1.Introduction

1.1:Nanoparticles:

Particles of 1–100 nm in diameter exhibit unique electronic, optical, photonic, and catalytic properties and have an ideal size for use as nanotechnological building blocks (Liz-Marzán, L. M., and Kamat, P. V. (2004).)They may be composed of all sorts of substances, including metals, semiconductors, non-metal (e.g., C), organometallics, core–shell composite architectures, hybrid nanocrystals, and organic polymers. These particles often display properties intermediate between properties of atoms or molecules and those of condensed bulk matter because of their specific size and high surface-to-volume ratios. (CaoThangDinh, and Trong On Doa, (2012).

1.2:Nanostructure:

Nanostructures is a structure of intermediate size between microscopic and molecular structures. Nanostructural detail is microstructure at nanoscale.In describing nanostructures, it is necessary to differentiate between the number of dimensions on the nanoscale. Nanotextured surfaces have one dimension on the nanoscale, i.e., only the thickness of the surface of an object is between 0.1 and 100 nm. Nanotubes have two dimensions on the nanoscale, i.e., the diameter of the tube is between 0.1 and 100 nm; its length could be much greater. Finally, spherical nanoparticles have three dimensions on the nanoscale, i.e., the particle is between 0.1 and 100 nm in each spatial dimension. The terms nanoparticles and ultrafine particles (UFP) often are used synonymously although UFP can reach into the micrometre range. The term 'nanostructure' is often used when referring to magnetic technology.Nanoscale structure in biology is often called ultrastructure.

1.3:What is nanotechnology:

Nanotechnology is the science and technology of small things – in particular, things that are less than 100nm in size. One nanometer is 10-9 meters or about 3 atoms long. For comparison, a human hair is about 60000 -80000 nanometers wide.

Scientists have discovered that materials at small dimensions—small particles, thin films, etc—can have significantly different properties than the same materials at larger scale. There are thus endless possibilities for improved devices, structures, and materials if we can understand these differences, and learn how to control the assembly of small structures.

There are many different views of precisely what is included in nanotechnology. In general, however, most agree that three things are important:

1. Small size, measured in 100s of nanometers or less

2. Unique properties because of the small size

3. Control the structure and composition on the nm scale in order to control the properties.

Nanostructures—objects with nanometer scale features—are not new and they were not first created by man. There are many examples of nanostructures in nature in the way that plants and animals have evolved. Similarly, there are many natural nanoscale materials, such as catalysts, porous materials, certain minerals, soot particles, etc., that have unique properties particularly because of the nanoscale features. In the past decade, innovations in our understanding of nanotechnology have enabled us to begin to understand and control these structures and properties in order to make new functional materials and devices. We have entered the era of engineered nanomaterials and devices. (LynnRathbun, Cornell, al et(2005)

The concept of nanotechnology is not new; it was started over forty years ago.According to the NationalNanotechnology Initiative (NNI), nanotechnology is defined as the utilisation of structures with at least one dimension of nanometre size for the construction of materials, devices or systems with novel or significantly improved properties due to their nano-size. Nanotechnology not only produces smallstructures, but also an anticipated manufacturing technology which can give thorough, inexpensive control of the structure of matter. Nanotechnology can best be described as activities at the level of atoms and molecules that have applications in the real world. Nano-particles commonly used in commercial products are in the range of 1 to 100 nm.Nanotechnology is increasingly attracting worldwide attention because it is widely perceived as offering huge potential in a wide range of end uses. The unique and new properties of nanomaterials have attracted not only scientists and researchers but also businesses, due to their huge economical potential.Nanotechnology also has real commercial potential for the textile industry. This is mainly due to the fact that conventional methods used to impart different properties to fabrics often do not lead to permanent effects, and will lose their functions after laundering or wearing. Nanotechnology can provide high durability for fabrics, because nano-particles have a large surface area-to-volume ratio and high surface energy, thus presenting better affinity for fabrics and leading to an increase in durability of thefunction. In addition, a coating of nano-particles on fabrics will not affect their breathability or hand feel. Therefore, the interest in using nanotechnologies in the textile industry is increasing (Y. W. H. Wong1,et al (2006)).

1.4:Why metal oxide nanoparticles:

Among all the functional materials to be synthesized at the nanoscale, metal oxides are particularly attractive, from a scientific as well as from a technological point of view. The unique characteristics of metal oxides make them the most diverse class of materials, with their properties encompassing almost all aspects of materials science and solid state physics .

1.5:Techniques of preparation of nanostructure:

There are two main techniques by which all nanomaterials and metal oxide nanoparticles in particular can be synthesized: the physical, or top-down, approach, and the chemical, or bottom-up, approach. In the top-down approach one starts from a bulk material and attempts to break it down into nanoscale objects through physical methods. Bottom-up approach refers to the buildup of a structure from the bottom, i.e., atom-by-atom, molecule-by-molecule, or cluster-by-cluster, growing from a solution. This technique has been attractive for researchers, primarily because of the simplicity with which experiments can be conducted in the laboratory. Scaling the process to production of industrial-scale quantities of powders is, however, not as straightforward. A major advantage of solution processing is the ability to generate encapsulated nanoparticles by utilizing surfactants as protective shell, leading to very homogeneous and well-dispersed nanoparticles (Cao, G. (2004). Nanostructures and Nanomaterials (Imperial College Press)CaoThangDinh,a, et al(2012)

1.5.1: a-Top-down technique: -

The top-down method is the method of breaking up a solid substance; it can be sub-divided into dry and wet grinding. A characteristic of particles in grain refiningprocesses is that their surface energy increases, which causes the aggregation ofparticles to increase also. In the dry grinding method, the solid substance is ground as a result of a shock, a compression, or by friction, using such popular methods as a jet mill, a hammer mill, a shearing mill, a roller mill, a shock shearing mill, a ball mill, and a tumbling mill. Since condensation of small particles also takes place simultaneously with pulverization, it is diffi cult to obtain particle sizes of less than 3 μ m by grain refining. On the other hand, wet grinding of a solid substrate is carried out using a tumbling ball mill, or a vibratory ball mill, a planetary ball mill, a centrifugal fluid mill, an agitating beads mill, a flow conduit beads mill, an annular gap beads mill, or a wet jet mill. Compared with the dry method, the wet process is suitable for preventing the condensation of the nanoparticles so formed, and thus it is possible to obtain highly dispersed nanoparticles. Other than the above, the mechanochemical method and the mechanical alloying method are also known top-down methods.

1.5.2:b-Buttom-up technique:

The bottom-up approach is roughly divided into gaseous phase methods and liquid phase methods. For the former, the chemical vapor deposition method (CVD) involves a chemical reaction, whereas the physical vapor deposition method(PVD) uses cooling of the evaporated material. Although the gaseous phase methods minimize the occurrence of organic impurities in the particles compared to the liquid phase methods, they necessitate the use of complicated vacuum equipment whose disadvantages are the high costs involved and low productivity.The CVD procedure can produce ultrafi ne particles of less than 1 μ m by the chemical reaction occurring in the gaseous phase. The manufacture of nanoparticles of 10 to 100 nm is possible by careful control of the reaction. Performing the high temperature chemical reaction in the CVD method requires heat sources such as a chemical flame, a plasma process, a laser, or an electric furnace. In the PVD method, the solid material or liquid material is evaporated and the resulting vapor is then cooled rapidly, yielding the desired nanoparticles. To achieve evaporationof the materials one can use an arc discharge method. The simple thermal decomposition method has been particularly fruitful in the production of metal oxide or other types of particles and has been used extensively as a preferred synthetic method in the industrial world.(Satoshi Horikoshi and Nick Serpone ,(2013).

The advantage of the physical methods is the possibility to produce a large quantity of nanoparticles, whereas the synthesis of uniform-sized nanoparticles and the control of their size remain very difficult by using the top-down route. The "Bottom up" approach is of primary interest for chemistry and materials science because the fundamental building blocks are atoms; thus colloidal chemical synthetic methods can be utilized to prepare uniform nanocrystals with controlled particle size. In the following, we will concentrate on solution phase synthetic methods that enable a proper shape and size control of metal oxide nanocrystals, methods whichincludesolvothermal/hydrothermal procedures, two-phase routes, microemulsions, and thermal decomposition. These techniques involve the use of surfactant molecules and, consequently, result in oxide nanocrystals comprising an inorganic core coated with a layer of organic ligand molecules. This organic capping provides electronic and chemical passivation of the surface dangling bonds, prevents uncontrolled growth and agglomeration of the nanoparticles, and permits chemical manipulations of the nanoparticles similarly to large molecules having their solubility and reactivity determined by the nature of the surface ligands {Rao, C. N. R., et al(2007)}. The shape of nanocrystals is crucial for the determination of their properties {Li, L.-S, et al. (2001). (Hu, J.,et al(2001)). The shape of nanocrystals can be classified according to their dimensionality, such as zero dimensional (0D) for isotropic spheres, cubes, and polyhedrons; 1D for nanorods and nanowires; and 2D for thin films, discs, prisms, and platelets. The shape of the nanocrystals can be controlled by adjusting a number of thermodynamic (e.g., relative stability of crystal polymorphs) and kinetic (e.g., diffusion of reactants, surface adhesion of surfactants) factors(Schmid, G. (2004).(CaoThangDinh,aThanhDinh, et al, (2012)

