

# **The Study of MgO Nano-Composite: Synthesis, Characterization and Probable Application**

**A**

**Project Report**

**SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT  
FOR THE AWARD OF THE DEGREE OF**

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**IN**

**NANO SCIENCE AND TECHNOLOGY**

**Submitted By**

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**CERTIFICATE**

This is to certify that Mr. Satyendra Kumar Satyarthi, a student of final semester M. Tech. (Nanoscience and Technology), Applied Physics Department, during the session 2014-2016 has successfully completed the project work on **“The Study of MgO Nano-Composite: Synthesis, Characterization and Probable Application”** at DTU, Delhi and has submitted a satisfactory report in partial fulfillment for the award of the degree of Master of Technology.

The assistance and help received during the course of investigation have been fully acknowledged. He is a good student and we wish him good luck in future.

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# Candidate Declaration

I hereby declare that the work which is being presented in this thesis entitled “**The Study of MgO Nano-Composite: Synthesis, Characterization and Probable Application**” is my own work carried out under the guidance of **Dr. Nitin. K. Puri**, Assistant Professor, Delhi Technological University, Delhi.

I further declare that the matter embodied in this thesis has not been submitted for the award of any other degree or diploma.

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## **ABSTRACT**

In This work we have investigated the role of complexing agents, oxalic acid, in the production of MgO Nano-crystals. It is found that the thermal profiles of the precursors can reveal the effects of crystal growth during thermal annealing. X-ray diffraction confirms that the final products are pure phase and It form in simple Cubic crystalline structure till any foaming agent or surfactant has been mixed, After adding foaming agent Sodium laureth Sulfate (SLS) particles are formed in Orthorhombic crystalline structure and after adding Surfactant Cetrimonium Bromide particles are formed in Monoclinic structure. It is also found that complexing agents can affect the rate of crystal growth. The structures of the oxalic acid as well as the complexation sites play very important roles in the formation of the Nano-crystals. The complexing agents influence the rate of growth which affects the final crystallite size of the materials. Surprisingly, it is also found that oxalic acid act as surfactants inhibiting crystal growth even at a high temperature of 950°C and a long annealing time of 36 h.

**Key words:** MgO, Nano-Structure, Sol-gel Method, Complexing Agents.

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**Chapter-1**  
**GENERAL INTRODUCTION**

# **1. Introduction**

## **1.1 Nano Structure and Nano-materials:**

Nano-materials are unit the particles (crystalline or amorphous) of organic or inorganic materials having sizes within vary of 1-100 nm [1]. Nano-materials are unit classified into Nano-structure materials and Nano-phase/Nano-particle materials. The previous consult with condensed bulk materials that area unit manufactured from grains with grain sizes within the micro-millimeter size vary while the latter area unit sometimes the dispersive Nano-particles to tell apart Nano-materials from bulk, it's vitally vital to demonstrate the distinctive properties of Nano-materials and their prospective impacts in science and technology [2].

Nanoparticles having completely different properties compared with bulk materials. Most of the researchers are operating with metal chemical compound Nano-particles due to their distinctive properties like hydrophobic, picture chemical process, stability and etc. therefore they're utilized in several applications named as coatings, catalysts, anti-bacterial, medical sciences, sensors, semiconductors, capacitors and batteries [3].

Technology within the 21<sup>st</sup> century needs the miniaturization of devices in to Nano-meter sizes whereas their final performance is dramatically enhanced. This raises several problems concerning to new materials for achieving specific practicality and property. Engineering is that the style fabrication and application of nanostructures or Nano-materials and therefore the fundamental understanding of the relationships between physical properties or phenomena and material dimensions. It's a replacement field or a replacement scientific domain. Engineering additionally guarantees the likelihood of making nanostructures of stability phases with non-conventional properties including electrical conduction and magnetism [4]. Another important side of Nano-technology is that the miniaturization of current and new instruments, sensors and machines which will greatly impact the globe we tend to sleep in. samples of possible miniaturization are computers with infinitely land that compute algorithms to mimic human brains, biosensors that warn America at the early stage of the onset of sickness and ideally at the molecular level and target specific medicine that mechanically attack the pathological cells on website, Nano-robots which will repair internal injury and take away chemical toxins in human bodies, Nano-scaled natural philosophy that perpetually monitor our native environment [5].

Nano-materials can be classified as completely different ways of classification together with origin, dimensions and their structural configuration

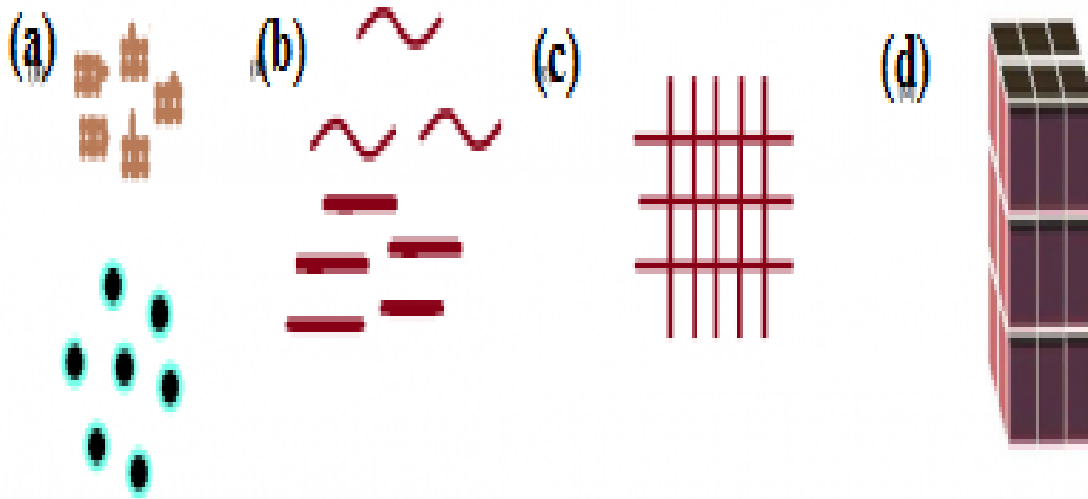
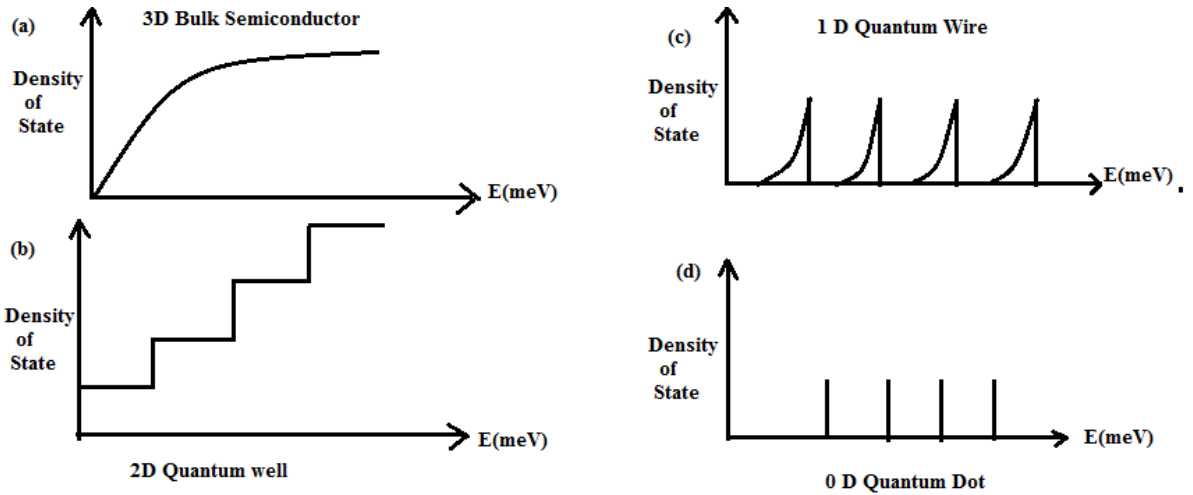


Figure 1.1 Different-Different Nano-structures <sup>[6]</sup>

## 1.2 Classification of Nano-materials:

Depending on the dimension during which the scale result on the resultant property becomes apparent, the Nano-materials is classified as zero-dimensional (quantum dots) during which the movement of electrons is confined in all 3 dimensions, one-dimensional (quantum wires) during which the electrons can solely move freely within the X-direction, two-dimensional (thin films) during which case the negatron will move within the X-Y plane, or 3 dimensional (Nano-structured material engineered of Nano-particles as building blocks) during which the free electron will move within the X, Y and Z directions [7].



**Figure 1.2 Variation of density of states with dimensionality** <sup>[8]</sup>

Nano-materials on the basis of Dimension.

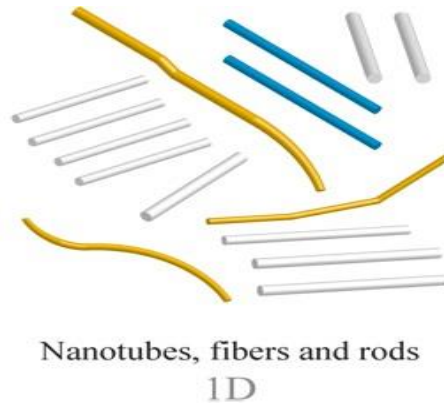
- (a) 3-D Nano-materials.
- (b) 2-D films, networks.
- (c) 1-D Nano-fibers, wires and rods.
- (d) 0-D spheres and clusters.

**(a) Zero Dimensional (0-D):** These Nano-materials have Nano-dimensions in all the three directions. Metallic Nano-particles as well as gold and silver Nano-particles and semiconductor such as quantum dots are the proper example of this sort of Nano-particles. Most of those Nano-particles are spherical in size and also the diameter of these particles are in the 1-50 nm vary. Cubes and polygons shapes are conjointly found for this sort of Nano-materials.



**Figure 1.3 Zero Dimensional (0-D)** <sup>[9]</sup>

**(b) One Dimensional (1-D):** In these Nanostructures, one dimension of the nanostructure will be outside the micro-millimeter varies. These include Nano-wires, Nano-rods, and Nano-tubes. These materials are long (several micrometer in length), but with diameter of solely a few micro-millimeter. Nano-wire and Nano-tubes of metals, oxides and other materials square measure few examples of this type of materials.



**Figure 1.4 One Dimensional (1-D)<sup>[10]</sup>**

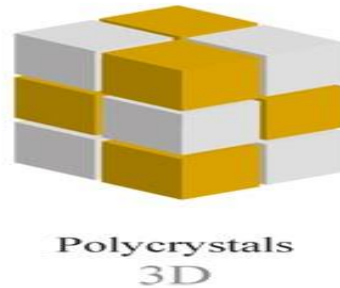
**(c) Two Dimensional (2-D):** In this kind of Nano-materials, two dimensions square measure outside the Nano-meter vary. These include totally different kind of Nano films like coatings and thin-film multi layers, Nano sheets or Nano-walls. The area of the Nano films will be massive (several sq. micrometer), but the thickness is perpetually in Nano scale vary.



**Figure 1.5 Two Dimensional (2-D)<sup>[11]</sup>**



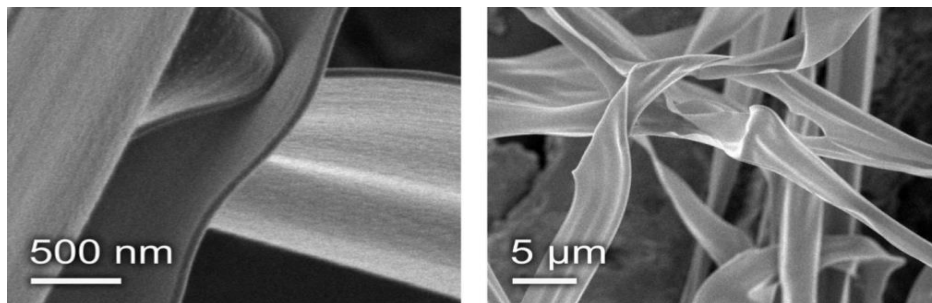
**(d) Three Dimensional (3-D):** All dimensions of these are outside the Nano meters vary. These include bulk materials composed of the individual blocks that square measure in the micro-millimeter scale (1-100 nm).



**Figure 1.6 Three Dimensional (3-D)** <sup>[12]</sup>

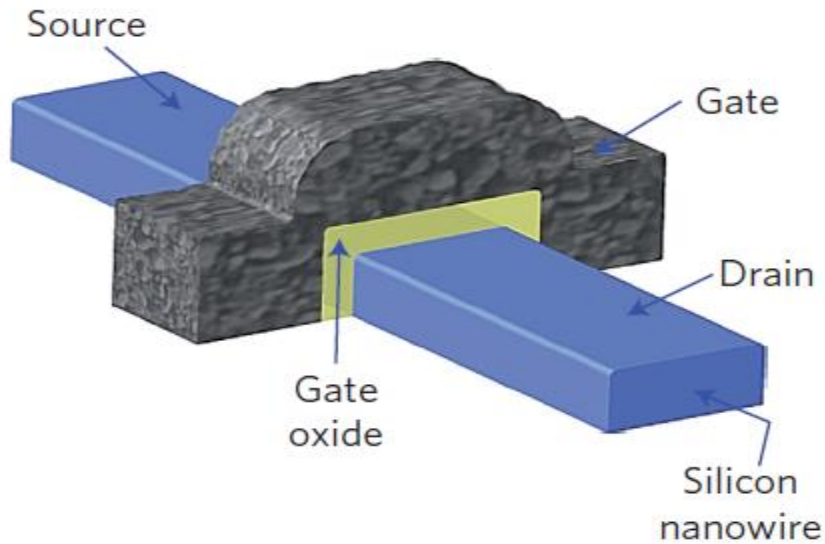
**1.3 Origin Based:** On the basis of origin Nano- Materials are categories as fallows.

**(a) Natural Nano-materials:** Nano-materials which are happiness to resource of nature are outlined as natural milli-micron. As per examples virus, protein molecules together with protein originated from nature are some natural Nano structured materials. In addition following are few examples, mineral such as clays, natural colloids, such as milk and blood (liquid colloids), fog (aerosol type), gelatin (gel type), mineralized natural materials, such as shells, corals and bones, Insect wings and opals, Spider silk, Lotus leaf and similar(Nasturtium), Gecko feet, volcanic-ash, ocean spray etc.



