A Study on Structural, Optical and Magnetic Properties of Transition Metal Based Binary Nanocomposites.

FOR

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Lovely Professional University
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By

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DECLARATION

I namely **Samiksha**, hereby declare that the dissertation entitled "**A Study on Structural, Optical and Magnetic Properties of Transition Metal Based Binary Nanocomposites**" submitted in partial fulfillment of the requirement for the award of the degree of Master Of Science and submitted to the Department Of Physical Science (Chemistry) of Lovely Professional University is entirely an authentic record of my original work and all ideas and references have been duly acknowledged. It does not contain any work for the award of any other degree or diploma, other than described, at any University.

DATE: Samiksha

ACKNOWLEDGEMENT

It gives me tremendous pleasure to work under the guidance of Dr.Pushpendra Kumar, Associate Professor, Department Of Chemistry, Lovely Professional University,Phagwara who supported me throughout this report . I want to give heartly thanks for his constant involvement for solving my problems. He gave me valuable criticism which helped me out to overcome each and every obstacle during my project report. His sincerity,attitude of research,art of management,timely encouragement,feeling of there always when I need him or approached him during course of my work in time. My family members deserve a very special thanks for their inseparable support and prayers. They encouraged me everytime.

I would also like to thank Dr. Ramesh Thakur , Head of Department of Chemistry, Lovely Professional University, who supported and encouraged me.

Samiksha

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CONTENTS

CHAPTER 1

INTRODUCTION

Now a days, the research is orienting around the word Nano and lot of research is going on by using the term Nanotechnology which involves the engineering of material at nanoscale. Nanotechnology offers a very immense tool for tailoring the material properties especially at nanoscale. In this field, the structures of different material are synthesized at Nanoscale which have many advantages over the bulk material.

NANOTECHNOLOGY, it is a field in which we study very small things in the range of nanometer and can be used across all scientific fields. By the help of nanotechnology, individual atom or molecule can be seen easily and controlled easily. It deals with the building things at their subatomic level. Nanotechnology executes on a scale <100nm. Its scope has been spread over different fields such as nanodevices, nanoengineering, nanoelectronics, nanooptics, even though nanomedicine also. Nanotechnology has also assured to do much more. It is expected that by 2020, no aspect of our life will remain untouched by widespread use of it.

NANOSCIENCE, it is the behavior of the physical systems that is when confined to the near atomic, nanoscale of < 100 nanometer dimensions along with the physical phenomenon which form at nanoscale, is latest one of most rapid and dynamic interdisciplinary research areas which are developing in applied science. It is in extent part because of nanotechnology, usage of these phenomenons and features or properties, is supposed for having the potential to revolutionize a large count of the fields of science and technology.

The study of Nanoscience deals with materials and their structures whose dimensions fall in the range of billionth parts of a meter that is called as nanometer. The huge amount can be defined through the length scales belonging to the range i.e. 10 to 200 Angstroms. At nanoscale, this field is one who enters a world where physics and chemistry meet with each other and the novel properties of matter has been developed[1,2].There are a large number of benefits of the nanostrctured materials over the bulk materials and in recent years their applications of potential in various scientific and technological fields have been announced[3].

Nanocomposite, is a solid material which have multiple phases in which one of the phases includes one, two or three dimensions of <100 nm. In a broad manner it may be called to have the inclusion of gels, colloids, copolymers and porous media, but it is more commonly choosen for doing the mean of the solid combination of the bulk matrix and a nano-dimensional phase which is differ in properties because they have dissimilar chemistry and dissimilar structures as well. The electrochemical, electrical ,thermal, optical, mechanical, catalytic features of nanocomposite will be differ from component materials of that one. Size limits for these effects have been proposed.

A. <5 nm, it is for the catalytic activity,
B. <20 nm, it is for preparation of a hard magnetic material into soft,
C. <50 nm, it is for the change in refractive index
D. <100 nm, it is for achieving superparamagnetism, prohibiting matrix dislocation movement or mechanical strength.