1.6:Effect of size:

The first important effect of size is related to the electronic properties of the oxide. In any material, the nanostruture produces the so-called quantum size orconfinement effects which essentially arise from the presence of discrete, atom-likeelectronic states. From a solid-state point of view, these states can be considered as beingasuperposition of bulk-like states with a concomitant increase in oscillator strength.Additional general electronic effects of quantum confinement experimentally probed onoxides are related to the energy shift of exciton levels and optical bandgap. Animportant factor to consider when dealing with the electronic properties of a bulk oxidesurface are the long-range effects of the Madelung field, which are not present or limitedin a nanostructured oxide. Theoretical studies for oxides show a redistribution ofcharge when going from large periodic structures to small clusters or aggregates whichmust be roughly considered to be relatively small for ionic solids while significantlylarger for covalent ones. The degree of ionicity or covalency in a metaloxygenbond can however strongly depend on size in systems with partial ionic orcovalent character; an increase in the ionic component to the metal-oxygen bond inparallel to the size decreasing has been proposed.Structural and electronic properties obviously drive the physical and chemicalproperties of the solid. the third group of properties influenced by size in a simpleclassification. In their bulk state, many oxides have wide band gaps and a lowreactivity. A decrease in the average size of an oxide particle do in fact change themagnitude of the band gap, with strong influence in the conductivity and chemicalreactivity. Surface properties are a somewhat particular group included in this subjectdue to their importance in chemistry. Solid-gas or solid-liquid chemical reactions can bemostly confined to the surface and/or sub-surface regions of the solid. As abovementioned, the two dimensional (2D) nature of surfaces has notable structural, typically arearrangement or reconstruction of bulk geometries, and electronic, e.g. presence ofmid-gap states,.consequences. In the case of nanostructured oxides, surface propertiesare strongly modified with respect to 2D-infinite surfaces, producing solids withunprecedent sorption or acid/base characteristics.45 Furthermore, the presence of undercoordinatedatoms (like corners or edges) or O vacancies in an oxide nanoparticle shouldproduce specific geometrical arrangements as well as occupied electronic states locatedabove the valence band of the corresponding bulk material,46,47,48 enhancing in this waythe chemical activity of the system.(Marcos Fernand.Garcia, Jose A.Rodiguez,(2007)

1.7:Effect oftemperature:

Main mechanical properties concern low yield stressand hardness and high superpasticity temperature observables. Information on oxidenanomaterials is scarce and mainly devoted to analyze sinterability, ductibility, andsuperpasticity. In particular, an important number of works have showed significantimprovement in sintering with up to 600 K lower temperatures with respect to counterparts.

where the initial constants describe friction stress and hardness, d is the primaryparticle/grain size and k the corresponding slope. The H-P effect in bulk materials isattributed to the particle/grain boundaries acting as efficient obstacles for slip transfer(stress) or dislocations (hardness). Typically, by decrease the particle/grain size down tothe order of a few tens nanometers the H-P slope, which is positive, gets smaller values.However, below such critical point it appears that conventional dislocation mechamisn(s)cease(s) to operate and a d-n ($|n| >$ ½) behavior or a “reversal” H-P mechanism wouldbecome progressively dominant. 101,102,103 On top of this, these mechanical properties arealso found to be strain-rate dependent; an enhanced strain rate sensitivity at roomtemperature is observed for TiO_2 and ZrO_2 with decreasing primary particle/grain size. Inspite of such facts, it is clear that oxide materials (like

$Al2O_3$, ZrO_2, CeO_2, and TiO_2)sintered under vacuum or using the spark plasma technique display enhanced yieldstrength and hardness with respect to conventional/bulk ceramic materials and have theadditional properties of being transparent (films), being potential materials for theaerospatialindustry. (Marcos Fernand.Garcia, Jose A.Rodiguez, (2007).

The thermal energies of the nanoparticles at 300 K andat 1800 K are 26 and 155 meV, respectively. Coagulation oftwo particles occurs after collision if the kinetic energy ofthe particles is lower than their interaction potential. Inparticular, the depth of the potential well De is low respectto the gas kinetic energy (kT) at flame temperatures. Itmeans that at 1800 K, most colliding particles have enough energy to escape from the potential well and hence toremain uncoagulated. Conversely, at lower temperaturesthe kinetic energy is reduced and the fraction of particleswith kinetic energy less than the potential depth increasesresulting in an increased sticking probability, e.g., risingfrom 0.0134 at 1800 K, to 0.279 at 300 K. At high temperatures,the kinetic energy of the relative motion of thenanoparticles exceeds the depth of the potential minimumand the time spent in the region of the interaction potentialis also reduced by a factor of (Tlow/Thigh)1/2.Particle size distributions for an ethylene flame ($C_2H_4/O_2/Ar$, U = 2.07) similar to the one presented here havebeen obtained by SMPS measurements . Bimodal distributions,were observed consisting of a dip at a particlediameter of 5 nm, which was attributed to an underlyingcompetition between nucleation and coagulation.Subsequently,the same authors reported SMPS measurementsof a slightly richer flame ($C_2H_4/O_2/Ar$, U = 2.5) and identifieda bimodality with the dip at around 15 nm. Since thisflame has a temperature profile (Tpeak = _1500 K) muchlower than the previous flame (Tpeak = _1750 K) it is plausiblethat this could cause the formation of larger clusters.Finally, these results might address the discrepancy commonlyseen in the PSD determined by different techniques, such as SMPS and TEM. Zhao et al. have recentlyreported a comparative study of nanoparticles by SMPSand TEM for the ethylene flame mentioned above(U = 2.5) showing notable disagreement between the twotechniques' sizes. In particular, TEM measurements didnot detect particles <10 nm. The cause could be due tothe different temperatures between the flame and the grid.Upon deposition on a TEM grid, nanoparticles willundergo a cooling process and as the computationsshowed, this will cause their clustering spreading the sizedistribution function towards higher values of the clusterdiameter. (stevenL.Fieger ,sergeilzvekev, et al (2007).

1.8:Size Control of Nanoparticles:

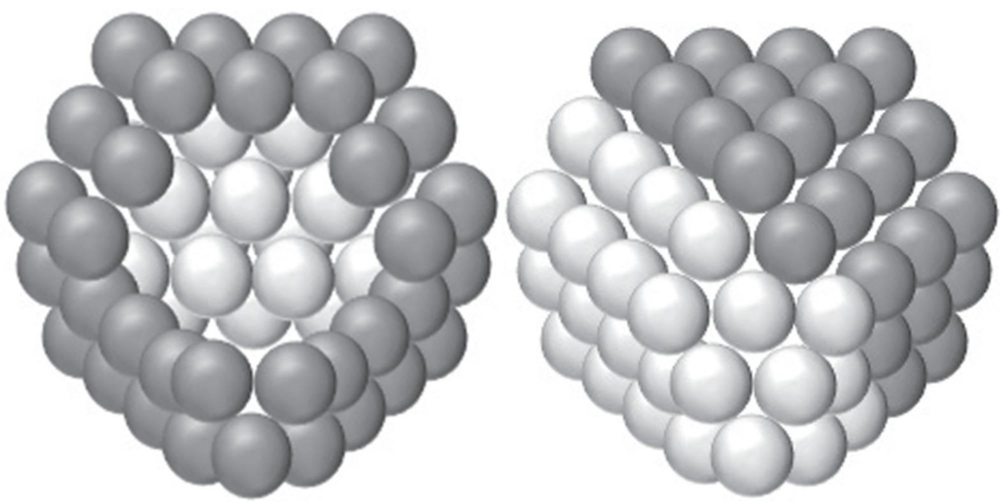
The physical and chemical properties of nanomaterials depend not only on their composition but also on the particle size (Henglein, A. (1989)} and shape(Burda, C. , Chen , X. , et al. (2005)) . Accordingly, a high quality synthesis protocol must fi rst of all provide control over particle size and shape. For example, if the diameter of an Au nanosphere is made to increase, thesurfaceplasmon resonance will be gradually shifted from 530 nm to the longerwavelength side (Liz-Marzán , L.M. (2006) . Thus, if nanoparticles differ in size, theiroptical characteristics will also change significantly. In optical applications of nanoparticles, simplification of the size distribution ofthe particles becomes a very important factor. Therefore, it is important to fabricate nanoparticles with a single target size in mind. Generally, in order to prepare monodispersednanoparticles, it is imperative that the nanoparticles grow very slowlyafter the rapid generation of the seed particles(Sugimoto, T. (2000). If the size of the nanoparticlesdecreases (i.e., increase in specifi c surface area), then the increase in the surfaceenergy of such nanoparticles will facilitate their aggregation. Consequently, aftertheir growth to the desired optimal size, it will be necessary to stabilize the particulatesurface by addition of a dispersing agent. The historical use of dispersingagents in nanoparticle syntheses is not new; for example, Ag colloids protected bycitrate were reported by Lea way back in 1889 (Lea , M.C. (1889) . However, where the concentrationof nanoparticles is unusually high, the decentralized stabilization will fall,because the protective action of the organic substrate (citrate) is no longer strongenough to prevent aggregation. Thus, several studies of dispersing agents thatmaintain a high dispersivity of the nanoparticles, and also at various concentrations,have been reported. According to the hard and soft acids and bases (HSAB)rule (Pearson, R.G. (1963) , Ag^+ , Au^+ , Pd^{2+} , Pt^{2+} are classified as soft acids in the Lewis sense (SA),and substrates possessing the thiol (R – SH) and the phospine ($P - R_3$) functionalgroups, classified as soft bases, have proven to be suitable dispersing agents (Prasad , B.L.V. ,et al. (2003) .Early research that examined organic thiol molecules as possible dispersing agentswas reported by Brust and coworkers (Brust,M.,Walker , et al (1994), if 1-dodecanethiol is used as the dispersing agent in Au nanoparticle synthesis the 1-dodecanethiol molecule can form a monomolecular layer on the Au nanoparticle surface, and fi rmly stabilize the dispersed Au nanoparticles. This particular paper has been the third most cited article in the journal Chemical Communications ever since 1965, and so can easily be said to have had a significan't impact in the chemistry fi eld. Moreover, an increase in the size of the nanoparticles can be achieved by the change in alkyl chain length from 1-dodecanethiol to alkyl chains of octane, decaneand hexadecane, as well as with a decrease in steric hindrance. For this reason, the 1-dodecanethiol dispersing agent is also used to control the particle size. Development of polymers as the dispersing agents has also been studied. In thiscase, the protection capability is determined by the affinity of the nanoparticle surface and by the molecular weight of the polymer. It is important to realize that the physical properties of a nanoparticle can change with the aggregation ratio, even though the colloidal solution may contain nanoparticles of identical siz([Yamaguchi, Y. (2008) . The images and the characteristics of the state of aggregation of nanoparticles are depicted. In the dispersed random structure , the dynamical physical properties and the optical properties are signifi cant. On the other hand, the electronic properties are displayed by the fractal structure, and ion andelectronic transport properties appear in the structure orientation c. The optical properties appear in the close-packed structure of the nanoparticles. whereas the discrete structures or otherwise orderly structures display dynamical physical properties, magnetism, optical, andelectronic properties. Methods to separate out particles of a given target size from a colloidal solution which contains nanoparticles of various sizes are known. They are (i) separation by precipitation, (ii) centrifugal separation, (iii) gel fi ltration column, and (iv) gel electrophoresis. As a feature of each screening method, the precipitation separation is suitable for a large distribution of colloid nanoparticles in the solution. The centrifugal separation and the gel fi ltration column are well suited for solutions of colloidal nanoparticles with a narrow size distribution. Gel electrophoresis is a suitable method to separate nanoparticles taking advantage of the difference in charge density of the particles, and is suitable for separating particles with a small cluster size. In fact, a combination of these various methods might prove advantageous. However, a problem with sorting the various sized nanoparticles using these methods is that only a fraction of the nanoparticles of a given size may be collected, and then only in small quantities. The digestive ripening method and high temperature melting technique have been proposed to resolve this problem (Stoeva , S. ,et al (2002).

1.9:Shape Control of Nanoparticles:

The shape of nanoparticles is an important factor that determines the nature of the surface plasmon resonance band just as the size of the nanoparticles did. Absorption spectra in the visible spectral region of various Au rodshapednanoparticles (i.e., nanorods) with changes in the aspect ratio (length of long side and short side) The diameters of the Au nanorods espousing a pillar form and used in this experiment ranged from 5 to20 nm and the lengths from 20 to 150 nm. It is worth noting that the change inthe ratio of a nanorod is related to the size ratio of a crystal face. An increase in the size ratio (aspect ratio) shifts the maximal absorption band to longer wavelengths. Therefore, the physical composition of the nanorods can easily change their spectroscopic features, such that various studies have been required to understand these characteristics.The preparation of Au nanorods using surfactants has been reported by Yu and coworkers {Yu , Y.-Y. ,et al(1997) . Gold nanorods were synthesized using an Au anode under ultrasonicirradiation with a template consisting of the cationic surfactant hexadecyltrimethylammoniumbromide(CTAB). Au exfoliates as a cluster from the electrode and is molded into the shape of a rod through the interaction with the CTAB micelle (at concentrations above the cmc). In the growth mechanism of nanorods, the CTAB dispersing agent is selectively adsorbed onto the {100} and {110} crystal faces of the Au nanoparticles. For this reason, the {111} crystal face grows and a rod-like metal nanoparticle is generated as a result. The use of CTAB as the dispersing agent subsequently quickly led to reports on nanoparticle research (Jana , N.R. ,et al. (2001).(Nikoobakht , B. , et al (2001)Nanoparticles of various forms and shapes have been prepared using theadsorption characteristics of a dispersing agent. Chen and coworkers reported an unusual composition of branched Au nanoparticles .usinghigh concentrations of CTAB (Chen , S. ,et al (2003) . Evidently, the molecular association of a surfactantas a dispersing agent determines the various shape features of metallic nanoparticles.The physical aspects of Au nanorods prepared using a hard template, such as mesoporous alumina, are similar to those when using a soft template like CTAB. In an early report that made use of a hard template, the Au nanorods were synthesized in the inner fi ne pores of mesoporous alumina (van der Zande,B.M.I. ,et al (1997) . In the initial stage, the nanosize porous alumina electrode is produced and the metal is then electrochemically deposited sequentially in the fi ne pores, which provide a fi rmmold; the diameter of the short axis of the Aunanorod which grows inside the fi ne pores is regulated by the size of the pores. Subsequently, the alumina moldis dissolved and removed; the so-formed Aunanorods are then taken out of the template. An interesting feature of the above method is the fabrication of nanorods of multiple layers of different metals such as Au–Ag–Au. Therefore, a nanoparticle with various features can be synthesized. As for the plural layer-type nanorod, applied research can lead to a nanosize system that might be considered a nanosizedbar code (Nicewarner-Peña,S.R.,et al(2001).

1.1:Structure Control of Nanoparticles:

Nanoparticles that are composed of two or more metals differ in their catalytic, magnetic, and optical characteristics from nanoparticles that consist of a single metal. Such nanoparticles can be sub-divided into three kinds of structures: (i) the alloy structure that exists randomly in a crystal); (ii) the core–shell structure in which the metal at the center differs from the peripheral metal .and (iii) the twinned hemisphere structure wherein two sorts of hemispheres are joined. The latter heterojunction structure facilitates phase separation . Nanostructures consisting of complex metal nanoparticles tend to hide the various new features. The core–shell structure is comparatively easy to fabricate in complex metal nanoparticles with effective functional control, which has led to several studies and reports in the literature. For instance, although the color of an Au nanoparticle liquid dispersion is purplish red (the purple of Cassius) and that of an Ag nanoparticle liquid dispersion appears yellow, whenever Au forms the core and Ag the shell the structure then takes an orange color. Moreover, if a structured matter



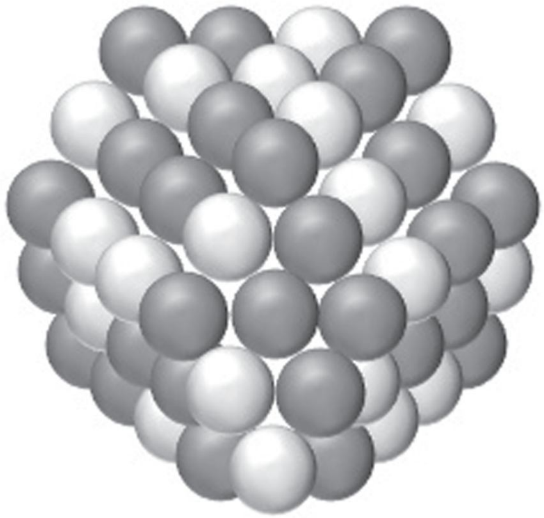


Figure 1.1: Schematic images of bimetal nanoparticles: alloy structure (a), core–shell structure (b), and heterojunction structure (c) of complex metal nanoparticles