**Figure 1.7 Spider silk (This silk is not cylindrical but a thin ribbon)** <sup>[13]</sup>

**(b) Artificial Nano-material:** Artificial Nano-particles are those that are ready deliberately through a well-defined mechanical and fabrication method. The examples of such materials are carbon Nano-tubes, semiconductor Nano-particles like quantum dots etc.

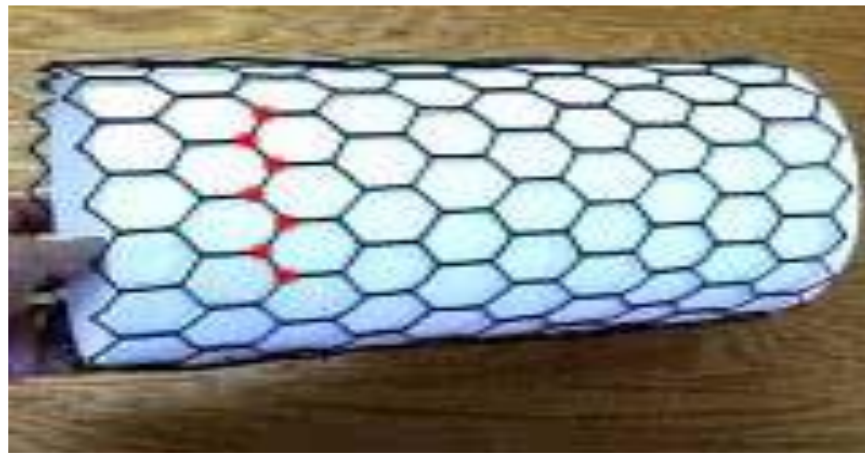


**Figure 1.8 Schematic of an n-channel Nano-wire transistor.** [14]

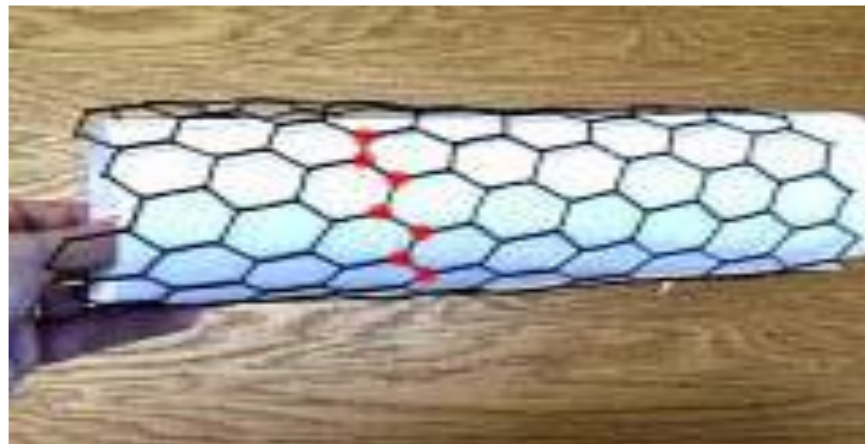
**1.4 Carbon Based Materials:** Carbon Nano-tubes are long, skinny cylinders of carbon, and were discovered in 1991 by Sumio Iijima. These are quite massive macromolecules that are utterly distinctive for their form, outstanding physical properties and size. Their intriguing structures have initiated a lot of excitement within the recent years and a considerable quantity of analysis has been dedicated to perceive their entirety. Presently, the physical properties are still within the method of being discovered and controversial. The Nano-tubes have a really wide selection of electronic, structural, and thermal properties that amend in line with the various forms of fullerene (defined by its length, diameter, and twist, or chirality). So as to form things way more fascinating, aside from possessing one cylindrical wall (SWNTs), these Nano-tubes will possess multiple walls (MWNTs) i.e., cylinders within the inner cylinders.



**Figure 1.9 Armchair arrangements of carbon atoms** <sup>[15]</sup>



**Figure 1.10 Zigzag arrangements of carbon atoms** <sup>[16]</sup>



**Figure 1.11 Chiral arrangements of carbon atoms** <sup>[17]</sup>

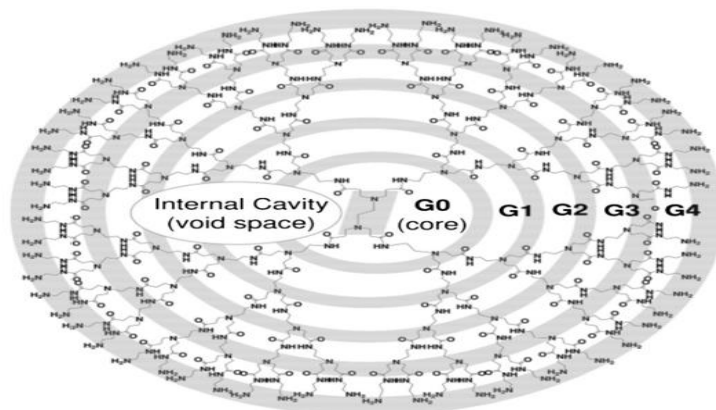
## 1.5 Metal Based Materials:

The existence of metallic Nano-particles in solution was first recognized by Faraday in 1857 and a quantitative explanation of their color was given by Mie in 1908.e.g Silver, Gold and other metallic Nano-Particles.

## 1.6 Dendrimers:

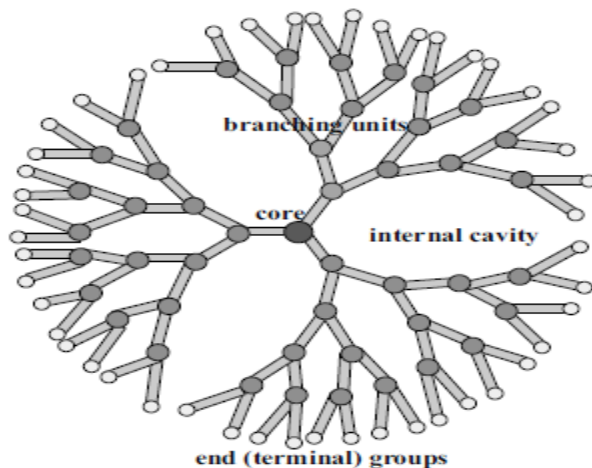
Dendrimers are unit Nano-sized, radially isosceles molecules with well-defined, homo-genised, and mono disperse structure consisting of tree-like arms or branches [18]. These hyper-branched molecules were 1st discovered by Fritz Vogtle in 1978, by Donald Tomalia and associates in the early Eighties, and at an equivalent time, however severally by martyr R. Newkome. The second cluster known as synthesized macromolecules ‘arborols’. Dendrimers may also be known as ‘cascade molecules’, but this term isn't the maximum amount established as ‘Dendrimers’. Dendrimers are unit nearly mono-disperse macromolecules that contain isosceles branching units built around a little molecule or a linear chemical compound core. ‘Dendrimer’ is just a field of study motif and not a compound. Polyionic Dendrimers don't have a persistent shape and will bear changes in size, shape, and flexibility as perform of accelerating generations. Dendrimers are unit hyper-branched macromolecules with a carefully tailored design, the end-groups (i.e., the groups reaching the outer periphery), which might be functionalized, therefore modifying their chemistry or biological properties. Dendrimers have gained a broad vary of applications in supramolecular chemistry, particularly in host-guest reactions and self-assembly processes. Dendrimers are unit characterized by special options that build them promising candidates for lots of applications. Dendrimers are unit extremely outlined artificial macromolecules, which are unit characterized by a mixture of a high number of practical teams and a compact molecular structure. The rising role of nerve fiber macromolecules for antitumor therapies and diagnostic imaging is outstanding [19]. The advantages of those well-defined materials make them the latest category of molecule Nano-scale delivery devices. Therefore, Dendrimers became a perfect delivery vehicle candidate for specific study of the results of chemical compound size, charge, and composition on biologically relevant properties such as macromolecule bi-layer interactions, toxicity, acquisition, blood plasma retention time, bio-distribution, and filtration.

There is an elementary distinction between these 2 construction ideas. In the divergent strategies, Dendrimers grows outwards from a multifunctional core molecule. The core molecule reacts with chemical compound molecules containing one reactive and 2 dormant groups giving the primary generation Dendrimers. Then the new boundary of the molecule is activated for reactions with additional monomers [20].



**Figure 1.12 Schematic representation of a generation G4 Dendrimers** [21]

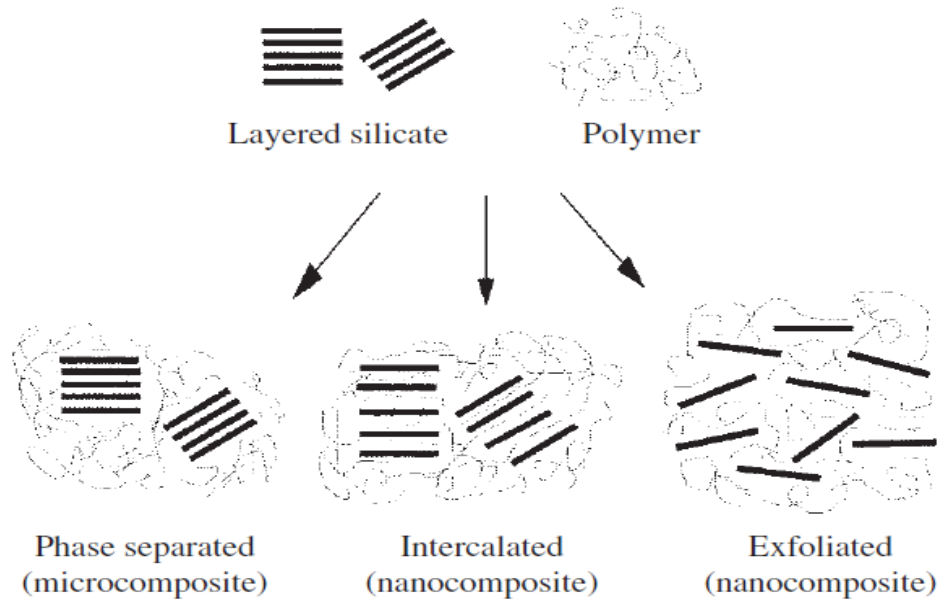
The above shown Dendrimers is with 64 amino groups at the periphery. These Dendrimers start from an ethylene di-amine core; the branches or arms were attached by exhaustive Michael addition to methyl acrylate followed by exhaustive aminolysis of the resulting methyl ester using ethylene di-amine.



**Figure 1.13 Representation of a fourth generation Dendrimers.** [21]

## 1.7 Composite:

Composites are shaped by incorporating multiple part components in an exceedingly material in such the way that the properties of the resultant material ar distinctive and not otherwise getable.



**Figure 1.14 Scheme of 3 main sorts of superimposed silicates in chemical compound matrix<sup>[22]</sup>**

The transition from micro-particles to Nano-particles yields dramatic changes in physical properties. Nano-scale materials have an outsized area for a given volume. Since several vital chemical and physical interactions are unit ruled by surfaces and surface properties, a Nano-structured material will have considerably totally different properties from a larger-dimensional material of constant composition. Within the case of particles and fibers, the area per unit volume is reciprocally proportional to the material's diameter, thus, the smaller the diameter, the larger the area per unit volume. Common particle geometries and their various surface area unit-to-volume ratios are shown in for the fiber and stratified material, the surface area/volume is dominated, especially for Nano-materials, by the primary term within the equation. The second term (2/l and 4/l) incorporates a terribly tiny influence (and is commonly omitted) compared to the primary term. Therefore, logically, a modification in particle diameter, layer thickness, or fibrous material diameter from the micrometer to Nano-meter vary, can have an effect on the surface area-to-volume magnitude relation by 3 orders of magnitude. Typical Nano-materials presently underneath investigation embody Nano-particles, Nano-tubes, Nano-fibers, fullerenes,

and Nano-wires. In general, these materials are unit classified by their geometries, broadly speaking the 3 categories are unit particle, layered, and fibrous materials. C, oxide Nano-particle, polyhedral oligomeric silsesquioxanes (POSS) may be classified as Nano-particle reinforcing agents while Nano-fibers and carbon Nano-tubes are unit samples of fibrous materials. Once the filler incorporates a Nano-meter thickness and a high ratio (30–1000) plate-like structure, it is classified as a stratified Nano-material (such as an organo silicate) [23].

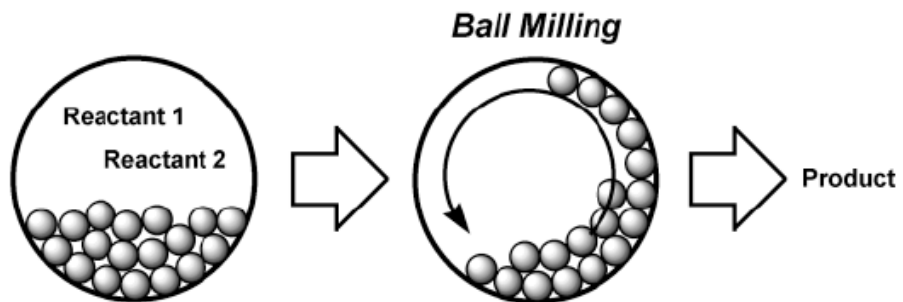
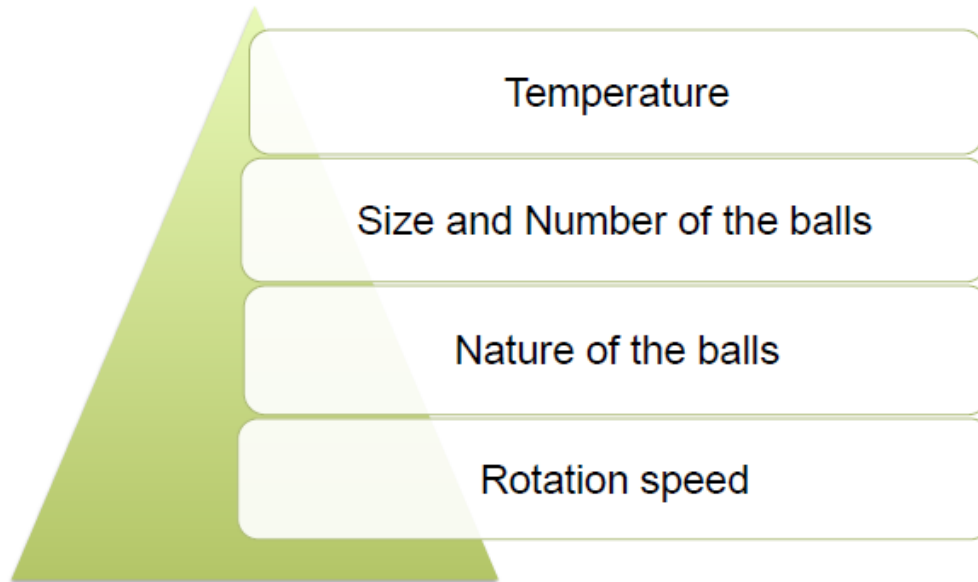
## **1.8 Method of preparation of Nano-materials:**

There is different-different method are use to Synthesized Nano-Particles Which depends on Requirements and availability of resources.