Nanocomposites, a high performance material which exhibit the unique combination of properties and designs. These are the materials which integrate the nanosized particles into the medium of ordinary material. Nanocomposites contain multiple phase domains where one of these domains(atleast) is having a nanoscaled structure [4]. Nanocomposite is a new class introduced to the composites, for which one dimension(at least) of the dispersed particle should belong to the nanometer range. Nanocomposites are distinguished in three different types, which is based on number of dimensions of dispersed particles fall in nanometer range[5](table2). The material can have good chemical and physical features , depending upon morphological and interfacial properties of the material by which the component is made. As nanocomposites are environmentally friendly, their applications provide new opportunities related to technology and business for various sectors such as in automotive, aerospace, electronics and biotechnological industries. Nanocomposite materials known as the suitable alternatives for solving the problems that microcomposites have. These are said to be the materials of 21st century, as they have unique combinations of properties and designs,which conventional composites do not have. The basic understanding of the properties is yet to be recognized, even though first inference on these was reported in 1992[4].

It is notified that the changes in features of particle may be recognized when size of particle is less than a proper level, which is known as 'the critical size' (Table 1)[6]. More to it, as the dimensions got closer to nanometre level, interactions which are on phase interfaces increased largely and it is very crucial for enhanceing the features of materials. In the context, surface area or the volume ratio of reinforced materials which are applied in the formation of Nanocomposites, is very important for the understanding of the relationships what they have of structure-property. Latestly, nano composite offers a technology which is new and opportunities related to buisness for all the industry sectors which is environmentally friendly also[7].

TABLE 1:*Features of sizes by changing properties which are reported in nano composite system.*

PROPERTIES	ASPECT SIZES AT WHICH CHANGE CAN BE OCCURRED
Size for Catalytic activity	Less then 5
Converting hard magnetic materials into soft magnetic materials	Less than 20
Size for producing changes in refractive index	Less than50
Size for production of super paramagnetism and various others phenomenons of electromagnetism	Less than100
Size for formation of toughening and strenthening	Less than100
Size for modification in plasticity and hardness	Less than100

Nanocomposite materials can be classified in three categories , according to the materials of medium,which are as shown in Table 2 [8].

TABLE 2:*Differenet types of nanocomposites*

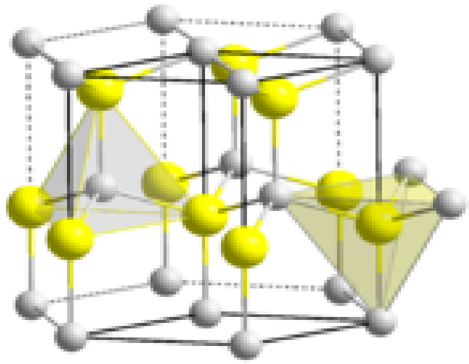
CLASS	EXAMPLES
Metal	Fe-Cr/Al_2O_3,Ni/Al_2O_3, Fe/MgO ,Co/Cr, ,Al/CNT, Ni/Al_2O_3
Ceramic	Al_2O_3/SiO_2, SiO_2/Ni, Al_2O_3/TiO_2, Al_2O_3/SiC, Al_2O_3/CNT
Polymer	Polymer/doubled layered hydroxides ,polymer/layered,Thermoplastic/thermoset silicates,polyester/TiO_2,polymer/CNT,