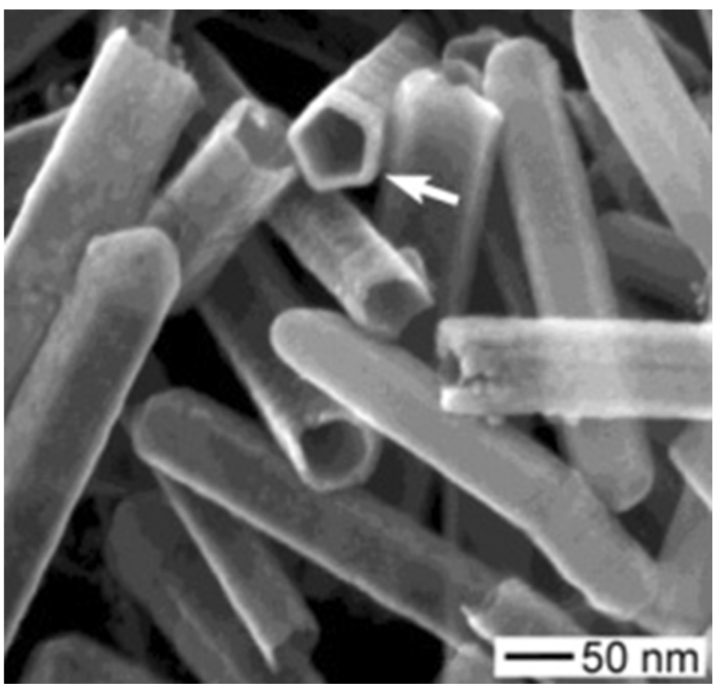


Figure 1.2: SEM image of gold nanotubes that had been broken through sonication to show their cross-sections. The gold nanotubes were prepared by reacting silver nanowires with anaqueous HAuCl 4 solution. From ref.. Copyright 2003 by the American Chemical Society

has magnetic properties, such as magnetite nanoparticles, then the magnetic metal particles could be used to form the structure'score, such that the structure will now be embodied with both magnetic and optical characteristics. Synthetic methods of preparing core–shell nanoparticles are roughly divided into two categories: (i) involving a simultaneous reduction reaction and (ii) involving a sequential one-electron reduction reaction. As an example of the simultaneous reduction reaction, consider the core being made up of Pt nanoparticles and the shell composed of Pd nanoparticles (Toshima,N.,et al. (1993) . A unique method that uses differences in the oxidation potentials of Ag and Au has also been reported (Sun,Y. ,Mayers , et al (2003)} . Here, a silver nanoparticle is added to an HAuCl 4 solution, following which an oxidation– reduction reaction takes place (Eq. 1.1) wherein gold is deposited on the surface of a Ag nanoparticle yielding the core–shell structure.

{$3Ag(s)+HAuCl_4\,(aq)\rightarrow Au(s)+3AgCl\,(aq)+HCl\,(aq)$}

The development of this method has led to the fabrication of Au nanotubes by fi rst making the pentagonal prismatic Ag nanowires to use as the template ([Sun,Y. ,et al (2002) .(Satoshi Horikoshiet al,(2013).

1.11:Copper oxide:

The oxides of transition metals are an important class of semiconductors, which have applications in magnetic storage media, solar energy transformation, electronics and catalysis .Among the oxides of transition metals, copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer application. For example, it has beenreported that 4 % addition of CuO improves the thermal conductivity of water by 20 % [10]. CuO is a semiconducting compound with a narrow band gap and used for photoconductive and photothermal applications.(Amrut.S.Lanje , SatishJ.sharma , et al (2010)).

Some transition metal oxides like ZnO, TiO_2 and $Fe3O_4$ etc. proved as potential candidates for so many applications. In the same way CuO is also one of the useful metal oxides and which has so many applications in various fields. The uniqueness of CuO nanoparticles is even though, they are metallic in bulk but they behave like semiconductors when they are in nano size. Semiconducting materials have been particularly interesting because of their great practical importance in electronic and optoelectronic devices, such as electrochemical cell, gas sensors, magnetic storage devices, field emitters, high-tc super conductors, nanofluid and catalysts etc. Due to the potentiality of CuO, it acts as a catalyst; whereas all metal oxides are not useful for the catalytic activity. In the fabrication of super capacitors also CuO is very useful and in nano range it has the wide band gap nearly equal to ZnO. The favourable band gap of CuO around 2.6 eV makes it useful for solar energy conversion and it can be used as solar cell window material. CuO nanoparticles act as a good catalyst in some of the chemical reactions. CuO nanoparticles were prepared by sol-gel method. In this method $CuCl2.2H_2O$ (0.2 M) is added with acetic acid and heated to 100 0C with continuous stirring. To control the pH of the above solution, NaOH is added to the solution till pH reached desired value. The color of the solution changed from sky blue to black with precipitation. The black precipitation was washed 3-4 times with distilled water. Finally the solution was centrifuged and dried in air for one day. The CuO nanoparticles were characterized by studying their structure with X-ray diffraction and composition by energy dispersive X-ray analysis. The size of the nanoparticles is estimated by XRD and transmission electron microscopy. These nanoparticles are used to prepare nanofluid with base fluid as deionised water and experiment is conducted to determine critical heat flux at different weight percent concentration in which maximum enhancement in CHF at 1.5 weight percent of CuOnanofluid observed 57.26 percent. (JagdeepM.KSHIRSAGAR1 Ramakant SHRIVASTAVA1 Prakash S ADWANI1,).

1.11.1:Preparation of CuONanoparticles:

1.11.1.1:Sol- gel method of synthesis:

There are various techniques to prepare nanocrystals e.g. sputtering, laser ablation, cluster deposition, sol-gel method etc. In the present work the synthesis of CuO is preferred by sol-gel route because this method is easy and economical The sol-gel process involves the formation of colloidal suspension (sol) and gelatine of the sol to form a network in continuous liquid phase (gel). The precursors for synthesizing these colloids consist usually of a metal or metalloid element surrounded by various reactive legends. The aqueous solution of CuCl2.2H2O (0.2 M) is prepared in cleaned round bottom flask. 1 ml of glacial acetic acid is added to above aqueous solution and heated to 100 °C with constant stirring. 8 M NaOH is added to above heated solution till pH reaches to 7. The colour of the solution turned from green to black immediately and the large amount of black precipitate is formed immediately. The precipitate is centrifuged and washed 3-4 times with deionised water. The obtained precipitate was dried in air for 24 hours. This powder is further used for the characterization of CuO nanoparticles. The chemical reaction is as follows,

$$CuCl_2 + 2\ NaOH\ Cu(OH)_2 + 2\ NaCl$$

And copper hydroxide decomposes into copper oxide on heating as follows,

$$Cu(OH)_2 CuO + H2O$$

1.11.1.1.1: Structure and microstructure of CuO nanoparticles:

The structural and micro structural properties of the CuO nanoparticles prepared by Sol –Gel method are as follows;

1.11.1.1.2: Structure analysis CuO of Nanoparticles:

Figure 2 shows the XRD pattern of prepared CuO nanoparticles. All the peaks in diffraction pattern shows monoclinic structure of CuO, and the peaks The average grain size calculated by using Debay-Scherrer formula is approximately 20.4054 nm are comparable with JCPDS file No .[72-0269]. The lattice parameters were calculated from XRD data is as follows. a = 0.4529 nm, b = 0.343 nm, c = 0.507 nm.Debye- Scherrer formula,

$$D = 0.9\lambda / \beta \cos \theta \ (1)$$

Where β is full width at half maxima of the peak in XRD pattern, θ is angle of the peak, wavelength of X-rays. Elastic strain is also calculated from XRD results. The strain results suggested that if the particle size is less than 20 nm than they have more strain and greater than 20 nm particles have less strain. It clearly shows that the smaller particles have high strain and bigger particles have less strain. These values are in agreement with the literature values {M. Nagaraju, K. Mukkanti, et al,(2012)}. Morphology index was calculated from XRD full width at half maxima values and it is found that if the size of the particle increases, then the morphology index is also increases.

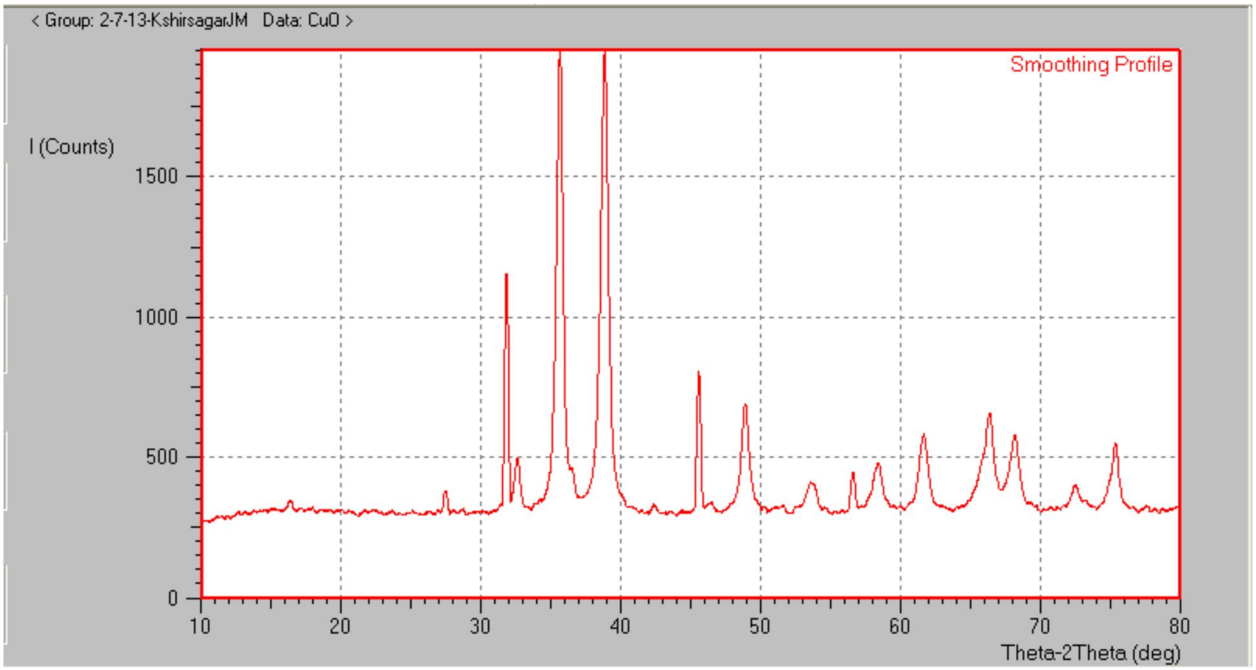


Figure 1.3: XRD pattern of CuO nanoparticle

1.11.1.1.3: Scanning Electron Microscopy (SEM) Examination:

SEM micrographs of the synthesized CuO are shown in the figure 3. From the figure it is quite evident that there is no definite morphology in the sample. It seems that the particles were agglomerated and form a cluster. As the particle size calculated from the XRD is in nano range and not getting any exact information about the surface morphology of the sample from the SEM micrograph,the morphology observed in the sample not showing any hard grains which gives the idea that size of the particle is small and further needs to be characterized by Transmission electron microscopy (TEM) to obtained exact morphology and size of the particles as shown in figure 4 and figure 5 Select-area electron diffraction (SAED) image of the particles is shown.