**(a) Physical methods:** Different physical methods are explained as follows.

### **(i) High Energy Ball Milling:**

Mechanical edge as a solid state synthesis sometimes performed mistreatment ball edge equipments that usually divided to “low energy” and “high energy” class supported the worth of evoked the energy to the powder mixture. The target of edge is to scale back the particle size and mixing of particles in new phases. The various form of ball edge is often used for synthesis of Nano-materials during which balls impact upon the powder charge. High-energy ball edge may be a convenient thanks to manufacture Nano-sized powders. It’s the foremost common technique reportable within the literature for the synthesis of inter-metallic Nano-particles. Before a mechanical edge is started, powder(s) is loaded at the side of many serious balls (steel or wolfram carbide) during an instrumentality. By smartly shaking or high-speed rotation, a high energy is applied on the powders due to collision with serious balls. A sort of ball mill is shown in Figure one. Mechanical ball edge has been wont to mix metallic element with metal and carbon so as to change its chemical properties and combustion behavior .The studies to create blends with metal used particles tens of micrometers in size.



**Figure 1.15 Major Parameter for Ball Milling and Ball Milling Process [24]**

Type of Ball milling

- Drum ball mills
- Jet-mills
- Bead-mills
- Horizontal rotary ball mills
- Vibration ball mills
- Planetary ball mills



**(ii) Pyrolysis:**

It involves transmutation of hydrocarbons like alkynes at 7000C within the presence of Fe-silica or Fe graphite catalyst underneath inert conditions [25].

**(iii) Laser Evaporation Method:**

It involves vaporization of plumb ago containing bit of Co and Ni, by exposing it to shaft of light at 12000C during a quartz tube reactor. Associate in nursing gas like Ar is allowed to pass into the reactor to comb the gaseous carbon atoms from the chamber to the copper collector, on that the Nano-materials condense [26].

**(iv) Carbon Arc Method:**

It is administrated by applying electrical energy (60- one hundred A and 20-25 V) arc between black lead electrodes of 10-20 $\mu$ m diameter [27].

**(b) Chemical methods:****(i) Sol-gel method:**

The sol-gel process may be described as: "Formation of associate degree chemical compound network through poly-condensation reactions of a molecular precursor during a liquid".

A sol may be a stable dispersion of mixture particles or polymers during a solvent. The particles are also amorphous or crystalline. Associate degree aerosol is particles during a gas phase, whereas a sol is particles during a liquid.

A gel consists of a 3 dimensional continuous network that encloses a liquid part in an exceedingly colloid, the network is constructed from agglomeration of colloidal particles in an exceedingly compound gel the particles have a compound sub-structure made by aggregates of sub-colloidal particles. Generally, the sol particles might interact by van-der Waals forces or chemical element bonds.

A gel may be fashioned from linking chemical compound chains. In most gel systems used for materials synthesis, the interactions are of a valence nature and therefore the gel method is irreversible. The gelation method is also reversible if different interactions are concerned.

The sol-gel methodology was developed within the Sixties chiefly owing to the requirement of latest synthesis strategies within the nuclear business. a way was required wherever dirt was reduced (compared to the ceramic method) and that required a lower sintering Temperature. Additionally, it ought to be potential to try to the synthesis by remote control [28].

### **(ii) Chemical vapour deposition**

It involves decomposition of vapour of hydrocarbons like gas, ethylene, acetylene, etc., at  $1100^{\circ}\text{C}$  in presence of catalysts like metallic element, Co, atomic number 26 supported on MgO [34].

### **(iii) Co-precipitation method**

### **(iv) Hydrothermal method**

## **1.9 Properties of Nano-Materials**

Three different Properties of Nano scale materials are as such [29].

### **(a) Electrical properties**

The electrical properties of Nano-materials vary between argentiferous to conductive materials. It depends on the diameter of the Nano-materials. The terribly high electrical physical phenomenon of Nano-material is as a result of minimum defects within the structure.

### **(b) Thermal conductivity:**

The thermal conduction of Nano-materials are terribly high, is thanks to the vibration of valency bonds. Its thermal conduction is ten times larger than the metal. The terribly high thermal conduction of Nano-material is additionally thanks to minimum defects within the structure.

### **(c) Mechanical properties:**

Nano-materials square measure terribly robust and face up to extreme strain. Most of the materials fracture on bending as a result of the presence of a lot of defects; however Nano-materials possess solely few defects within the structure.

## **1.10 Applications of Nano-materials:**

Below we tend to list some key applications of Nano-materials. Most current applications represent evolutionary developments of existing technologies: for the reduction in size of physical science devices [30].

### **(a) Sun-Creams and Cosmetics:**

The traditional chemical ultraviolet radiation protection approach suffers from its poor semi-permanent stability. An emollient supported mineral Nano-particle like pigment supplies many blessings. Titanium oxide Nano-particles has a comparable ultraviolet radiation protection property. Nano-sized pigment and philosopher's wool is unit presently employed in some sunscreens, as they absorb and mirror ultraviolet (UV) rays and however area unit clear to visible radiation then area unit additional appealing to the consumer. Nano-sized iron compound is gift in some lipsticks as a pigment. The utilization of Nano-particles in cosmetics has raised variety of considerations regarding client safety.

### **(a) Paints:**

Incorporating Nano-particles in paints might improve their performance, as an example by creating them lighter and giving them completely different properties, diluents paint coatings ('light weighting'), used as an example on craft, would cut back their weight, that may well be useful to the setting.

### **(c) Displays:**

The huge marketplace for giant space, high brightness, flat-panel displays, as utilized in TV screens and laptop monitors, is driving the event of some Nano-materials. Nano-crystalline metallic element selenide, metallic element compound, mineral and lead compound synthesized

by sol gel techniques square measure candidates for consequent generation of light-emitting phosphors.

#### **(d) Batteries:**

With the expansion in transportable equipment (mobile phones, laptop computer computers, remote sensors), there's nice demand for light-weight, high energy density batteries. Nano crystalline materials synthesized by sol-gel techniques ar candidates for extractor plates in batteries attributable to their foam-like (aero gel) structure, which may hold significantly additional energy than typical ones. Nickel-metal binary compound batteries product of Nano-crystalline nickel and metal hydrides are pictured to need less frequent recharging and to last longer attributable to their massive extent.

#### **(e) Catalysis:**

In general, Nano-particles have a high expanse, and therefore give higher chemical change activity. Chemical action is very important for the assembly of chemicals. Nano-particles function associate economical catalyst for a few chemical changes, owing to the extraordinarily massive surface to volume magnitude relation. Atomic number 78 Nano-particles area unit currently being thought-about within the next generation of automotive chemical change converters as a result of the terribly high expanse Nano-particles might scale back the number of atomic number 78 needed. Some chemical reactions are dole out mistreatment Nano-materials for instance, reduction of nickel compound to the bottom metal nickel.

#### **(f) Medicine:**

Nanotechnology has been a boon in medical field by delivering medication to specific cells victimization Nano-particles. The general drug consumption and aspect effects are lowered considerably by depositing the active within the morbid region solely and in no higher dose than required. This extremely selective approach reduces prices and human suffering. Engineering may facilitate to breed or to repair broken tissue. "Tissue engineering" may replace today's conventional treatments like organ transplants or artificial implants. As an example, bones are regrown on fullerene scaffolds. The utilization of gold in healthful preparations isn't new, within the Indian medical system known as Ayurveda; gold is employed in many preparations. One widespread preparation is named Saraswatharishtam, prescribed for the memory improvement.

Gold is additionally side in sure medical preparations for babies so as to reinforce their mental capability. Over 5000 years past, Egyptians used gold in medical specialty. In Alexandria, alchemists developed a strong mixture elixir referred to as liquid gold, a preparation that was meant to revive youth. In china, folks cook their rice with a gold coin so as to assist fill up gold in their bodies.

**(g) Sensors of gases:**

The gases like  $\text{NO}_2$  and  $\text{NH}_3$  is detected on the premise of increase in electrical physical phenomenon of Nano-materials. This can be attributed to extend in whole concentration in Nano-materials owing to charge transfer from Nano-materials to  $\text{NO}_2$  because the gas molecules bind the Nano-materials.

**(h) Food:**

Nanotechnology will be applied within the production, processing, safety and packaging of food. A Nano-composite coating method might improve food packaging by putting anti-microbial agents directly on the surface of the coated film. New foods are among the engineering science created client product coming back onto the market at the speed of three to four per week. per company data announce on PEN's information processing system, the vegetable oil, by Shemen Industries of Israel, contains associate additive referred to as "Nano-drops" designed to hold vitamins, minerals and phytochemicals through the system and carbamide.

**(i) Construction:**

Nanotechnology has the potential to create construction quicker, cheaper and safer. Automation of technology construction will leave the creation of structures from advanced homes to huge skyscrapers rather more quickly and at abundant lower value. The silicon dioxide ( $\text{SiO}_2$ ) is gift in typical concrete as a part of the conventional combine. When Nano silicon dioxide is further to concrete the particle packing is improved mechanical properties. The addition of Nano silicon dioxide to cement primarily based materials can even management the degradation of the basic C-S-H (calcium salt hydrate) reaction of concrete caused by Ca leach in water furthermore as block water penetration and thus result in enhancements in sturdiness. The strength of concrete can even be increase by adding iron ore ( $\text{Fe}_2\text{O}_3$ ) Nano-particles. Steel has been wide obtainable

material and includes a major role within the industry. The employment of technology in steel helps to boost the properties of steel. The Nano size steel manufacture stronger steel cables which might be employed in bridge construction. The glass is additionally a vital material in construction. There's plenty of analysis being meted out on the appliance of technology to glass. Oxide ( $\text{TiO}_2$ ) Nano-particles area unit wont to coat glazing since it's sterilizing and anti-fouling properties. The particles change state powerful reactions that breakdown organic pollutants, volatile organic compounds and microorganism membranes. Most of glass construction is on the outside surface of buildings. The light weight and warmth coming into the building through glass should be prevented. The technology will offer a stronger answer to dam lightweight and warmth returning through windows. Coatings are a vital space in construction. Coatings area unit extensively use to color the walls, doors and windows. Coatings ought to provide a protecting layer that is guaranteed to the bottom material to supply a surface of the specified protecting or purposeful properties. Technology is being applied to paints to obtain the coatings having self healing capabilities and corrosion protection below insulation. Since these coatings area unit hydrophobic and repels water from the metal pipe and might conjointly defend metal from salt water attack.

#### **(j) Agriculture:**

Applications of applied science have the potential to alter the whole agriculture sector and food trade from production to conservation, processing, packaging, transportation, and even waste treatment.

#### **(k) Energy:**

The most advanced engineering comes associated with energy are: storage, conversion, manufacturing enhancements by reducing materials and method rates, energy saving and increased renewable energy sources. Today's best star cells have layers of many completely different completely different } semiconductors stacked along to soak up light-weight at different energies however they still solely manage to use forty p.c. of the Sun's energy. Commercially out there star cells have abundant lower efficiencies (15-20%). engineering may facilitate increase the potency of sunshine conversion by victimization nanostructures.

## **Other Applications**

Some business merchandise is on the market today utilizing Nano-materials embrace stain resistant textiles and bolstered lawn tennis rackets. Firms like Kraft foods area unit heavily funding Nano-materials based mostly plastic packaging. Food can keep longer if the packaging is a smaller amount pervious to atmosphere. Coors Brewing Company has developed new plastic brewage bottles that keep cold for extended periods of your time.

### **1.11 Advances in Nano-materials:**

The history of Nano-materials began directly when the massive bang once Nano-structures were shaped within the early meteorites. Nature later evolved several alternative Nano-structures like seashells, skeletons etc. Nano-scaled smoke particles were shaped throughout the employment of fireplace by early humans. The scientific story of Nano-materials but began a lot of later [31]. One of the first scientific report is that the mixture gold particles synthesized by physicist as early as 1857. Nano-structured catalysts have conjointly been investigated for over seventy years. By the early 1940's, precipitated and treated silicon oxide Nano-particles were being factory-made and oversubscribed in USA and Federal Republic of Germany as substitutes for ultrafine carbon for rubber reinforcements [32]. Nano-sized amorphous silicon oxide particles have found large-scale applications in several fields. Every-day client merchandise starting from non-diary low ewer to automobile tires, optical fibers and catalyst supports. Within the Nineteen Sixties and 1970's auriferous Nano-powders for magnetic recording tapes were developed. In 1976, for the primary time, Nano-crystals produced by the currently standard inert- gas evaporation technique was printed by Granqvist and Buhrman. Recently it's been found that the Maya blue paint may be a Nano-structured hybrid material [33].

The origin of its color and its resistance to acids and bio-corrosion are still not understood however studies of authentic samples from Jaina Island show that the fabric is (formed is created) of acicular palygorskite (clay) crystals that form a super lattice with a amount of one 4 nm, with intercalates of amorphous salt substrate containing inclusions of metal (Mg) Nano-particles. The gorgeous tone of the blue color is obtained only if each these Nano-particles and therefore the super lattice are gift, as has been shown by the fabrication of artificial samples [34].

## Chapter 2

### **2.1 History:**

MgO is a vital material, that utilized in several applications like contact action, waste redress, paint, superconducting merchandise, anti-bacterial activities against food borne pathogens. Atomic number (12 metallic component metal) is IIA cluster component with number 12 and eight chemical element gas} is VIA cluster element with number 8.