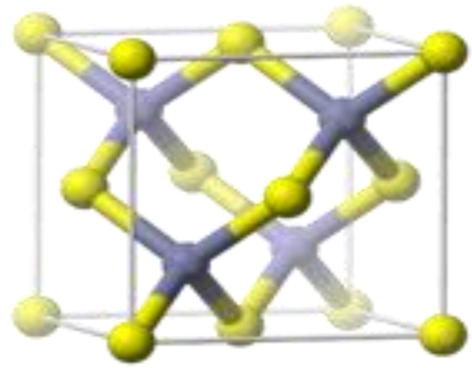
CHAPTER 2

LITERATURE SURVEY

Zinc Oxide (ZnO), is a material having a good prospective towards a range of the practical utilizations, for example: optical waveguides, piezoelectric transducers, surface acoustic wave devices, varistors, phosphors, transparent conductive oxides, chemical and gas sensors, spin functional devices, and UV-light emitters. Zinc Oxide has a broad bandgap (3.37 eV)which makes it capable material for utilization in photonics in the UV region or blue spectral range, whereas the energy of high exciton-binding is (60 meV) which permits its useful excitonic emission at room temperature. Additionly, zinc oxide that is doped with transition metals shows very great assurance for applications in spintronic. It ia said that ZnO is also sensitive to many gas species which includes ethanol i.e.C_2H_5OH, carbon monoxide (CO), acetylene (C_2H_2), which makes it perfect for the sensing applications. More to it, its piezoelectric property makes it perfect for the electromechamical sensors . Moreover, Zinc oxide is also biocompatible because of which it becomes perfect for the applications in biomedical field. Zinc oxide is a chemical and environmental friendly material. More to it, there is noticeable interest in studying Zinc oxide powder form, thin films , single crystals, or nanostructures[9,10].

Zinc oxide is crystallized in two forms, which are cubic zincblende and the hexagonal wurtzite Wurtzite structure is more stable in ambient conditions and due to which it is most common. Zincblende form may stable if Zinc oxide is grown on surface of substrates along with the cubic lattice structure. Both cases have zinc and oxide centers in tetrahedral form, which is most characteristic geometry for the Zinc(II). Zinc oxide is converted to the rocksalt motif at very high pressures which is about 10 GPa [18].





Wurtzite structure A zincblende unit cell

Zinc oxide is not a material which is new in discovery. The research on Zinc oxide has been in continuous use for many years with a good interest and also follow a pattern of roller-coaster . In its characterization term, if observe back to 1935 or more prior. For an example, the lattice parameters of Zinc oxide had been in investigation for many years. In a similar way, the optical properties and processes in Zinc oxide including its refractive index had been advancely known many decades ago. Vibration properties of ZnO through the techniques such as Raman scattering were been determined earlier . Investigations of Zinc oxide properties investigates that ZnO samples were there. The crystals of ZnO bulk have been developed by the large number of strategies, and large-sized substrates of ZnO are also availed. ZnO films which are of higher quality can also be grown at the relatively low temperatures which are <700 °C. On laser emission, there are a large number of reports of structures of ZnO at room temperature and further also. It must be noticed that above and beyond properties of ZnO , there are properties in addition to those which make it preferred over the other broad band gap materials: 1.)its high strength to the wet chemical design and 2.)higher energy radiation strength. Several experiments have also been established that ZnO is a very resistive to the high-energy radiation, which make it a perfect candidate for the space applications also. Zinc oxide is an easily used material in all the acids and alkalis and this property of ZnO also gives a chance for the fabrication of devices which have small sizes [12].

Zinc Oxide has been latestly found other niche applications also, for instance fabrication of the translucent thin-film transistors, where the covering which is protective prevents the light exposure has eliminated since the transistors which are ZnO-based are very inprotective to the visible light. It has upto charge carriers 2×10^{21} cm^{-3} which may be produced through heavy doping onto ZnO If doping level is controlled the electrical properties can be changed from the insulator by n-type semiconductor to the metal at time of maintaining the optical transparency which prepares it very effecient for the transparent electrodes in a flat-panel display and solar cells. Zinc oxide is also a very promising candidate for the spintronics applications. Dietl et al predicted that a Curie temperature in range of 300 K for Mn-doped ZnO. n-type doping in Fe-, Co-, or Ni-alloyed ZnO had been predicted for stabilizing high-Curie-temperature ferromagnetism. There are a large number of publications that are appeared to ensure these predictions, as are reviewed latestly[13,14] .