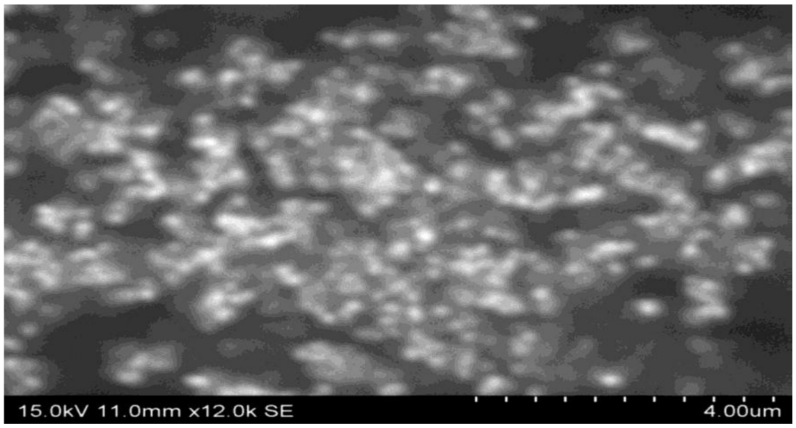


Figure 1.4: SEM images of CuO nanoparticles

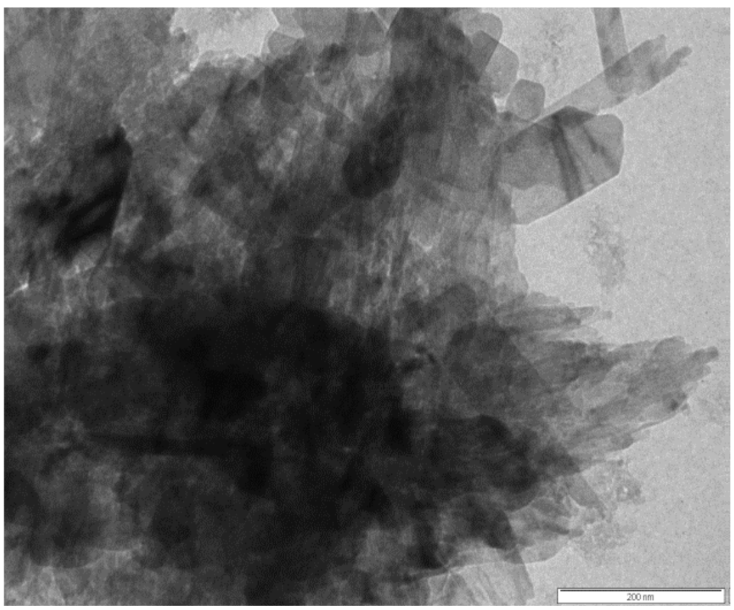


Figure1.5: image of CuO Nanoparticles

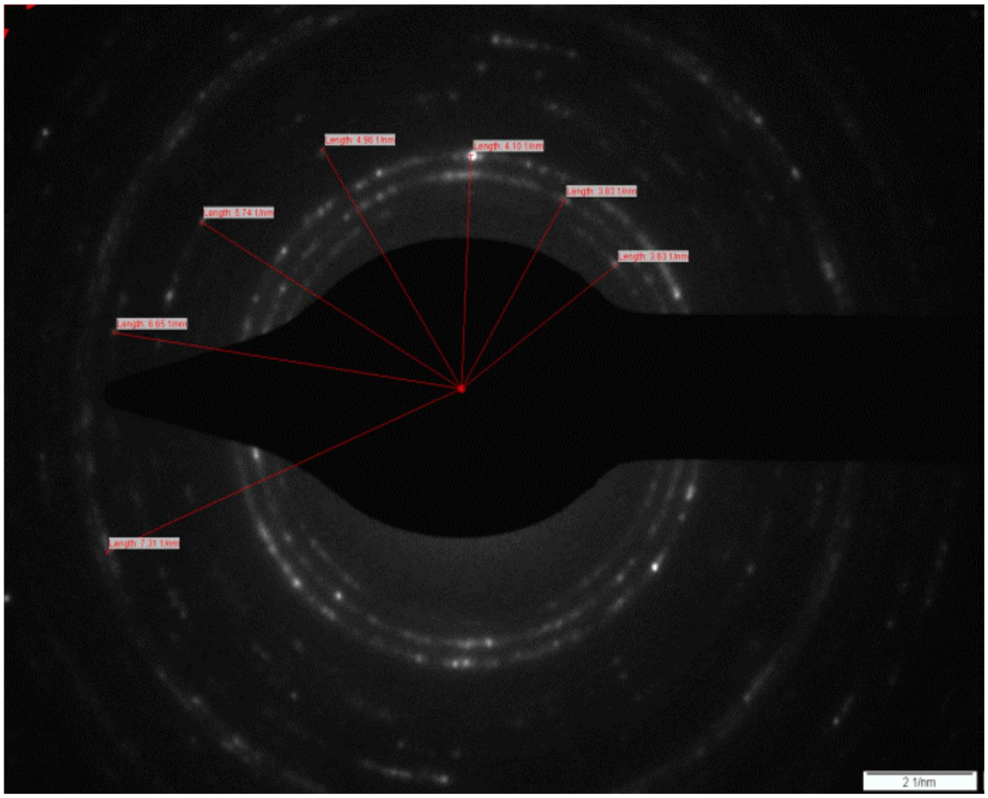


Figure 1.6: Select-area electron diffractionNanoparticles (SAED) of prepared CuO

1.11.1.1.4: Nano fluid preparation (CuO):

In the experiment, nanofluid is prepared by using two-step method, dispersing nanoparticles in to the base fluid afterward magnetic stirring continuously for four to five hours. Deionised water is used as base fluid and CuO nanoparticles are used without using any additives. The CuO nanoparticles are prepared in the laboratory by Sol-Gel method. It is observed that surfactants could be used to stabilize the nanoparticles suspension, but it may have influence on the rheological behaviour of the fluid and boiling heat transfer (Wen D, Ding Y (2005). Das et al. noted that nanoparticles dispersed without surfactant did not change the surface tension of the base fluid (Rodríguez, J. A.,et al. (2007).

In an experiment, surfactants are not used and ultrasonic excitation was performed for 4 hours just before the experiment. A glass beaker of 500 ml capacity is used for the preparation of nanofluid. Magnetic stirrer makesManeto company, Model # 08849-00, rating 150 B & 230 V AC supply is used. A magnetic needle of 10 x 10 mm used to stirrer the solution. Mercury thermometer is used for temperature measurement. CONTECH 0.1 mg weighing balance is used(Jagdeep M.KSHIRSAGAR1 Ramakant SHRIVASTAVA1 Prakash S ADWANI1,).

1.11.1.2:AnotherMethod:

1.11.1.2.1: Synthesis: -

Aqueous solution of copper acetate (0.02 mol) is prepared in round bottom flask. 1 ml glacialacetic acid is added to above aqueous solution and heated to 1000C with constant stirring. About0.4 g of NaOH is added to above heated solution till pH reaches to 6-7. The large amount ofblack precipitate is formed immediately. It is centrifuged and washed 3-4 times with deionizedwater. The obtained precipitate was dried in air for 24 h.

1.11.1.2.2: Characterization: -

The powder X-ray diffraction (XRD) was performed using Philips Holland, XRD system PW 1710 with nickel filtered CuKa (l = 1.5405 Å) radiation. The average crystallite size (t) has been calculated from the line broadening using Scherrer's relation: t = 0.9l /Bcosθ, where l is the wavelength of X-ray and B is full width of half maximum (FWHM). Photoluminescence (PL) measurements were performed by F-4500 FL spectrophotometer with 150 W xenon lamp at room temperature. Powder samples are spread over a glass slide and mounted inside the sample holder. The morphology of CuO nanoparticles was studied using scanning electron microscope (JEOL JSM 5600). The transmission electron microscopy (TEM) was performed with Tecnai 20 G2 under 200 KV. Samples are prepared by dispersing drop of colloid on copper grid, covered with the carbon film and the solvent is evaporated.