The compound MgO has boiling and melting points as 3600°C and 2852°C. These compound materials will be ready by totally different synthesis ways like answer combustion, Co-precipitation, Sol-Gel, hydrothermal Salvo-thermal, Microwave motor-assisted Sol-Gel, inexperienced synthesis. In these ways Co-precipitation may be a one amongst the most effective methodology to synthesis Nano-particles while not agglomeration within the yield, size will be simply controlled [37].

Nano-particles have attracted an excellent attention in recent years owing to their distinctive physical and chemical properties like high strength with sensible thermal conduction, higher damping property and mechanical stability [18]. The high surface reactivity, high chemical and thermal stability of MgO makes it a promising material for the appliance in sensors, catalysis, paint and additives etc [38].

Mineral Nano-particles and small particles area unit for the most part used as a reinforcing chemical agent, likewise as a element in super conductors. Owing to the high surface reactivity, extremely chemical and high thermal stability with the chemical action properties, the atomic number 12 oxide Nano-particles have explicit interest [39].

The huge applications of mineral Nano-materials inclined to figure on this material, numerous types of fabrication techniques area unit employed to grow mineral Nano-particles like vapor-liquid-solid (VLS), chemical vapor deposition (CVD), plasma increased chemical vapor deposition (PECVD), periodical optical maser deposition (PLD), optical maser ablation, molecular beam grow in (MBE) and sputtering methodology are frequently utilized of these ways need warm temperature or refined and/or expensive instruments [40]. The chemical route,



Sol-gel processes, has become a promising possibility for the synthesis and large-scale production of Nano-structured materials likewise as mineral [41].

In particular, periclase (MgO), a flexible compound material with different properties finds intensive applications in contact action, ceramics, waste matter rectification, and as an additive in paint and superconductor product. Also, as a result of its terribly giant band gap (7.8 eV) [42], wonderful thermodynamically stability, low material constant and refractive index, it's been used for growing varied skinny film materials [43]. From literature it's been found that MgO nanostructures could also be synthesized by dehydration of Mg (OH)<sub>2</sub> or by decomposition of assorted Mg salts victimization totally different techniques like sol-gel method [44], thermal evaporation, flame spray transmutation, combustion aerosol synthesis, chemical vapour deposition, hydrothermal, and wetter ways etc. These ways involve advanced procedures, refined apparatus/ equipments, rigorous experimental conditions, high-temperature tempering.

Magnesium oxide is one in all the foremost representative materials among the oxides for vital technological application because of its wide band gap (7.8 eV) and a decent chemical and thermal (melting points is 2800°C) stability. In the same time MgO with its easy rock-salt structure is one the foremost vital metal oxides for theoretical and experimental studies. Optical properties of crystals are often basically changed by the presence of impurity ions and radiation induced defects. These impurity ions are often used as a research for understanding of defects structure. MgO contains a terribly easy lattice which may be a bunch for variety of transition metal ions. The local structure round the supplemental metals plays an important role for the chemical process [45].

## **2.2 Hydrogen storage properties of MgO:**

Hydrogen energy is that the promising energy supply for fuel as a result of its abundance, potency and environmental outcast. Three basic issues as well as H production, storage and burning should be resolved for the appliance of H energy. The H storage is one amongst the key challenges within the future property energy system. The storage system should meet the fundamental needs of high hydrometric and volu-metric densities beside quick mechanics and favorable physical science. In step with the target of DOE (U.S. Department of Energy), the hydrometric density of H storage ought to be half dozen World Trade Center. Moreover, the

storage materials ought to be able to reversibly adsorb/desorb  $H_2$  within the temperature vary of 20–50 K below moderate pressures (maximum one hundred atm) [46]. The standard modes of Hydrogen storage by propellant or within the liquid type are unit high prices and with safety issues. Compared to gaseous and liquid H storage, the utilization of solid state materials for H storage has been paid nice attentions because of its blessings in terms of safety and H contents. In step with the surface assimilation strength, the storage materials may be divided into physical and chemical surface assimilation materials. For physical surface assimilation materials, the interaction between solid materials and  $H_2$  is simply too weak (adsorption energy but zero 1 eV) to catch a lot of  $H_2$  [47]. Therefore the density of H storage is quite low. Though the chemical surface assimilation materials will get higher storage density, the stronger interaction (adsorption energy unremarkably larger than two to three eV) ends up in the problem for natural process of  $H_2$ . None of solid materials will meet the necessities of high storage density and quick natural process of H at the same time. To get moderate surface assimilation strength is that the one amongst the key factors for H storage in step with the thermo dynamical calculations, the optimum surface assimilation energy for H storage materials and  $H_2$  is 0.1 to 0.2 electron volt at 298 K and 1.5–30 bar, that is between the physical and chemical surface assimilation. To explore the strategy of H storage with acceptable surface assimilation strength may be a basic and necessary subject [48].

MgO is engaging for each basic and applicable analysis areas. The MgO has been in central theme of analysis because it offers applications in micro-electronic circuits, sensors, electricity devices, fuel cells, and semiconductor merchandise. MgO Nano-particles exhibiting (111) faces area unit in and of itself unstable and will endure a structural transformation [49]. In its bulk state, MgO may be a extremely ionic compound and a large band gap (7.8 eV) material. For little Nano-particles of MgO, a discount within the band gap may well be measured by victimization optical absorption techniques [50]. Bulk MgO is extremely insulating crystalline solid with cubic form NaCl crystal structure and displays a spread of properties like chemical immobility, electrical insulation, optical transparency, extreme temperature stability, high thermal conduction, secondary lepton emission, appropriate for surface assimilation and dissociation of polar molecules, and waste material redress, trying to the necessary applications in bulk style of MgO, numerous strategies are adopted for the preparation of Nano-sized materials as well as chemical precipitation, sol–gel synthesis and hydrothermal reaction [51].

## **2.3 Application of MgO and MgO's Composites:**

### **(a) Anti-Bacterial Agent:**

Nano-particles are unit recognized as bactericide agents because of their size, structure, and surface properties. Thus, technology offers some way to enhance the activity of inorganic bactericide agents. Nano-particles like ZnO, MgO and CaO are investigated as inorganic bactericide agents. MgO is a very important inorganic material with a good band-gap. It's been utilized in several applications like chemical action, catalyst supports, waste product correction, refractory materials and adsorbents, additive in serious fuel oils, reflective and anti-reflecting coatings, superconducting and ferroelectric skinny films because the substrate, superconductors and metal particle batteries, etc. In medication, MgO is employed for the relief of symptom, sore abdomen, and for bone regeneration. Recently, MgO Nano-particles have shown promise for application in growth treatment. MgO Nano-particles even have hefty potential as Associate in nursing bactericide agent [52].

### **(b) Anti-Bacterial Mechanism of MgO:**

The exact medicinal drug mechanism of MgO Nano-particles remains unknown. variety of mechanisms, like the formation of reactive atomic number 8 species (ROS), the interaction of Nano-particles with bacterium, later damaging the microorganism cell, Associate in Nursing a basic impact are projected to elucidate the medicinal drug mechanism of MgO Nano-particles. Many similar mechanisms are projected to elucidate the restrictive impact of silver Nano-particles on bacterium. Silver has long been famed to cause microorganism inhibition. The medicinal drug mechanisms of silver embody induction of aerophilous stress because of generation of ROS, which can cause the degradation of the membrane structure of the cell. Unleash of ions from the surface of Nano-particles has been reportable to cause microorganism death because of binding to semi-permeable membrane, several studies have indicated that the medicinal drug mechanism of MgO Nano-particles is because of the formation of ROS like superoxide ( $O^{2-}$ ). It's been reportable that the rise of the area of MgO particles ends up in a rise of the  $O^{2-}$  concentration in answer and therefore ends up in a simpler destruction of the cell membrane of the bacterium. However, once the particle size of MgO is below fifteen nm, the aggregation impact becomes terribly vital because of the terribly high surface energy of the

particles. the massive size of collective MgO inhibits the interaction with bacterium and particles in order that the disinfectant potency becomes lower. Some researchers reportable that the sweetening of the medicinal drug activity of MgO Nano powder against E. coli was because of the generation of an oversized quantity of  $O_2^-$  by the surface of MgO powder. Recently, researchers evaluated the medicinal drug activity of MgO Nano-particles against the gram-negative bacterium. MgO Nano-particles exhibited medicinal drug activity with MIC of 500  $\mu\text{g}/\text{Ml}$  against E. coli and 1000  $\mu\text{g}/\text{mL}$  for P. aeruginosa and S. aureus. It had been prompt that the mechanism of the medicinal drug activity of MgO Nano-particles could also be lipide peroxidation and ROS because of the presence of defects of atomic number 8 vacancy at the surface of the Nano-particles [53].

### **(c) MgO application in Cement Industries:**

Cement could be a basic material used for building and applied science construction. Portland cement (PC) production generates a considerable quantity of  $\text{CO}_2$  from the decomposition of stone at  $\sim 1450^\circ\text{C}$ , the combustion of fuels within the oven, still as from power generation. it's calculable that the cement production accounts for 5–8% of artificial  $\text{CO}_2$  emissions. As a result, varied tries to cut back cement clinker production are created, one in all that is that the intensive usage of commercial by-products. Alkali-activated binders supported ground coarse blast-furnace scum (GGBS) square measure property alternatives to computer because of their low initial cost of capital of the staple and therefore the saving of energy and resources leading to lower  $\text{CO}_2$  emission. It's calculable that over eightieth of  $\text{CO}_2$  will be reduced by subbing alkali-activated binders for computer [54].

However, many problems regarding the employment of robust alkali exist, that prevents the widespread use of alkali-activated binders together with over-rapid setting, the issue of handling the caustic alkali and uneconomical potency during this sense, alkaline-earth hydroxides like  $\text{Ca}(\text{OH})_2$ ,  $\text{Sr}(\text{OH})_2$  and  $\text{Ba}(\text{OH})_2$  were studied and located to be ready to facilitate the association of GGBS[55].

Recently, reactive MgO emerged as a good substance for GGBS. Researchers according that reactive MgO inflated the association of GGBS considerably and scum activated by 10-20% MgO outperformed corresponding lime-activated scum with strength values of up to four times higher [56].

The impact of MgO within the alkali-activated scum system has been investigated by several researchers, either within the sort of internal MgO from scum composition, or external MgO addition. It ought to be noted that since the GGBS is typically created between 1400-1600°C, the interior MgO is classified as dead burned MgO, whereas reactive grade (calcined beneath 1000°C) is usually designated because the MgO else outwardly. it's investigated that the impact of MgO content (internal) on the performance of alkali activated cement and unconcealed that the most association merchandise square measure Ca salt hydrate (CSH) gel and hydrotalcite ( $\text{Mg}_6\text{Al}_2\text{CO}_3(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$ )-like phases (Ht). Higher content of MgO created additional Ht that is additional voluminous, leading to higher strength. Reactive MgO was accustomed replace four-hundredth of scum, activated with  $\text{K}_2\text{CO}_3$  and water glass, and it had been found that the strength was solely slightly below the management [57].

The light-burnt dolomite (mainly contains reactive MgO and CaO) was else in metallic element silicate-activated scum and ash blends, and therefore the results indicated that association of reactive MgO slashed the shrinkage of the paste, and no interaction between MgO and alternative parts was discovered by the micro-structural analysis. Though it's best-known that the characteristics of reactive MgO vary considerably, which can have an effect on the activation method, none of those studies investigated or bestowed the impact of the characteristics of MgO on the performance of MgO-GGBS blends [58].

#### **(d) MgO in Zinc Polycarboxylate Dental Cement Preparation:**

During the previous couple of years, synthesis of Nano-structured compound materials are attracted significant attention. The metal oxides are extraordinarily vital technological materials to be used in electronic and photonic devices and as catalysts in chemical industries. They're additionally usually used as active materials of dental cements. In recent years, researchers have targeted a lot of on the synthesis of each Nano-particles and Nano-composite of ZnO and MgO as a result of their application in advanced technologies. Varied chemistry techniques are utilized to construct Nano-sized ZnO and MgO particles. Many techniques are additionally developed to organize Nano-composite of ZnO/MgO. This Nano-composite has attracted abundant attention

as a result of it's a bigger band gap than. However, most of the techniques would like high temperatures and perform underneath a pricey inert atmosphere [59].

One of the primaries with chemicals adhesive dental materials is metallic element poly-carboxylate cement. Metallic element poly-carboxylate cement could be a water-based material that hardens following associate degree acid-base reaction between zinc-rich powder associate degreed an solution of poly-acrylic acid the most advantage of this cement lies in its robust adhesion to dentin [60].

However, its low operating time (~1-2 min) and comparatively low compressive strength (40-70 N/mm<sup>2</sup>), causes the cement to be used solely as a base or cementing restorative. Some makes an attempt are created to beat these disadvantages by modifying poly-carboxylic acid exploitation copolymers of carboxylic acid, adding polyvinyl phosphonic acid or incorporating varied fillers into the zinc-containing powder exploitation metal compounds, silica, periclase [61].

### **(e) MgO Nano-composites as Insulation for HVDC Cables:**

Since the electrical power demand is apace increasing in associate degree populated area within the world, a protracted distance dc power transmission becomes a lot of and a lot of vital. To develop the long distance power transmission cable, the insulating materials with smart performance underneath a high field of force square measure desperately required. In recent years, chemical compound Nano-composites (PN), a replacement insulant, are attracting more and a lot of attentions of researchers. Chemical compound Nano-composites square measure outlined because the second generation of what we tend to decision stuffed resins within the insulation engineering. Many p.c of Nano-sized inorganic filler extra into chemical compound will improve numerous properties considerably, compared with an oversized quantity (the order of fifty wt %) of micro-sized filler. several analysis results recommend that Nano-MgO square measure chosen because the inorganic filler of chemical compound Nano-composites for top voltage electrical energy (HVDC) cables. The LDPE/MgO Nano-composite material that is formed of low-density polythene (LDPE) and Nano-sized mineral (MgO) filler is one amongst such fresh developed materials. The LDPE/MgO Nano-composites bear a better breakdown strength and lower house charge accumulation underneath high dc stress than those of LDPE. house charge distribution in insulating materials for the dc power cables underneath high dc

stress is incredibly crucial. Once a dc voltage is applied to the chemical compound insulant, the house charge accumulates in it and consequently, the electrical field within the insulant is usually increased, which can lead to associate degree sudden breakdown. Several analysis works have established that Nano-MgO will suppress house charge formation within the LDPE expeditiously. However most of them are targeted on the electrical properties and suppression mechanism. The preparation method of LDPE/MgO Nano-composites has not been mentioned nonetheless. During this paper, totally different forms of silane coupling agent treated Nano-MgO influence on house charge distribution of LDPE Nano-composites underneath high field of force were measured [62].