The methods of fabrication which are for Zinc oxide nanostructures may be alienated into two major groups: spontaneous synthesis based on growth and templates . Process of Fabrication which is not including a template can be occured either by the use of metal catalysts or can be they are self-catalyzed also. The metal catalysts usage, for example Ag, Au, Cd may be a benefit for getting the associated and selective growth of area. Nanorods which are aligned may also be got by the method called as hydrothermal method with no help of any metal catalyst. The alignment degree and characteristic ratio which was achieved, was totally based on the seed layer which is second-hand and the fabrication conditions as well. An enhancement has been made in the association of the rods which is at right angles to the substrate, was obtained when zinc acetate was taken for preparing the nanocrystalline seed layer rather than of ZnO nanoparticles . The growth of Zinc oxide by vapour deposition is very classically affected by the temperatures of the substrate and the source both, the distance between substrate and source, heating rate, gas flow rate, tube diameter and the initial originator. The authority of these various factors on Zinc oxide morphology has been considered in recent times[15], but effections of these factors upon the optical properties of the fabricated nanostructures are not known yet. The unlike experimental circumstance for example, the solution concentration ,substrate pretreatment and temperature, also affects the growth of ZnO by the hydrothermal strategies. Because of the low growth temperature (usually less than 1008^{0}C), crystalline qualities of these type of samples are frequently lesser than those which are fabricated by vapor deposition. However, optical feature of the samples may be enhanced by doing annealing in very appropriate conditions [16].

Zinc oxide is a very important material and it has received excellent attention due to its various applications like in field of electrical, optical, scientifical, mechanical research and in industries also[26].The usage of ZnO nanomaterial as a role of photocatalyst has particular strength because of its large surface area, wide band gap, easy and cost effective synthesis, biocompatible and environmental benign nature[21].

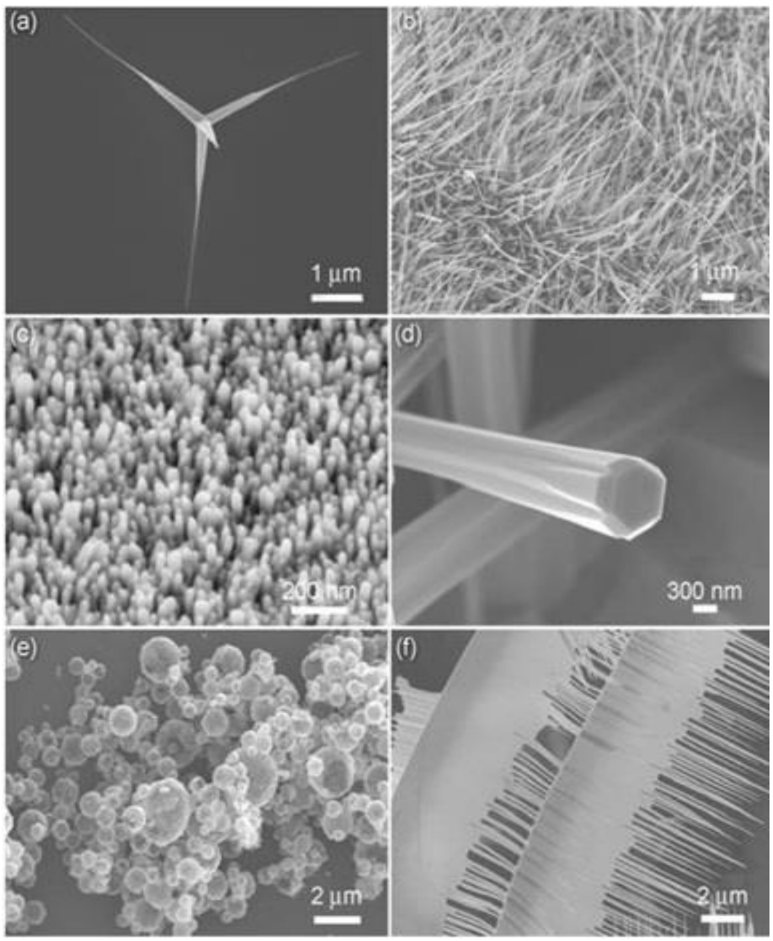


Fig 1: Scanning Electron Microscopy(SEM) pictures of different ZnO nanostructured morphologies[17].