1.11.1.2.3: XRD study: -

XRD pattern of as prepared CuO nanoparticles is shown in figure 1. It gives a single-phase with a monoclinic structure. Lattice parameters are a = 4.84 Å, b = 3.47 Å, c = 5.33 Å. The intensities and positions of peaks are in good agreement with the reported values (JCPDS file No. 05-661).

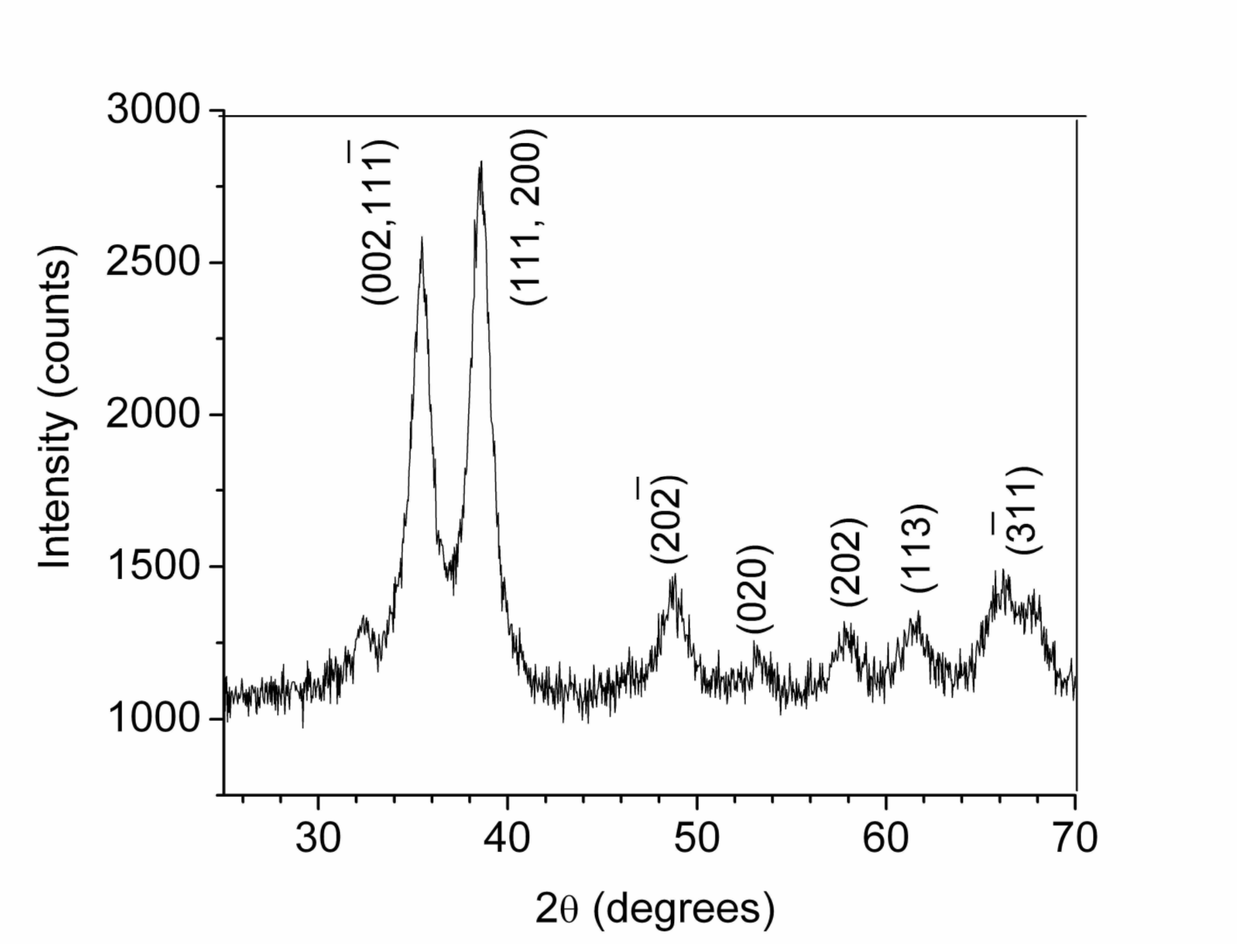


Figure1.7: XRD of CuO nanoparticles

No peaks of impurities are found in XRD pattern. The peaks are broad due to the nano-sizeEffect. The average crystallite size of CuO nanoparticles is found to be 8 nm using ScherrerFormula

1.11.1.2.4: SEM and TEM study:

Figure 2 shows the SEM image of as prepared CuO nanoparticles. It shows that the CuOnanoparticles are in rectangular shape. Figure 3 (a) shows the TEM image of as preparednanoparticles. The size of particle observed in TEM image is in the range of 5-6 nm which is ingood agreement with calculated by Scherrer formula using XRD. Figure 3 (b) shows the selectedarea diffraction pattern (SAED) of as prepared CuO nanoparticles. It shows that the particles arewell crystallized. The diffraction rings on SAED image matches with the peaks in XRD patternwhich also proves the monoclinic structure of as prepared CuO nanoparticles {H. Wang, J. Zhang, J. J. Zhu, H. Y. Chen, J. Cryst. Growth, **2002**}.

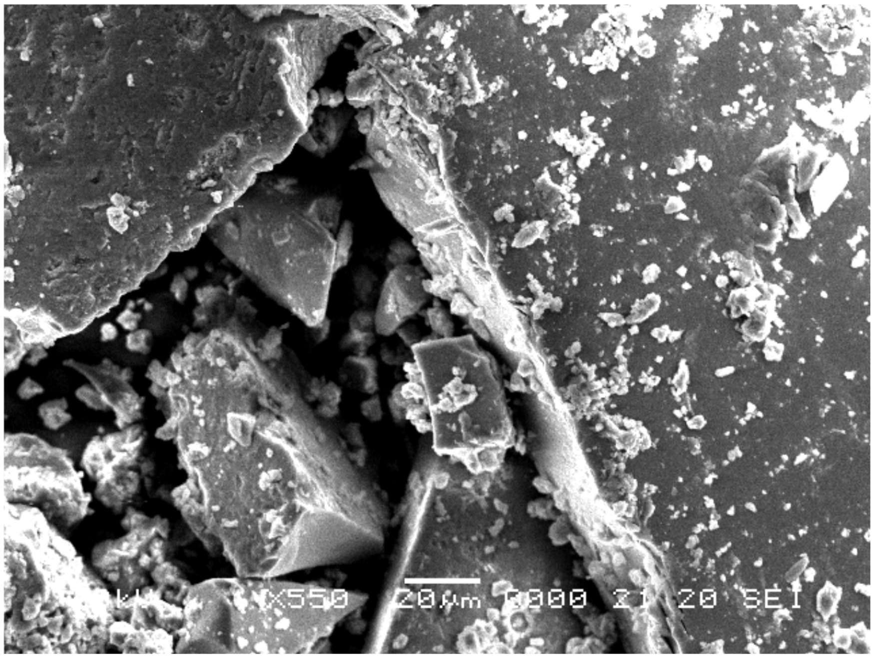


Figure1.8: SEM image of as prepared CuO nanoparticles

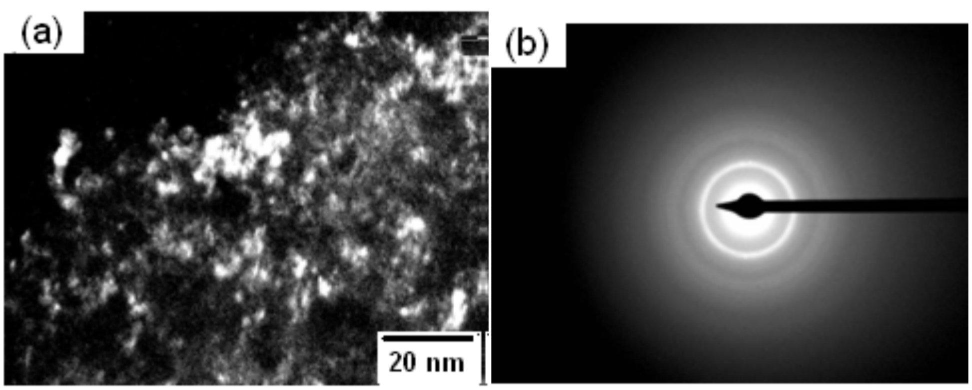


Figure 1.9: (a) TEM image, (b) SAED pattern of as prepared CuO Nanoparticles.

1.11.1.2.5:PL study: -

The room temperature photoluminescence spectra of as prepared CuO nanoparticles are shown infigure 4 after excitation at 330 nm. Three emission peaks are observed at 398 nm (violet), 470nm (blue) and 527 nm (green) for CuO. The first one corresponds to the band-edge emission

(R. S. Ningthoujam, et al(2008) The second one is due to artifact. The third one arises from the singly ionized oxygenvacancy resulting in green emission of CuO materials because of recombination of a photogenerated hole with a singly ionized electron in valence band (R. S. Ningthoujam (2008),(K. Vanheusden (1996).(A.S. Lanje (2010). showsexcitation spectra of CuO nanoparticles monitored at 470 nm. It is clearly observed that the band-edge absorption is found at 355 nm. The band gap is found to be 3.5 eV which is higherthan the reported values (R. S.Ningthoujam, V. Sudarsan (2007).(Amrut.S.Lanje , et al(2010).