## Chapter 3

### **3.1 Literature Survey**

The literature survey in this chapter presents an overview of the processing of MgO Nano composite. The relationships between the properties of MgO Nano composite and their constitutions are also covered, with particular focus on MgO Nano-Composite and Their Present and future applications.

Mohd Sufri Mastuli *et.al.* have worked on Growth mechanisms of MgO Nano-crystals, In the preparation of Nano-structured materials, it's vital to optimize synthesis parameters so as to get the required material. This work investigates the role of complexing agents, oxalic acid, within the production of MgO Nano-crystals. it's found that the thermal profiles of the precursors will reveal the consequences of crystal growth throughout thermal hardening. X-Ray diffraction confirms that the ultimate product are pure, single part and of solid form. It's conjointly found that complexing agents will have an effect on the speed of crystal growth. The structures of the oxalic acid, still because the complexation sites play vital roles within the formation of the Nano-crystals. The complexing agents influence the speed of growth that affects the ultimate crystallization size of the materials. Astonishingly, it's conjointly found that oxalic acid act as surfactants inhibiting crystal growth even at warmth of 950°C and a protracted hardening time of thirty six h. The crystallization formation routes are planned to be via linear and branched chemical compound networks attributable to the various structures of the complexing agents [37].

Mohd Sufri Mastuli *et.al.* have also worked on Effects of Cationic Surfactant in Sol-gel Synthesis of Nano Sized Magnesium Oxide. They used cationic surfactant (cetyl trimethyl ammonium bromide, CTAB) in the sol-gel reaction to minimize agglomeration of the nanoparticles. Both the samples (MgO and MgO-CTAB) were characterized using simultaneous thermo gravimetric analyzer (STA), X-ray diffractometer (XRD), field emission scanning electron microscope (FESEM) and nitrogen adsorption-desorption measurement. Growth of the samples via a sol-gel method is discussed and confirmed using the STA results. The precursor formed was identified as magnesium tartrate and decomposed to MgO and MgO-CTAB after the calcinations, and gave a single phase of samples as shown by the XRD patterns. The used of



CTAB in this sol-gel method gives the MgO Nano-particles with less agglomeration. This was proved by the FESEM micrographs, the MgO-CTAB has spherical shape and the agglomeration seems to be less than the MgO. It suggests that the cationic surfactant controls the morphology of the samples [38].

Hongji Li *et.al.* having the work of Synthesis of MgO Nano-crystals for biosensing applications. Sample were Synthesized using a simple direct current arc plasma jet chemical vapor deposition method. Magnesium nitrate was used as supply material and Mo film was used as a substrate and catalyst. The high-temperature plasma created ensured speedy synthesis of the MgO Nano-crystals. The as-prepared Sample were characterized by FESEM, HRTEM, XRD-diffraction, energy-dispersive chemical analysis, FTIR, UV-Visible spectrophotometry, and photoluminescence measurements. The as-synthesized samples were found to carries with it cubical MgO nanobelts and Nano-sheets with giant surface areas and low coordination chemical compound ions, and contained various contacts, rough edges, vacancies, and doping defects. The nanostructures exhibited glorious chemistry sensing properties with high sensing sensitivity toward water-soluble vitamin. Their high electro-catalytic activity was attributed to the result of defects and therefore the surface negatron transfer ability of the one-dimensional MgO Nano-belts [39].

M. S. Wagh *et.al.* worked on Preparation of MgO Nanostructure in Powder form via Sol-gel Method. The sol-gel process for the synthesis of oxide base materials is currently attracting since it is a cheap and low temperature technique that allows for the fine control on the product. For the preparation of MgO powder magnesium chloride was used as precursor by sol-gel technique. This process involved hydrolysis and condensation. The prepared powder was fired at different firing temperature. Then prepared powder was characterized with X-ray and SEM techniques. The sol-gel method for the synthesis of metal oxide based compound materials is presently attracting since it's an inexpensive and vasoconstrictive technique that permits for the fine management on the merchandise. For the preparation of MgO powder metal chloride was used as precursor by sol-gel technique. This method concerned reaction and condensation. The ready powder was discharged at completely different firing temperature. Then ready powder was characterized with X-ray and SEM techniques [40-41].

K. Mageshwari *et.al.* has been Prepared MgO Nano-particles for applications of effective photo-catalytic. MgO Nano-particles were synthesized by template-free reflux condensation approach. XRD and FTIR confirmed the formation of single phase MgO exhibiting simple cubic crystal structure. SEM and TEM analysis verified the formation of MgO Nano-flakes. The optical band gap of MgO nanoparticles calculable from UV-Visible Spectrophotometer was within the vary from 5.40 to 5.45 eV. PL spectra of MgO Nano-particles showed visible emissions owing to the formation of defects within the band gap region of MgO. Synthesized MgO Nano-particles exhibited considerable photo-catalytic activity for the degradation of acid-base indicator and stain dyes underneath ultraviolet radiation ultraviolet illumination UV actinic radiation actinic ray} light irradiation. In Results contestable that the origin of photo-catalytic activity in MgO arises to owing the existence of high concentration of native defects on the surface of MgO [42].

Arpita Hazra Chowdhury *et.al.* have synthesized grainy rod-like porous MgO. Porous magnesia (MgO) with grainy rod-like small structure was synthesized by hydrothermal method at one hundred eighty °C/5 h in the presence of urea. The as-prepared particles crystallized as  $Mg_5(CO_3)_4(OH)_2 \cdot 5H_2O$  that remodeled to MgO phase at 600–700 °C. The BET surface area, total pore volume and average pore diameter of 600 °C-treated sample were found to be  $46 \text{ m}^2 \text{ g}^{-1}$ ,  $0.34 \text{ cm}^3 \text{ g}^{-1}$  and 30.4 nm, respectively. Micro structural analysis indicated grainy rod-like micro structure (aspect ratio 10–30) in which the Nano grain-like particles (20–50 nm) were assembled like architectural design [43].

M.R. Bindhu *et.al.* has been Worked on MgO Nano particles and its optical properties for antibacterial applications, they used wet chemical reaction method for sample Preparation. The structural, optical and morphologies of MgO Nano-particles have been characterized by X-ray diffraction, UV–Vis Spectro photometry, SEM and TEM, and they got well-dispersed spherical Nano-particles with the average size of 16nm. The SEM and TEM characterization result verified the formation of almost spherical shaped MgO Nano- particles. X-Ray diffraction shows the face cubic centered structure of the MgO Nano-particles. The observed anti- bacterial properties, suggest the possible utilization of prepared Nano-particles in water purification [44].

Yue-Hong Yin *et.al.* worked on synthesis of MgO Nano composites and its hydrogen storage properties. They synthesized  $(MgO)_9$  with a rock salt structure, a magic number cluster of

(MgO)<sub>n</sub> exhibits high stability. They studied that the hydrogen storage properties of (MgO)<sub>9</sub> under an external electric field are explored by DFT calculation. The Characterization result verified that H<sub>2</sub> can be absorbed on single Mg/O atoms because the (MgO)<sub>9</sub> and H<sub>2</sub> are effectively polarized by the external electric field, the adsorption strength of H<sub>2</sub> at certain adsorption sites is substantially grow-up. The adsorption energies of H<sub>2</sub> on three-coordinated Mg/O atoms increase from -0.071/-0.056 eV in the absence of an electric field to -0.186/-0.237 eV under a field intensity of 0.025 a.u. Under the field, (MgO)<sub>9</sub> can absorb a maximum of 18 H<sub>2</sub> molecules, and the corresponding mass density of hydrogen storage reaches 9.1 wt%. Our results suggest that subjecting the (MgO)<sub>9</sub> to an external electric field is a potential hydrogen storage method. In this paper, the interaction mechanism between H<sub>2</sub> and (MgO)<sub>9</sub> under the external electric field is also investigated through an electronic structure analysis [45-46].

## **Chapter 4**

This Chapter contains a close description of preparation of MgO Nano-particles by exploitation Sol-Gel methodology. Totally different-different MgO Nano Composite samples calcined at individual temperature for a hard and fast and are characterized by exploitation numerous characterization techniques.

### **4.1 Preparation and Characterization of MgO Nano-Composite:**

In the past decade, the distinctive properties of Nano-materials created an interest among the researchers to plot straightforward and cheap techniques for synthesis of nanostructures that have technological importance. Metal chemical compound Nano-materials with high area have attracted extensive interest for research project attributable to their potential applications within the field of Nano physical science, optoelectronics and sensing devices. Above all, periclase (MgO), a flexible chemical compound material with various properties finds in depth applications in chemical change, ceramics, waste product redress, and as an additive in paint and superconductor product. Also, as a result of its terribly massive band gap (7.8 eV), wonderful thermodynamically stability, low material constant and ratio, it's been used for growing varied skinny film materials.

From literature it has been found that MgO nanostructures is also synthesized by dehydration of  $\text{Mg}(\text{OH})_2$  or by decomposition of varied metallic element salts exploitation totally different techniques like sol-gel technique, thermal evaporation, flame spray transformation, combustion aerosol synthesis, chemical vapour deposition, hydrothermal, and surface-active agent ways etc. These ways involve advanced procedures, refined apparatus/ equipments, rigorous experimental conditions, high-temperature tempering. a scientific study of the structural, morphological and optical properties of the calcined and as-synthesized MgO Nano-particles was then disbursed by exploitation XRD, FTIR, EDX, SEM and UV–VIS qualitative analysis techniques.

## 4.2 Experimental Technique:

There are several synthesis techniques for preparation of Nano-materials that are delineated in Previous Chapter. However, for the current work, an easy and versatile technique, referred to as Sol-gel technique has been employed in the current work that is delineated below.

MgO Nano crystals have been synthesized by using sol-gel technique. Complexing agents are widely used in various industrial processes and household products throughout the world. Complexing agents, also called “sequestrants” are substances which form chemical complexes with metallic ions. All the chemicals were used analytical grade and directly used as received without further purification. Magnesium acetate tetra hydrate,  $\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ , Oxalic acid dehydrate  $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , and absolute ethanol  $\text{C}_2\text{H}_5\text{OH}$  were used to formation of MgO nanostructures. The MgO samples were synthesized using Oxalic acid as complexing agent and further CTAB and SLS as surfactant and growing agents. Magnesium acetate tetra hydrate of mass 53.2075 gm was initially dissolved 150 ml of absolute ethanol under constant stirring.

In the beginning 8 gm  $\text{H}_2\text{C}_2\text{O}_4$  (solid) + Dist. HOH 100 ml and 17.76 gm  $(\text{CH}_3\text{COO})_2 \text{Mg} \cdot 4\text{H}_2\text{O}$  + 50 ml  $\text{C}_2\text{H}_5\text{OH}$  drop wise added till ph 5 obtained and continuously stirred. At ph value 5 found white gel, then left this white gel over a night for further gelation. Again dried this white gel for 24 Hrs. at  $100^\circ\text{C}$  then carried Annealing process at  $950^\circ\text{C}$  for 36 hrs.

After annealing annealed substrate divided in few other parts like sample ‘1’, ‘2’, ‘3’, ‘4’, ‘5’, ‘6’ etc.

Sample (1) was taken in a quartz tube and then heated in muffle furnace to a temperature of  $950^\circ\text{C}$  for 36 hours.

Again the Sample (2) was taken in the tube and kept in the muffle furnace at temperature of  $950^\circ\text{C}$  for 3hrs.

Another Sample (3) was kept at vacuum oven and temperature is raised to  $250^\circ\text{C}$  for 3 hrs.

Sample (4) kept at muffle furnace for 3hrs ( $200^\circ\text{C}$ ).

Sample (5) and CTAB 0.1 gm each kept at for 3hrs ( $400^\circ\text{C}$ ).

Sample (6) and SLS 0.1 gm each kept at for 3hrs ( $400^\circ\text{C}$ ).