Strategies adopted for Material Modification:

Recently doping in ZnO nanostructures received a great attention, as by doping the properties of ZnO nanostructures can be tailored for specific and desired applications. Among various dopants, currently the doping of Cd in ZnO has received a great attention as by doping ZnO with Cd, its absorption edge can be red-shifted[22]. There are very rare reports to use Cd-doped ZnO nanomaterials as a photocatalyst for photocatalytic degradation of organic dyes. Cadmium oxide has a very narrow bandgap of 2.3 eV, but ZnO has the wide band gap of about 3.2 eV so it possesses a large exciton binding energy of (60 meV) . As ZnO has a very wurtzite structure and Cadmium has a rock-salt structure, which is totally incompetable and different from each other. So a heavy doping of Cd on ZnO is unstable [19,20]. Nanocomposites may increase the stability of dispersion of ZnO and also increase the interfacial adhesion which is between the metal ions and ZnO nanoparticles. As the way of the synthesis of powders which is in an order to obtain the sizes in nanometric range, therefore we used the coprecipitation method.

In production of semiconductors, **doping** is an introduction of the impurities in an intrinsic semiconductor which modifies its electrical properties . The material which is doped is referred as an extrinsic semiconductors. The semiconductor which is doped at that high levels where it behaves more as a conductor than a semiconductor is known as degenerate semiconductor. The process of doping was first given by John Robert Woodyard who worked at Sperry Gyroscope Company at the time of World War II, with the help of a US Patent which was issued in 1950. His work demands on radar were denied. Woodyard opportunity for pursueing the research on the topic of semiconductor doping [31]. The process of doping a semiconductor in a very good crystal forms the allowed energy states among the band gap, but was very close to energy band which corresponds to dopant type. In other language, impurities of electron donor result in creation of states near to the conduction band whereas impurities of electron acceptor result in creation of states near to the valence band. The gap which was between the energy states and nearest energy band is called as dopant-site bonding energy or also called as E_B and is small. Dopants also contain the important effection of the shifting energy bands which were related to the Fermi level. The energy band which corresponds to the dopant with the greatest concentration results in end up being closer to Fermi level. Even though the Fermi level have to remain constant in the system in thermodynamic equilibrium, being stacked layers of the materials with the different properties which leads to various constructive electrical properties which are induced by the band bending, if interfaces may be clean adequately.

Co-precipitation method allows the uniform particles distribution also the lower temperature procedure but it stimulate the impurities in particles from the initial material and the metal ions which have a very dissimilar melting temperature [27]. This is the method which occurs by the addition of some alkali is the process which is carrying down by the precipitate of different substances (alkali) which is usually soluble in given circumstances. Analogously, in a medicine, the co precipitation process is defined as the precipitation of an "antigen that is beside an antigen-antibody complex" which is not bound [28]. More to its applications in the chemical analysis and also in the radiochemistry, the process of coprecipitation is also a "potentially significant to many of the issues of environment which are closely in relation to the water resources, which also includes acid mine drainage, relocation of radionuclide in the fouled waste repositories, contaminant transport of metal at the industrial and metal defense concentrations in the aquatic systems and technology of wastewater treatment "[29]. Co precipitation may also be used as the method of a synthesis of magnetic nanoparticle [30].

The p-type doping of Zinc oxide has got great awareness in both manner hypothetically as well as experimentally, due to its prospective application for the next-generation short-wavelength optoelectronic devices. Same as many other metal oxides, p-type doping of Zinc oxide is hard. A range of dopants, importantly the elements of group-V as N, P, Sb and As are used for producing p-type Zinc oxide. Evenly, p-type ZnO of adequate high-quality along with lower resistivity and higher mobility is supposed to be achieved. The main cause for the problem of making p-type ZnO of high quality is the preparation of native donor defects, as O vacancies and Zn interstitials that may simply make under O-poor conditions [23]. A very recent experimental studies, however, has shown that Copper induce a green luminescence of about 2.4 eV.

