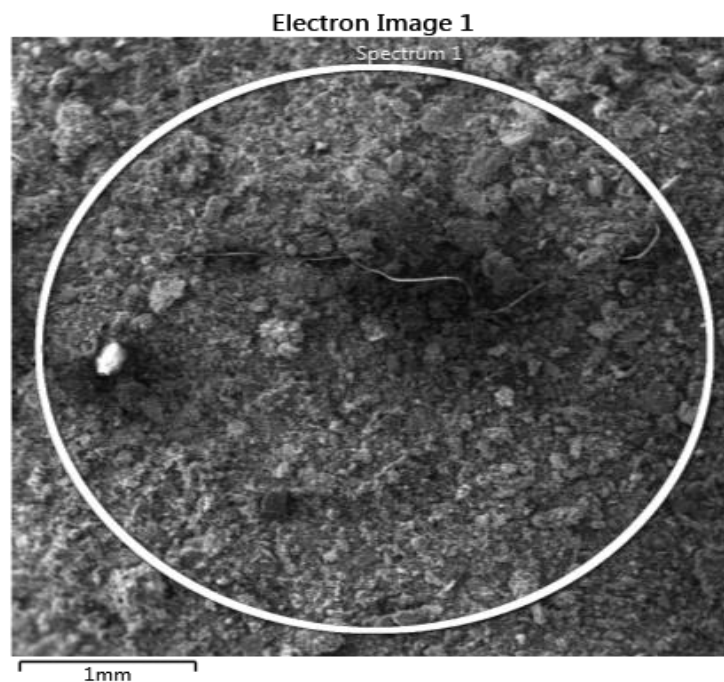
The Spectrum result of synthesiseis sample contain (68.7) percentage of carbon , oxegen contain (20.6), silicone (5.5) and another elements shown below



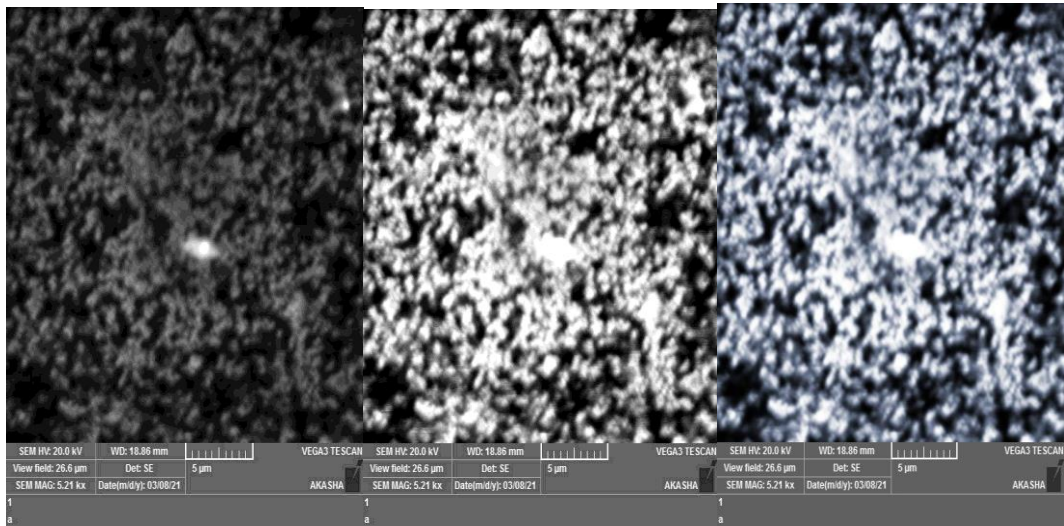
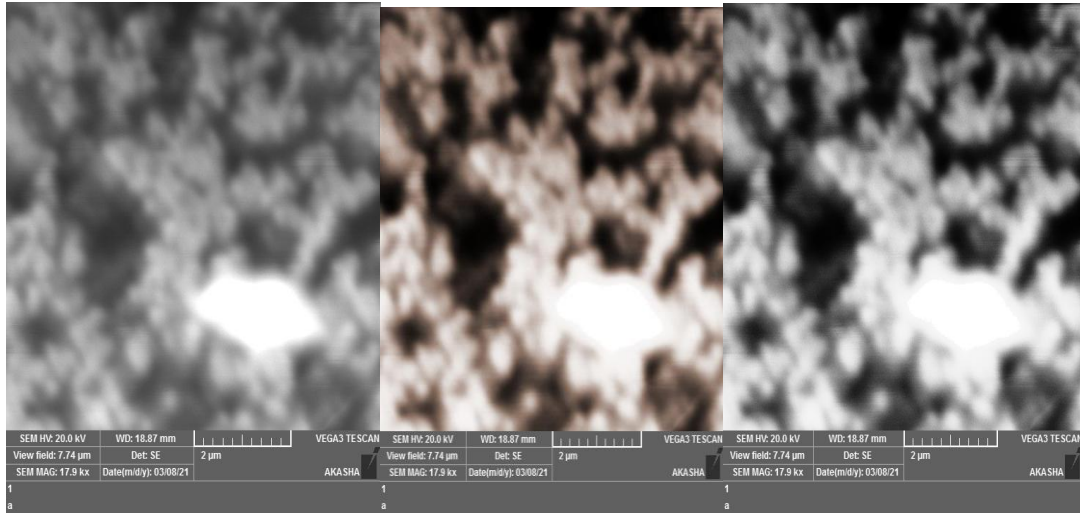
**Fig (5.4) spectrum of sample via SEM**

The morphology structure is carbon nano powder, figure (5.5) shown sample in milli scale it contain impurities this according to different parameters:

- After preparing sample , it took many days then had characterizes so the sample affected by long period time.
- The number of round per minute for Centrifuge device w used not as required. Figure (5.6) shown sample in micro scales. With different view field (7.74 and 26.6) and different magnification range (18.87 and 18.86) and 20.00kv images indicate for existence of carbon nano powder which have same behavior of carbon nanotubes.



**Fig (5.5): Synthesised sample**



**Fig (5.6): Synthesised sample with different range.**

# CHAPTER SIX

## Conclusions and Recommendations

### 6.1 Conclusions:

A mix of methanol and ferric acid was added to surface of substrate to act as catalyst. franksice was burned and covered all substrates surfaces, the sample was collected in powder form, sulphuric and ferric acid was added to the powder in order to purification distiller water been added for dilution purpose then used centrifuge to remove impurities sample been left until dry.

In order to know functional group, FTIR test was done for sample then SEM result obtained which include spectrum and image for natural and synthesis sample.

Result of synthesis sample was carbon nano powder which is consider as a part of CNTs component with same characterise of carbon nanotube.



## 6.2 Recommendations:

- In flame method we should observe (distances between substrates, burning time and amount of elements which form catalysis).
- After centrifuge, sample, should be wash at least three time using distiller water to assure removing all impurities.
- Although I used centrifuge with (14600 per/s), with increasing time, but impurities do appear in image, so I recommend to use centrifuge with (16000 per/s), to obtain good result.
- XRD test is necessary.
- TEM test should done.
- Try to use Carbon nano powder in various application

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