# Preparation method

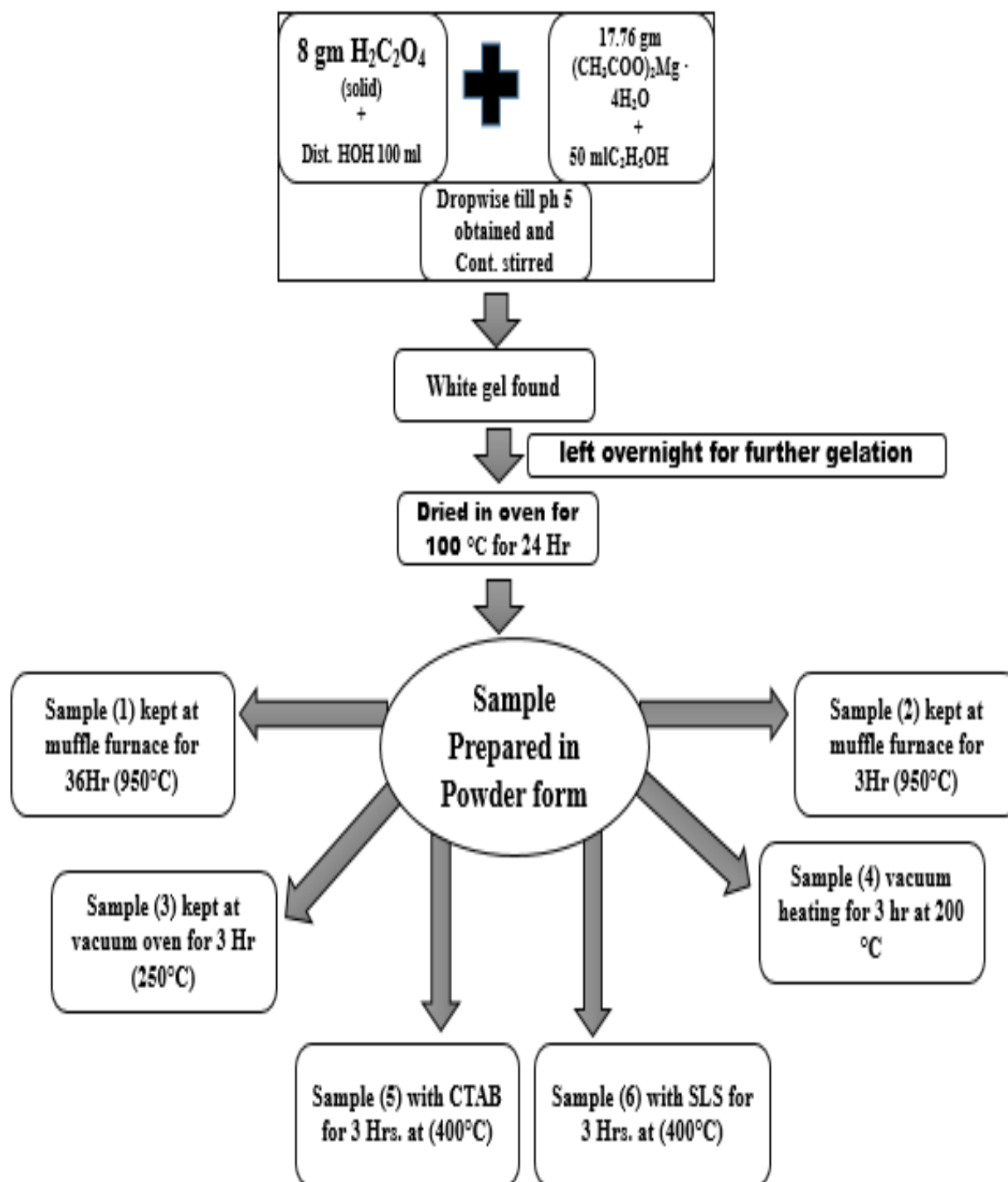
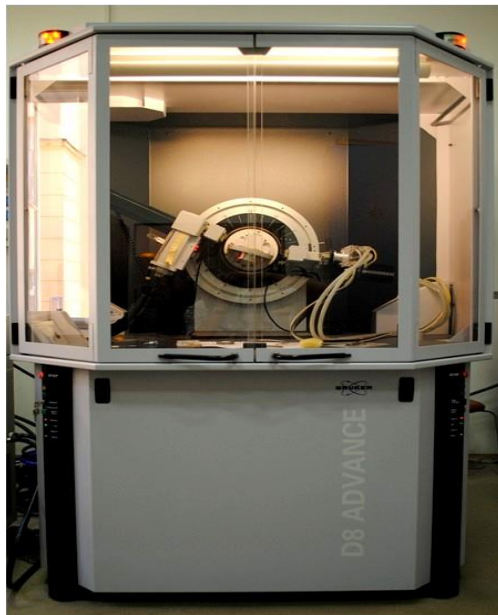


Figure 4.1 Flow Chart: Synthesis of MgO Nano crystals via a Sol-gel method using different complexing Agents.

### 4.3 Characterization Techniques and used Instruments:

The MgO Nano-particles were analyzed by XRD using a Bruker D8 Advance powder Diffractometer with CuK $\alpha$  radiation ( $\lambda=1.5406\text{\AA}$ ) in the range of  $20^{\circ}$ – $50^{\circ}$ .



**Figure 4.2 Bruker D8 Advance Powder Diffractometer @DTU**

The band gap energy of Nano-particles was estimated by absorption spectra recorded using Cary-300 (Agilent Technologies Cary Series) spectrophotometer.



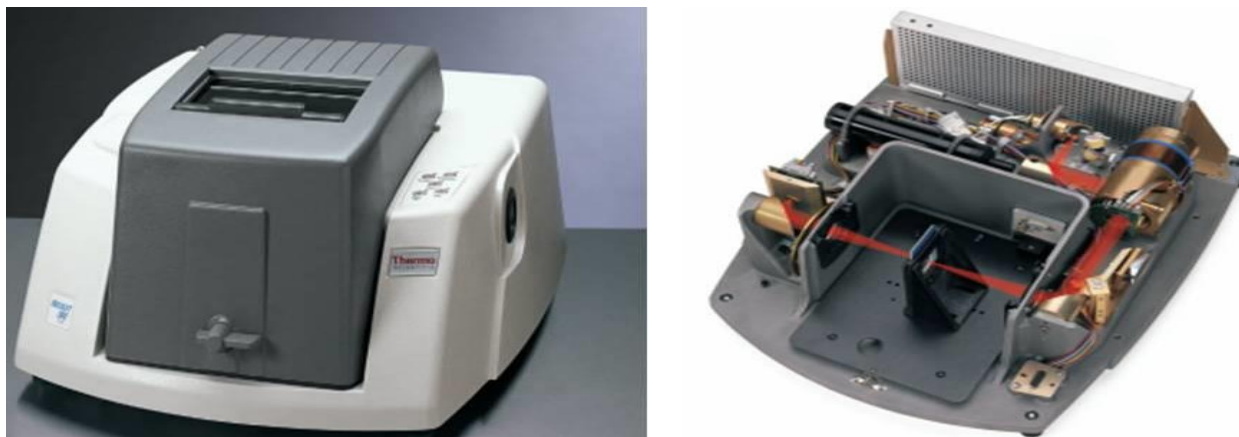
**Figure 4.3 Cary-300 Spectrophotometer @DTU**

The size and morphology of the Nano-composites were also observed from images recorded from Hitachi S-3700N Scanning Electron Microscope. The Energy Dispersive Spectroscopy Characterization also done by this Instrument.



**Figure 4.4 Hitachi S-3700N Scanning Electron Microscope @DTU**

FTIR spectra were recorded on a Thermo Scientific Nicolet-380 Series FTIR Spectrometer. The details of experimentation are described in previous chapter and results obtained are discussed below.



**Figure 4.5 Thermo Scientific Nicolet-380 Series FTIR Spectrometer @DTU**



## Chapter 5

### 5.1 Results and discussion

Results of the Synthesized Nano-Composite are discussed as follows on the basis of different-different characterization graphs and images. All the characterizations are done in DTU's laboratories.

### 5.2 X-ray diffraction Analysis:

X-ray powder diffraction (XRD) patterns of the MgO Nano-composites calcined at fixed Different-different temperature for respective durations and those calcined for fixed duration at different calcined temperatures were recorded and presented in **Figures 3.6**.

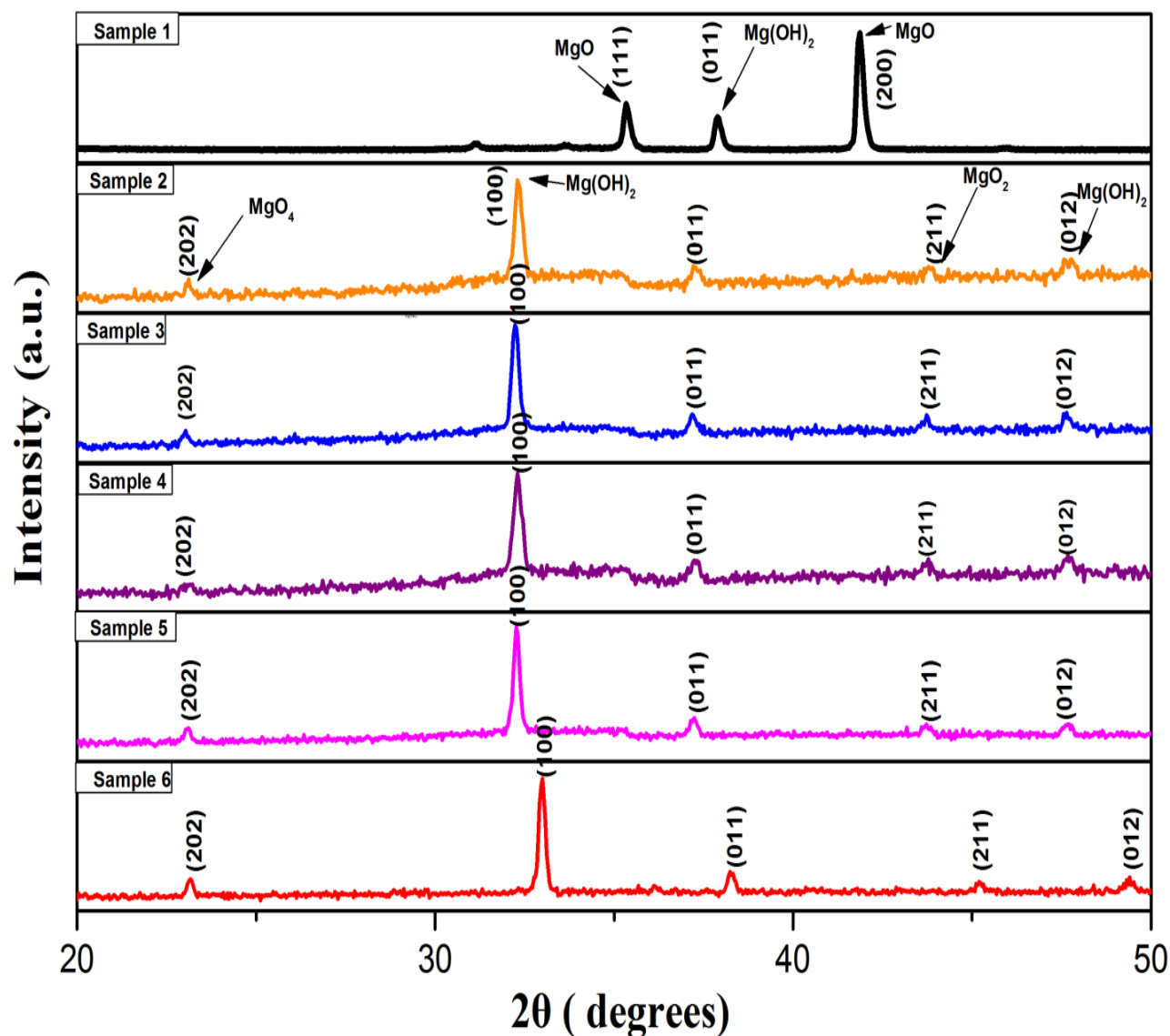


Figure 5.1 XRD Characterization Result of MgO Composite

The peaks in XRD pattern of as prepared MgO Composites sample observed at  $2\theta$  which match very well with those of JCPDS data file of MgO no. For MgO the most intense peak was observed at  $2\theta = 43.12^\circ$  and for Composites the most intense peak was observed at  $2\theta = 33.10^\circ$ . On calcination, the position of peaks does not change significantly. Width (FWHM- full width at half maximum) of this peak was used to determine the size (D) of crystallite/ particle using Debye-Scherrer's equation

$$D = 0.9 \lambda / \beta \cos\theta$$

Here D is the crystallite size,  $\lambda$  is the wavelength of X-ray beam,  $\beta$  is the full width at half maximum of the most intense peak, and  $2\theta$  is the Bragg diffraction angle of the most intense peak.

For **Sample (1)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 36.360411 degree, 39.086 degree and 43.3180 degree, and the respective crystallize size are at these angle are 33.023 nm, 32.307 nm and 34.90 nm. After all calculation we have taken average of all 33.41 nm.

For **Sample (2)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 23.326 degree, 33.1167 degree and 38.36384 degree, and the respective crystallize size are at these angle are 39.07 nm, 30.6 nm and 27.83 nm. After all calculation we have taken average of all crystallize size which is found 32.5025 nm.

For **Sample (3)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 33.0400 degree, 38.3180 degree and 55.28146 degree, and the respective crystallize size are at these angle are 29.7955 nm, 34.3422 nm and 31.352 nm. After all calculation we have taken average of all crystallize size which is found 31.8243 nm.

For **Sample (4)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 33.33.1201 degree, 55.3615 degree and 65.9244 degree, and the respective crystallize size are at these angle

are 27.53387 nm, 26.30530 nm and 22.485 nm. After all calculation we have taken average of all crystallize size which is found 25.44139 nm.

For **Sample (5)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 23.1670 degree, 33.0011442 degree and 38.245995 degree, and the respective crystallize size are at these angle are 38.223 nm, 35.996 nm and 35.3238 nm.

For **Sample (6)** the XRD characterization graph we have taken three prominent peaks to calculate average crystallize size. These peaks are consider at angle when  $2\theta$  is 33.08009 degree, 38.3970 degree and 55.2814 degree, and the respective crystallize size are at these angle are 37.3192 nm, 33.1064 nm and 36.4395 nm. After all calculation we have taken average of all crystallize size which is found 35.6217 nm. After all calculation we have taken average of all crystallize size which is found 36.513 nm.

### **5.3 Fourier Transform Infrared (FTIR) Study:**

**Sample (1)** In the above FTIR characterization result peaks of Mgo Found at 518.2 which denoted that is from Alkyl helide Group and the bonding is C-Br stretch.

**Sample (5) MgO+CTAB** In the above FTIR characterization result peaks Found at 520.5 and 572.1 which denoted that is from Alkyl helide Group and the bonding is C-Br stretch.

**Sample (6) MgO+SLS** In the above FTIR characterization result peaks Found at 991.6 and 1163.2 which denoted that is from alkenes Group and aliphatic amines group the bonding is =C–H bend and C–N stretch respectively.