It had also been noticed that the doping of silver in Zinc oxide results in reduction of donor density, which indicates that silver can be a nice acceptor in Zinc oxide. In recent times p-type conduction of the silver doped ZnO has been established. As a result, a deep appreciative of the doping with group-IB elements in ZnO is much required [24]. However, the formation of calculated energies are very less for these group-IB elements upon the substitution sites, but high at the interstitial sites. Therefore, self-compensation and stability can not be a big challenge for the doping with the group-IB elements. Ag/ZnO nanoparticles have been synthesized already by chemical, photochemical, co precipitation, sol gel, hydrothermal or solvothermal, flame spray pyrolysis, electro spinning and RF magnetron sputtering methods and also by hybrid induction and laser heating technique [24]. It is also well-known that the properties of materials can be easily determined by the structure, pore, defect morphology, size and composition [25].

CHAPTER 3

EXPERIMENTAL DETAILS:

In this project Nanocomposites of binary mixtures are prepared by the method of coprecipitation which is discussed above.The process of this method is given below as methodology.

REQUIRED APPRATUS AND MATERIALS:

Chemicals required in this process are Zinc acetate dihydrate (219.49g/mol) by Rankem, Water (18g/mol), Sodium hydroxide (39.99g/mol) by Alpha Chemika, Cadmium acetate dihydrate (266.52g/mol) by Burgoyne Burbidges & CO., Silver Nitrate(169.872g/mol) by Alpha Chemika.

APPARATUS REQUIRED: beaker(100 ml), beakers(250 ml), magnetic stirrer, glass rods, muffle furnance, oven, watch glass, funnel, crucible, spatula, tripod wtand, whatman filter paper, petri dish.

Cd/ZnO:

METHODOLOGY:

First of all we search out foe the area where more of researchers, industries, study are going on the particular class of transition metals as they possess various utilizations in different fields of science. That transition metals are discussed above along with their properties which make them comparable to others. From literature survey we find a well-organized way to prepare nanocomposite.

PROCEDURE:

Our next step is to choose a proper reference for preparation of composites. From literature survey we have studied different properties of ZnO of its higher potential applications and various other applications also which shows its perfections. The preparation of binary nano composites of Cd/Zno, Cadmium acetate dihydrate (266.52g/mol), Zinc acetate dihydrate (219.49g/mol) and Sodium hydroxide (39.99g/mol) are taken. The pH was maintained in all sets was 7-8. For 0%, 5%, 10%, 15%, 20% doping these are mix together in following manneras process chart is given below. After taking all these amounts the simple procedure in the form of flow chart i.e given below in which first set(A) that is reference one ZnAc.2H_2O exactly 10g taken by the analytical balance and fill with 100 ml distilled water in a 250ml properly cleaned beaker. For well mixing of this placed the beaker on stirring for 20-25 minutes. After well mixing we added NaOH slowly and we observed that there was formation of powder slowly that is the formation of nanocomposites. We stopped the addition of NaOH when pH reached at 8 and amount of NaOH at that pH was 31ml and was maintained same for all the five sets.