1.11.1.3: Synthesis of Copper Oxide Nanoparticles by a Novel Method:

1.11.1.3.1: Preparations

- Preparation of leaves extract: 0.1gm of dried leaves and stems of Centellaasiatica was weight out and to it 10 ml of the double distilled water was added, it was stirred for about 20 mins in a magnetic stirrer at room temperature. After stirring, it was filtered using watman filter paper no.4 and followed by centrifuged to settle down the unwanted solids. Extract was taken up by using a syringe to avoid the solid residues.This very leave extract is used for the preparation of copper nanoparticles.
- Environmental benign preparations of copper nanoparticles: 1% (w/v) of copper nanoparticles was prepared by dissolving 1 gm. of Cu (CH_2COO) $2.H_2O$ was dissolved in 100 ml of double distilled water; it was stirred for 20 mins. To the above solution 10 ml of the Centellaasiatica leaves extract was added and the mixture was stirred for about 3 hrs. The formation of the particles can be seen within 1 hr. The solution was aged for 12 to 13 hrs. Nanoparticles prepared were centrifuged and washed with double distilled water twice. Nanoparticles was collected, dried at 600C in an oven.2.2.3 Kinetic study of photocatalytic degradation of methyl orange: Thedegradation of methyl orange in the absence and presence of CuO NPs were studied spectrotometrically by using Perkil Elmer L-35 UV-Vis spectrophotometer determining the decrease in the absorbance at 464nm. To a mixture containing 300µl of M. O(10-3M) and 1ml of CuO NPs was added, distilled water was added to make up to 3ml.The reaction was study spectrophotometrically at room temperature (250C).The colour of the reaction mixtures faded, indicating that degradation had occurred. The same procedure was followed for uncatalyzed reactions, in absence of CuO NPs.

1.12: Opjective:-

Chapter two

Experimental

2.1 Chemicals: -

- Copper nitrite dehydrate ($Cu(NO_3)_2.2H_2O$) (assay 99.99%),density 1.84g/ml at 25°c (lit).impurities<0.2% water.sigma Aldrich)
- Potassium hydroxide (KOH) (semiconductor grade, assay 99.99% basis trace metals basis, impurities 15% water, sigma Aldrich)
- Absolute methanol (CH_3OH) for (HPLC-PLUS -Gradient, assay 99.9%, CARLO ERBA)
- Hexamethylenetetramine ($C_6H_{12}N_4$) (assay ≥ 99.0%, grade ACS reagent, loss≤ 2.0 loss on dry,sigm Aldrich)
- De ionized water

2.2: Instrument andequipment: -

2.2.1: Instrumentations: -

- Balance (BL 300, max=300g,d=0.01g).
- Hot plate stirrer (REMI 2MLH).
- Oven (super fit india)
- Uv –vis absorption spectrophotometer(JENWAY,model 6505uv/vis spectrophotometer)
- BET analyser (model and brand)

2.2.2: Equipment: -

Glass Beakers (250ml, 500ml) grade one, glassrode , stop watch, thermometer.

2.3: Preparation procedures: -

2.3.1: Seed solution for CuO nanostructure growth: -

Copper nitrite dehydrate was prepare in absolute methanol (99%).where 0.01M concentration of copper nitrite dehydrate (274mg) was used in 125ml of methanol under stirring (the solution was transparent),it was heated to 60°c under continuous stirring .109 mg of potassium hydroxide was dissolved in 65ml methanol (0.03M concentration), this solution was shacked well until it become transparent and added drop-wise to the heated solution of copper nitrate dehydrate under continuous stirring. the resulting solution was kept under stirring and heating (60°C) for 2 hours before it was become ready for use. Some drops of the solution were put on thesubstrate and coat follow by spinng . Finally, the substrates were left to dry then cleaned with methanol.

2.3.2:The aqueous chemical growth solution for the cuOnanowire:

copper nitrate (copper source salt) was added in 100ml of deionized water (25 Mm). Hexamethylenetetramine (HMTA) in 100ml DI-water was added to solution one (the final ratio between the cu concentration and HTMA was 1:1). The seeded substrates were loaded having its face down in the aqueous solution and then load the solution in side an oven heated at temperature 50-96°C for 4 hours. then the unloaded substrates were cleaned carefully in deionized water and dried. Samples were become ready to characterization.

2.3.3: Standard cleaning procedure:

Some acetone was pour on the substrates and shacked well for 1 minute, then was cleaned it with deionized water. again isopropanol was pour and the same steps was repeated. Finally, the substrates was dried

2.4: Characteraization methods:

2.4.1:XRD insterument:-

Samples of copper oxide were finely ground in to very fine homogenous powder were put in holder and passed to obtain a flat surface placed in the diffractometer and exposed to X-rays, the wide-angle X-ray scattering of samples was measured using an X-ray diffractometer D8 advance powder diffractometer with Cu K α radiation. Diffraction patterns were collected at 5–90◦ using Cu K α radiation with a step size of 0.028◦, and CaF_2 as internal standard itwasusedPanan analytical X'Pert³ MRD, Copper Kα radiation at 45 kv/ 35 mA with a goniometer speed of 2°/ min .in the 2ɵ range scanning from 2◦ to 50◦ fig (2.1).

2.4.2: BET test:

Measuring the number of N_2 molecules adsorbed at monolayer or multilayer coverage, gives information needed for calculating the surface areas, which was calculated by the instrument.first degassing samples prior to analysis in about 500 to 700°C, then weigh and record the weight of thesamples, put the samples in cell and heating mantle, The cell was inserted into The station hole and the nutwas tighten gently, The computer and run the samples to adsorption and desorption by Nitrogen gas at 77 k .The total pore volume was estimated on the basis of the amount of nitrogen adsorbed at a relative pressure (P/P^o) of ca. 1.0. The pore size distribution (PSD) was determined using the Barrett–Joyner–Halenda (BJH) method applied to the adsorption branch of the isotherm. The name of test is BET -Autosorb-1C, Model #:AX1C-MP-LP.Manufacturer: QuantachromeInstruments.

Chapter three

Results and discussion

3.1: Characterization Of CuOnanoparticles:-

The synthesize nanoparticles ware charaleized by adsorption isotherms following BET technique and computational procedure for determination of the crystal structures of the nano Copper Oxide .