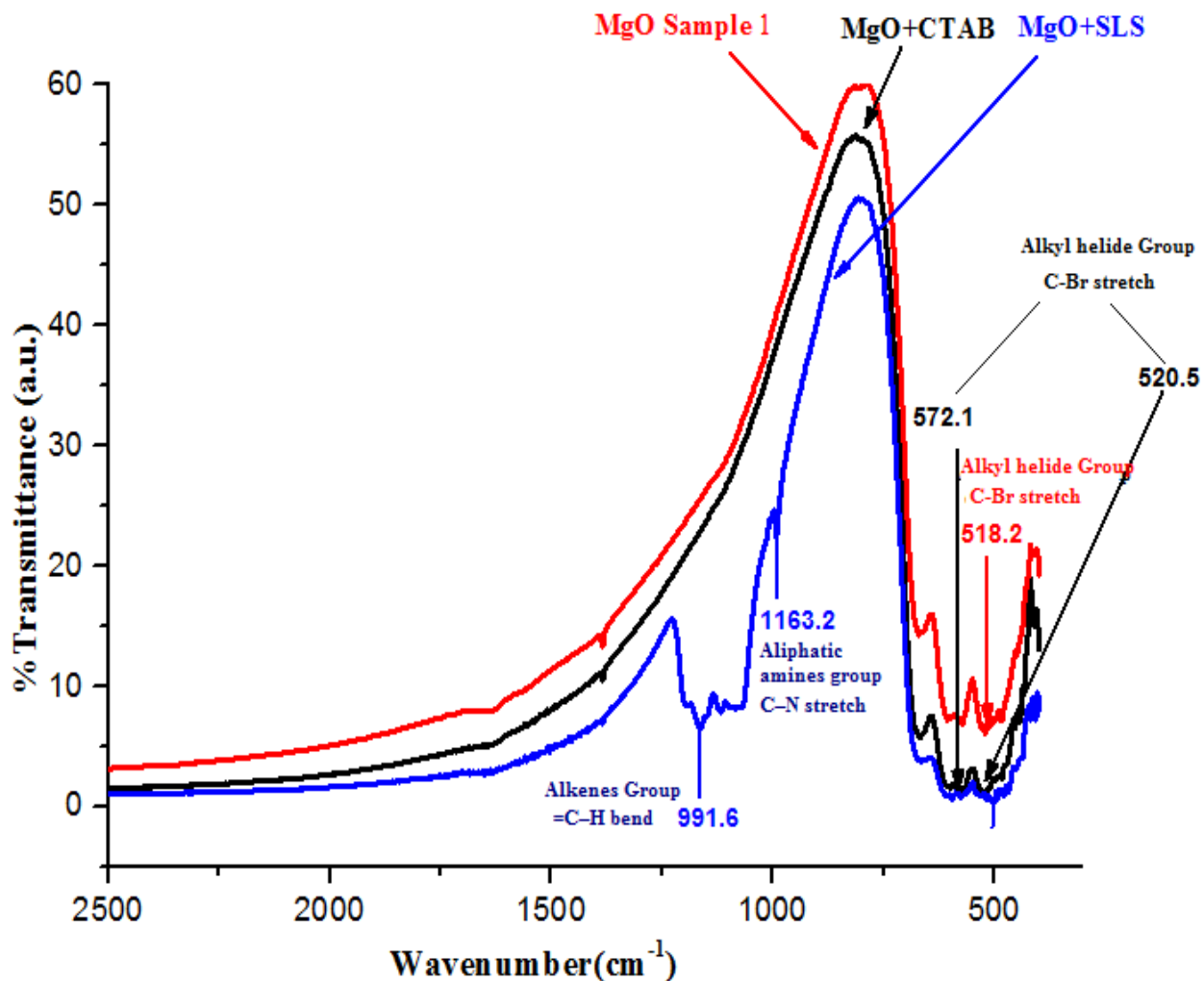


Figure 5.2 FTIR Characterization Result of MgO Nano Composite

#### 5.4 UV-VIS Spectral Studies:

UV-Vis spectra of all the samples were recorded in the wavelength range 200 nm to 800 nm. For the UV-Vis absorption measurement, the calcined prepared MgO samples are put in pot inside the Spectro-photometer and recorded the graph between absorbance versus wavelength for MgO Nano-composites.

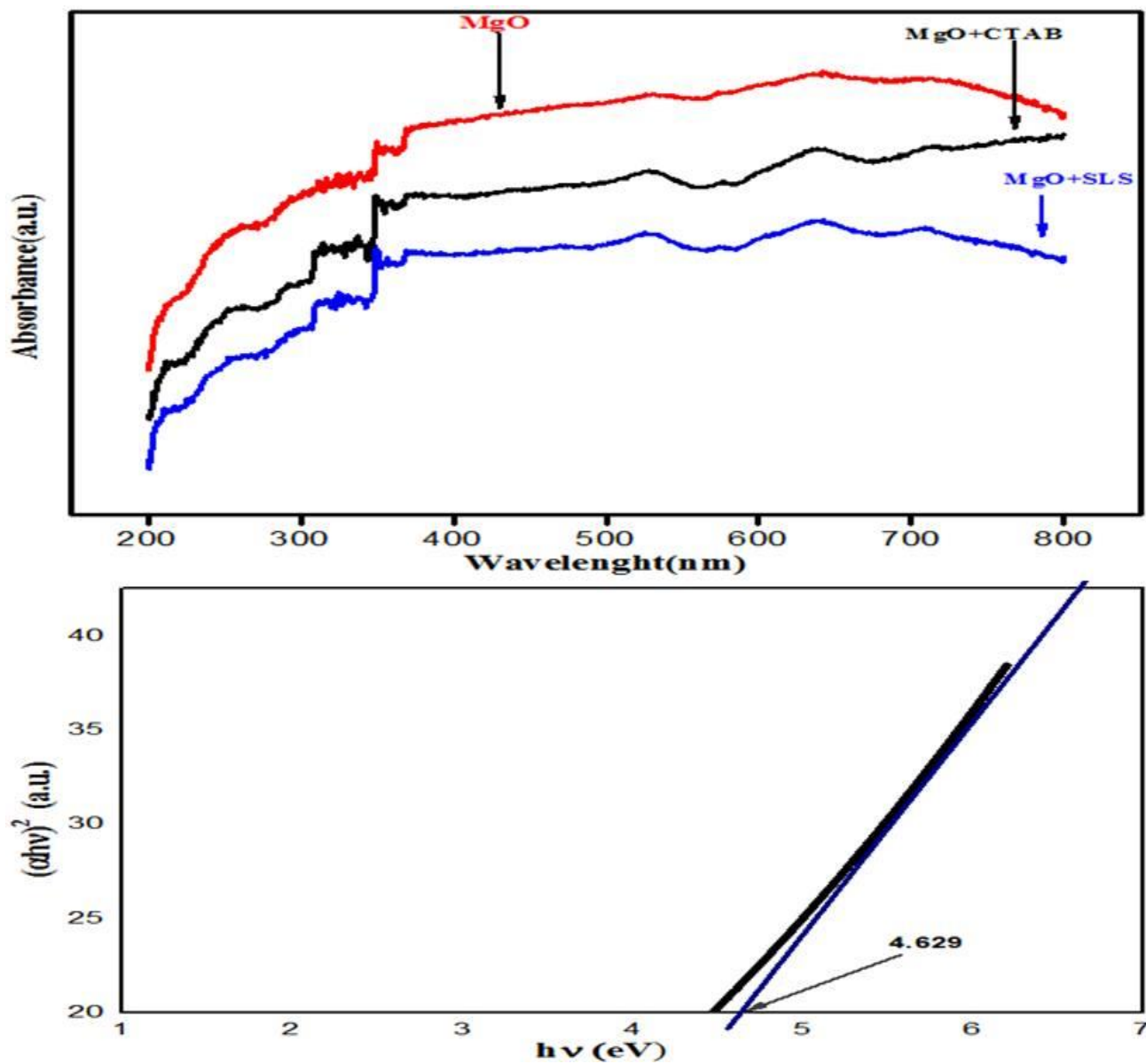


Figure 5.3 UV-Visible Characterization graph and Tauc Plot of MgO Nano-Composite

UV-Visible spectra of MgO Nano-Composite are shown through Above Tauc Plot. For all the sample we have recorded that the wavelength range from 200 nm to 800 nm. Tauc plot were used to determine the optical energy band gap of samples as shown in and the band gap energy of Nano composites are determine by using the transition rate equation for direct transition of semiconductor. The absorption coefficient of direct transition is given by the equation

$$\alpha(h\nu) = A(h\nu - E_g)^n$$

where  $h\nu$  = photon energy,  $\alpha$  = absorption coefficient with  $\alpha = 4\pi k/\lambda$ ;  $k$  is the absorption index or absorbance,  $\lambda$  is the wavelength in nm,  $E_g$  is the band gap energy.  $A$ = constant. For the present work,  $n = 1/2$  corresponding to the allowed direct transition was found to hold and the corresponding Tauc plot. Finally it found that the Optical Band Gap of MgO is 4.626 eV.

## 5.5 Scanning Electron Microscopy (SEM) Result studies

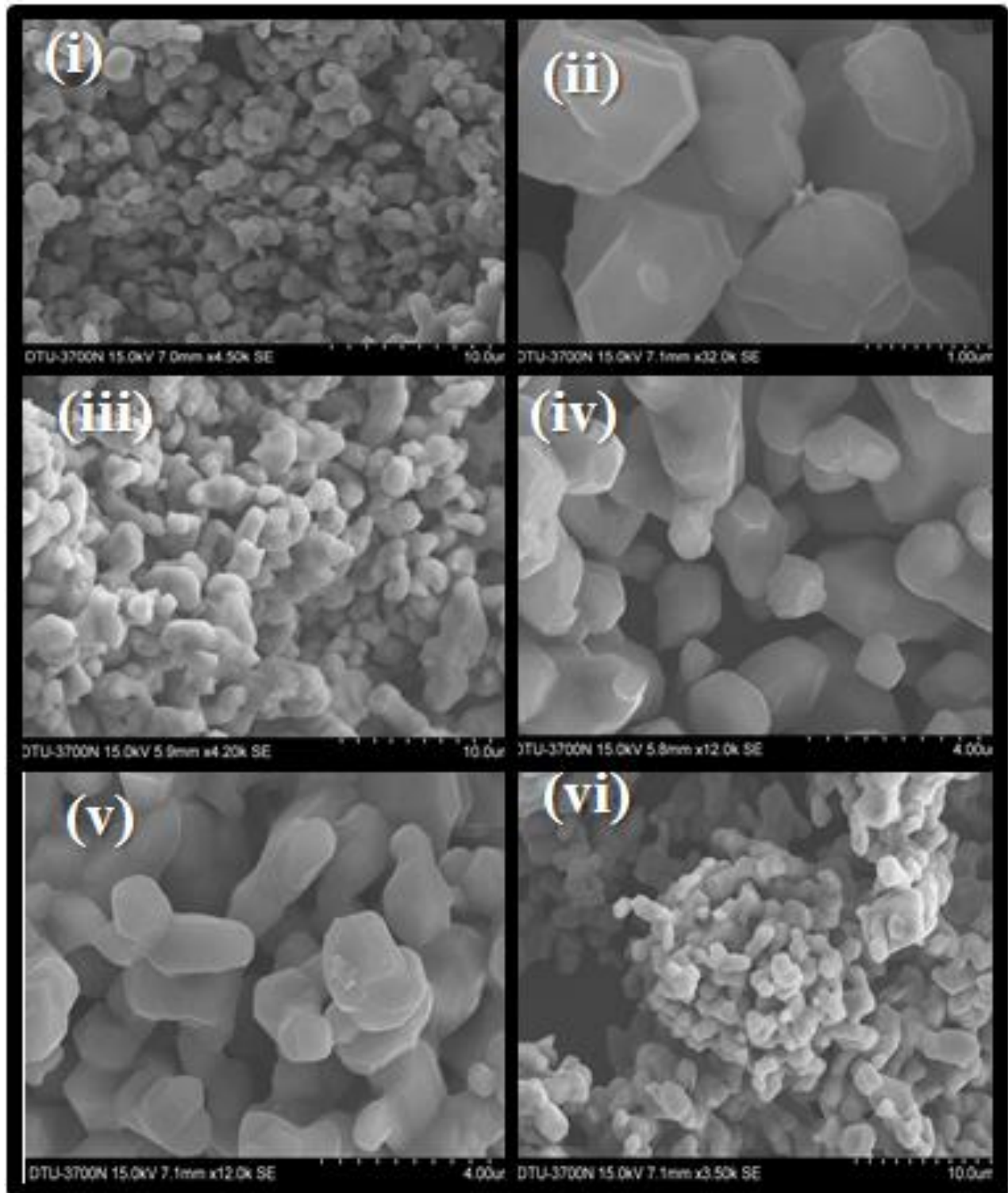
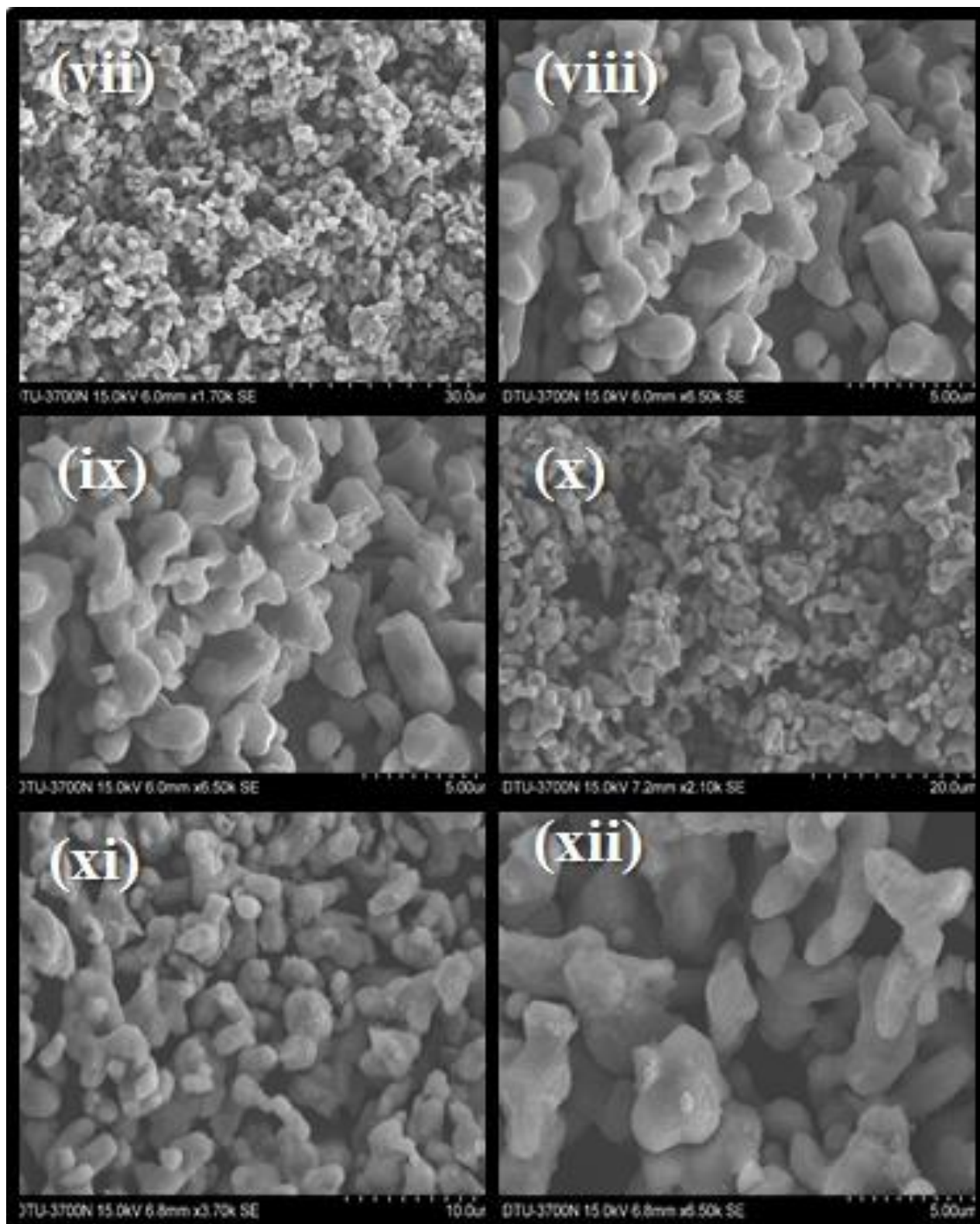


Figure 5.4 SEM Characterization Images of MgO NanoComposite





**Figure 5.5 SEM Characterization Images of MgO NanoComposite**

**Figure (i, ii) Sample (1) Annealed substrate kept in Muffle furnace at 950 °C for 36 hrs.**

These shown both images are of sample (1) which is annealed at 950°C for 3 hrs. From SEM characterization images It seems that after annealing most of the particles agglomerated and became bigger in size compare to previous state and Particles are with Simple Cubic crystalline structure with regular shape and size.