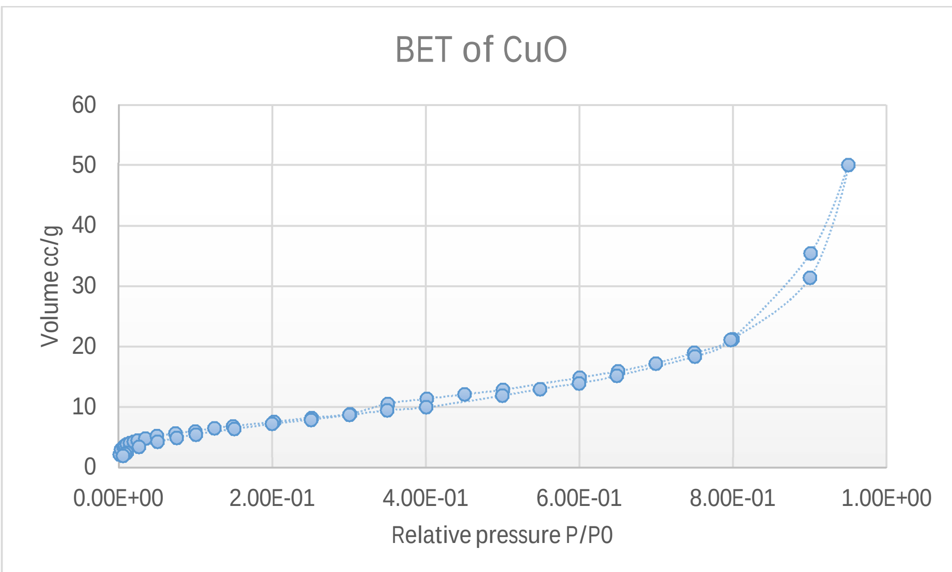


Figure3.1: BET of CuO nanoparticles

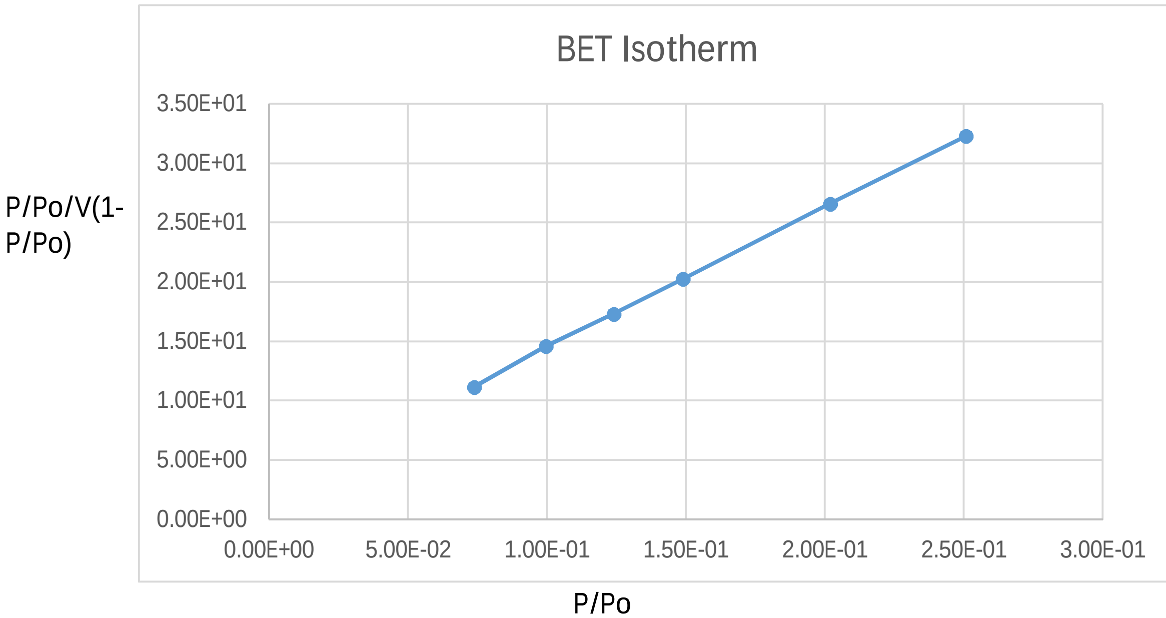


Figure3.2: BET Isotherm

3.1.1: BET Isotherm :-

The BET isotherm of the of the syntheside Copper Oxide nanoparticle is shown in Figure 3.2 ,where the amount of adsorbate at dissenet relative vapour pressure was shown . it is dear from the adsarption curve that the process is reveible .

The specific serface area of the Copper Oxide nanoparticles was found to be 28.672 m^2 pe gram . the constant C in the BET isotherm was found from the plot of reactive vapour pressure /adserpate Vs p/p , C=47.207, the slop was 118.9 whele the intercpt was 2.753e+00 with corelatiev facter of 0.9998.

3.1.2:XRD characterization :-

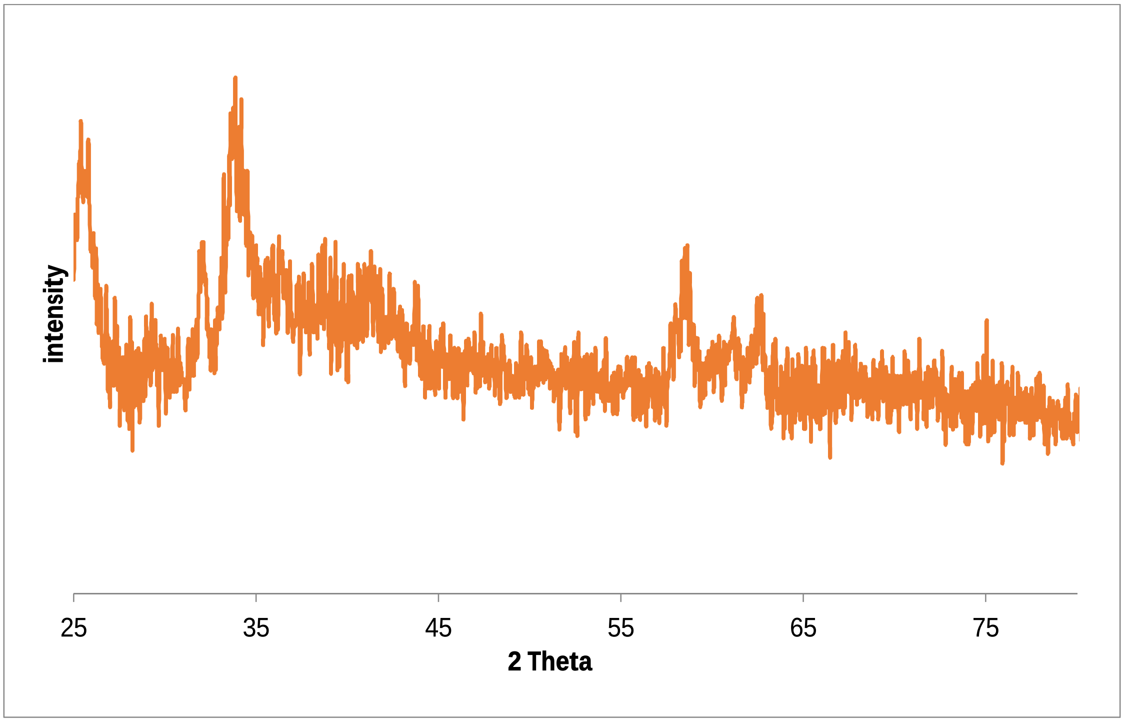


Figure3.3: XRD of CuO nanoparticles

Phase analysis of Copper oxide was carried out by XRD, as shown in figure 3.3:Tthe main diffraction peaks were observed at 2^{θ} of 25.12 ,29.29, 31.87 , 32.32 , 33.67 and 40.75 were attributed to Copper oxidenanoparticle.The broadness of XRD peaks is atypical bealure of nanostructure.

The Other peaks observed indicate that.the material has some impurities.

3.1.3: Computation procedure for characterization of CuOnanoparticle: -

The XRD data were inserted and fitted to software to interpret the structureof the synthesized Copper oxide nanoparticles, and figure fig(18) and table I it is clearly that the angle values of the nanoparticles were **α=β=γ=90** areidenticalto the cubic Copper Oxide values and x, y and z the sites in the space werealso calculated and were recorded in the given table. The **(a)** value of the synthesized Copper Oxide nanoparticles was determined and it was equal to 4.29 whech was very close to the value of the standard cubic lattice which is equal to 4.25 indicating that the structure of the synthesized Copper oxide nanoparticles is distorted, although the distortion of the structure is negligible.

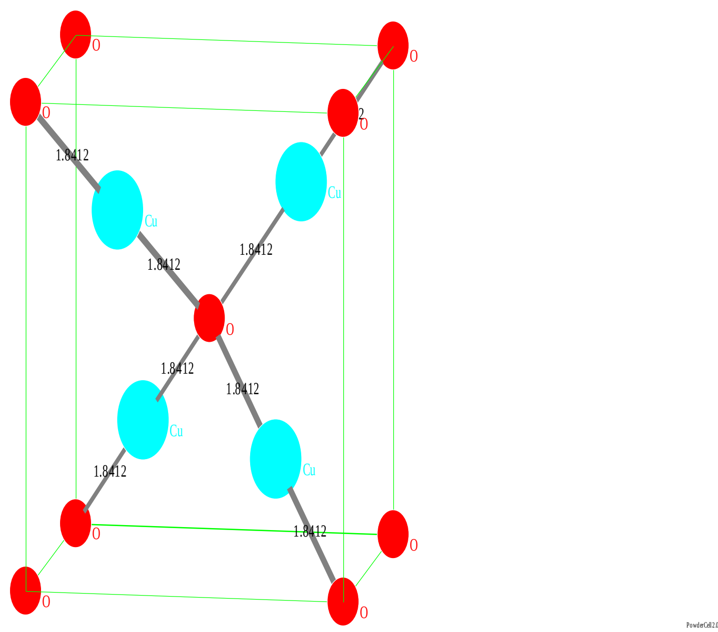


Figure 3.4: projection of the single crystal of Copper oxide

Table 3.1: Angles values of XRD for the Copper Oxide

a = 4.29 **α=β=γ=90**

Ions	X	Y	Z	SOF
Cu	0.25	0.25	0.25	1
O	0.0	0.0	0.0	1

3.1.4:Conclusion:-

The Copper oxide nanoparticles were successively synthesized using very simple and effective method (hydrothermal method) in low temperature about 60^{o}C. the synthesized nanoparticles were characterized using several characterization techniques such as BET surface analysis, powder crystal X-ray diffraction and the structural analysis of the copper oxide nanoparticles.

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مستخلص البحث

في هذا البحث تم استخدام الطريقهالهيدورثيرميه وعبر خطوات بسيطة لتخليق أكسيد النحاس النانوي . تم التحضير من أكسيد النحاس النانوي الذي تم توصيفه عن طريق تقنية فلورة الاشعه السينيه ومطيافيه الاشعه فوق البنفسجيه–المرئيهوايزوثيرم الامتصاص متعدد الطبقات والذي استخدم في حساب توزيع المسافات الناتويهعلى سطوح جسيمات أكسيد النحاس النانوي . وكان توزيع المسافات النانويه على سطح جسيمات أكسيد النحاس النانوي في حدود10^{-9} x 47.2 وكانت المسافهالسطحيه$24.672 m^2/g$نم استخدام طريقة الحوسبه لتقدير معاملات البنيهالبلوريه للجسيماتالنانويه التي تم تخليقها.

Chapter one

Introduction

Chapter two

Experimental

Chaper Three

Results and Discussion