**Figure (iii, iv) Sample (2) Muffle furnace (950°C) 3Hr.**

From SEM characterization above two SEM images are of sample (d) which was kept in Muffle furnace for 3 hours at 950°C. These two SEM images clearly show that particles are heated at favorable temperature according to their agglomeration of particles. Formation of particles in this sample is also very regular in shape and size. The particles are existing with Simple cubic structure.

**Figure (vii, viii) Sample (3) vacuum oven for 3 hrs at (250°C)**

In above both SEM Characterization images of sample (3), which was kept at 250°C in vacuum oven for 3 hour is shown. After vacuum oven heating particles became closer to each other and formation of all particles are regular in shape and size with Simple Cubic crystalline structure.

**Figure (v, vi) Sample (4) heated in vacuum for 3 hrs. (200 °C)**

In above both images which are of sample (4) which is heated in vacuum at 200°C for 3 hrs. By Scanning Electron characterization Technique we had characterized sample (4) and found that all particles are properly and the crystal structure of this sample is Simple Cubic.

**Figure (ix, x) Sample (5) (CTAB +400°C, 3hrs)**

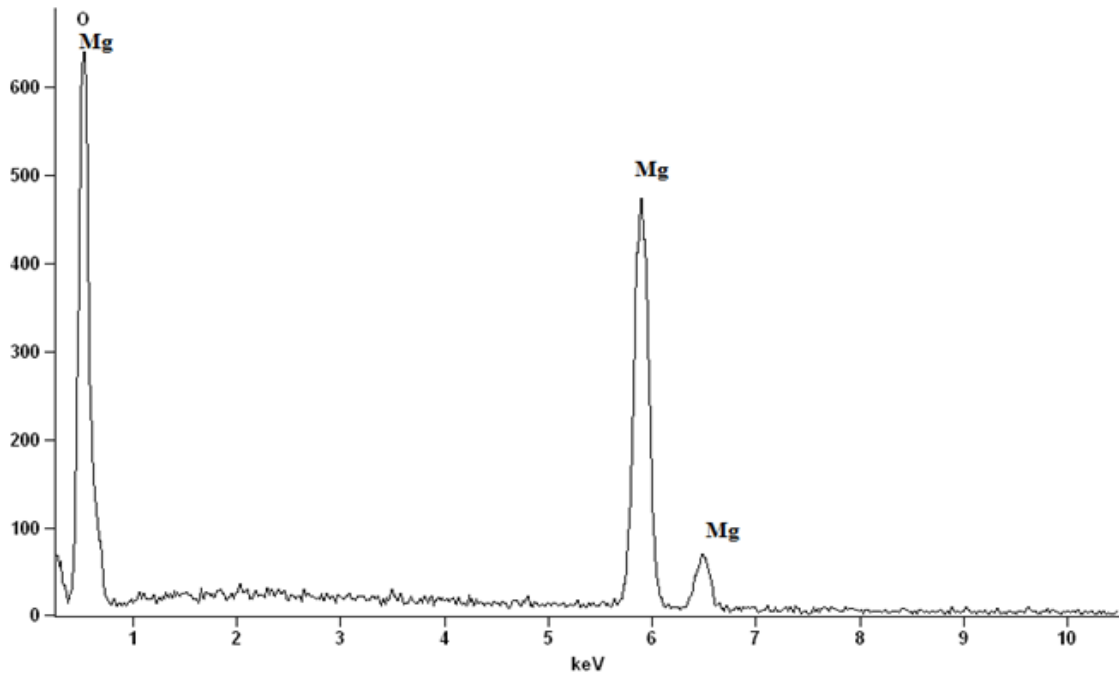
Above both SEM characterization results images are of sample(5) which prepared by using initial sample mixed with CTAB is an amine based cationic quaternary surfactant and kept in ordinary furnace in presence of air at 400°C for 3 hours. After using CTAB particles are separated with a proper regular shape and size with monoclinic crystalline structure.

**Figure (xi, xii) Sample (6) (SLS + 400°C, 3hrs)**

In above both SEM characterization images are sample (6) which is mixed with Sodium laureth sulfate (an anionic detergent, inexpensive and very effective foaming agent) and kept in normal furnace at 400°C for 3 hour and then particles agglomerated and form bigger particles which are regular in shape and size. The particles are existing with orthorhombic crystalline structure.

**5.6 Energy Dispersive Characterization Analysis:**

EDX allows the elemental composition of the specimen to be measure.



Acc.Voltage: 15.0 kV Take Off Angle: 70.0 deg.

Quantitative Results for: Base(782)

Element Line	Net Counts	Int. Cps/nA	Weight %	Weight % Error	Atom %	Atom % Error	Formula	Standard Name
O K	5079	---	15.54	+/- 0.32	38.71	+/- 0.79	O	
Mg K	7982	---	84.46	+/- 1.61	61.29	+/- 1.17	Mg	
Mg L	1973	---	---	---	---	---		
<b>Total</b>			100.00		100.00			

**Figure 5.7 EDX Characterization result of Sample (1)**

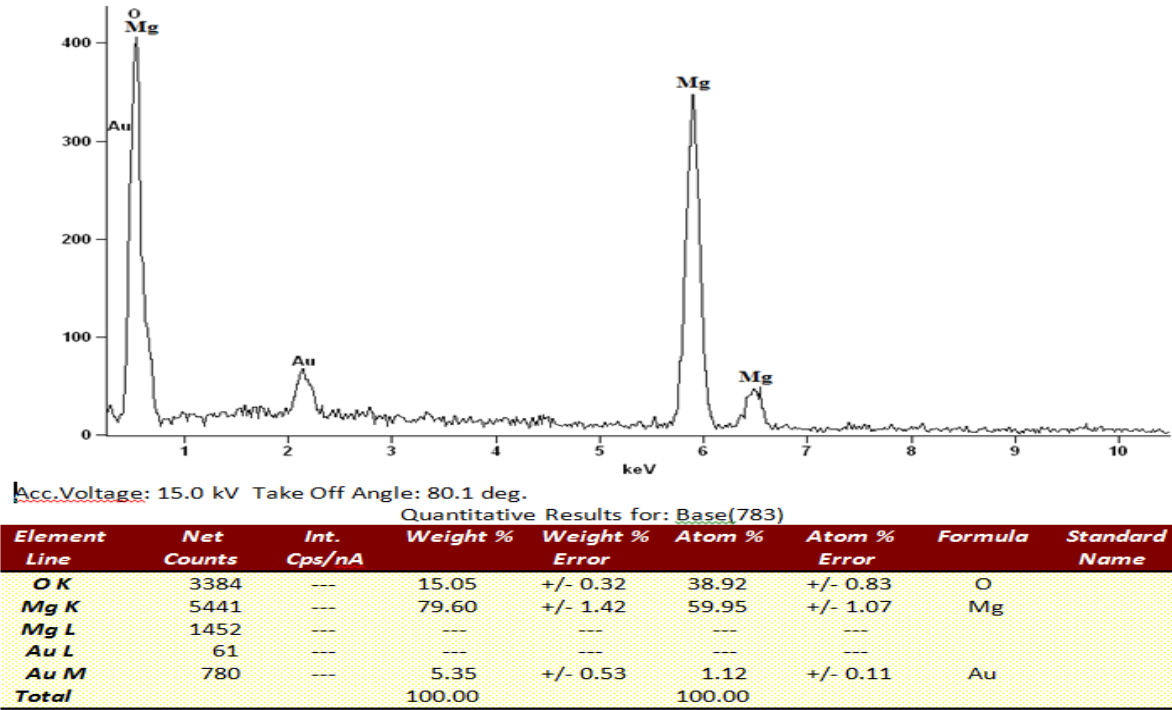


Figure 5.6 EDX Characterization result of Sample (V)

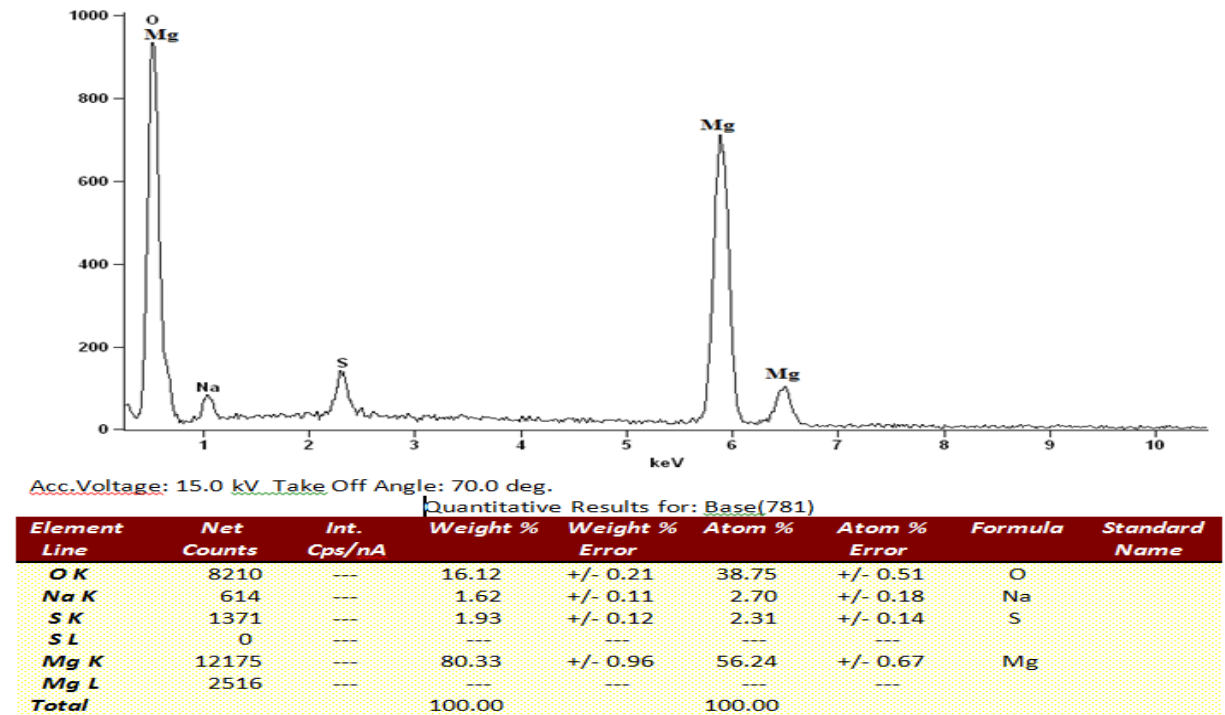


Figure 5.8 EDX Characterization result of Sample (VI)

## Chapter 6

### **6.1 Conclusions**

- 1.** The desired material has been prepared with Simple Cubic, Mono clinic and Orthorhombic Crystal Structure with regular repeated shape and size.
- 2.** MgO Nano-Composite has been prepared by Sol-gel method. The crystallite size of synthesized Nano-particles calcined at Different-different fixed temperature and respective hours are estimated by using Debye-Scherrer formula. The average crystallite size 33.41 nm, 39.07 nm, 32.5025 nm, 31.8243 nm, 25.44139 nm, 36.513 nm, 35.3238 nm were observed.
- 3.** FTIR Spectra of the MgO Nano-Composite calcined at 950<sup>0</sup>C for 36 hrs and sample with CTAB and SLS Heated up 400 hrs for 3 hours show presence of the IR peaks band at around 518.2 cm<sup>-1</sup> for sample (1) (which exist with Alkyl helide Group and the bonding formation is C-Br stretch), 572.1 cm<sup>-1</sup>, 520.5 cm<sup>-1</sup> for sample (5) having Alkyl helide Group and the bonding is C-Br stretch, and 991.6 cm<sup>-1</sup>, 1163.2 cm<sup>-1</sup> for sample (6) having alkenes Group and aliphatic amines group the bonding is =C–H bend and C–N stretch respectively.
- 4.** The energy band gap of the calcined samples, determined by Tauc plot, was found 4.629 eV. It might be due to quantum confinement effect i.e. increase in the crystallite size, decreases the band gap.
- 5.** From absorption spectroscopy results, it has been found that firstly the absorbance increases with an increase in wavelength near the band edge (348.60 nm) indicating the formation of nanostructure samples and thereafter the value of absorption coefficient becomes more or less constant indicating the uniformity of size of synthesized Nano-particles.
- 6.** SEM image shows a general view of the morphology of calcined Nano-particles. It has been observed that all the synthesized Nano-materials are agglomerated in nature and Simple Cubic, Orthorhombic and Monoclinic in shape.
- 7.** EDX Characterization result shows presence of good percentage of “Mg” and “O” contaminants in prepared Nano Composite.

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