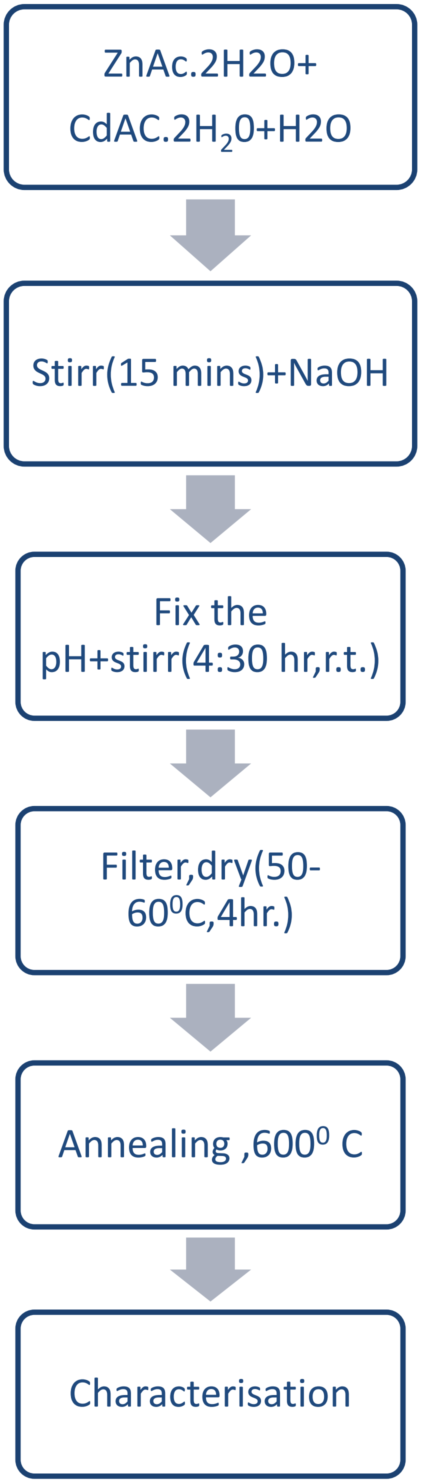


Fig2: doping with Cd

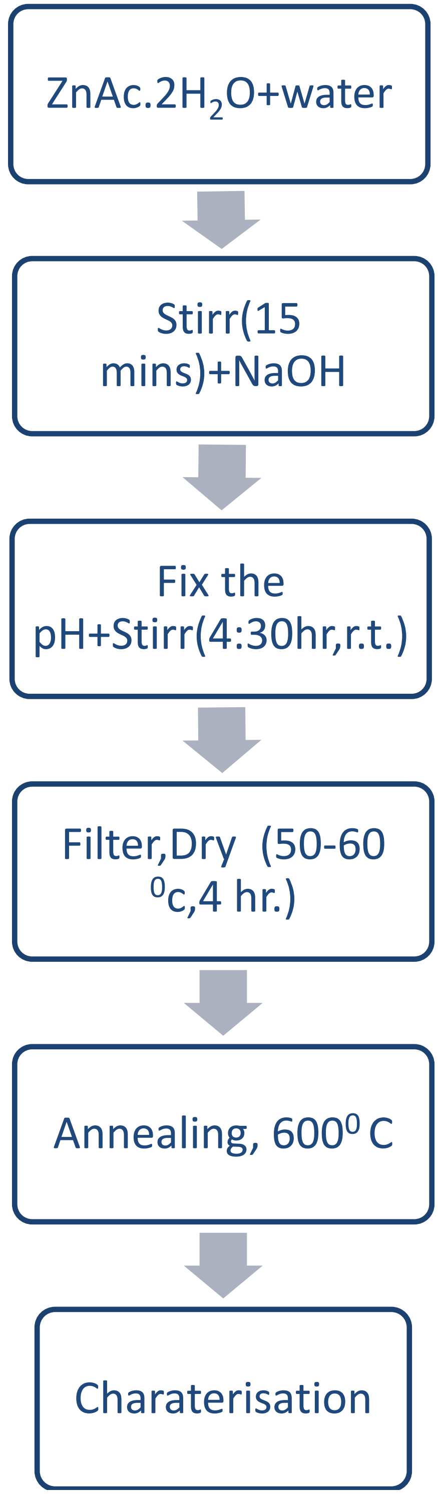


Fig3: Reference

As a result ,the solution of powder becomes thicker and for making it homogeneous solution. We kept the beaker at constant stirring for 4:30 hrs.

By doing same procedure in other sets, the difference is that we have added $CdAc.2H_2O$ with $ZnAC.2H_2O$ at a very first stage. One main thing is that we kept constants is amount of NaOH and water as given above in table. We kept these solution for constant stirring at room temperature.

After keeping every set at constant stirring and placing them for constant time i.e. 4:30 hrs, we kept them at a safe place for 24 hrs. So that water and other organic layer settle down and powder accumulate on the upper part. Then we filtered it by using the whatman filter paper (normal quality) and also give washing with water for three times so that we can get fresh powder. For removal of water and other organic layer we placed all the sets in heating oven at 50^0C for 4 hrs. The dried powders are kept safely in a petri dish and marked them 1, 2, 3, 4, 5 respectively.

The last stage is annealing in which powders are placed in different crucibles and kept them in Digital muffle furnace and by increasing the temperature slowly and kept these samples for 10mins at 100, 200, 300, 400, 500 and at 600^0C kept these sample for 1 hr for complete calcinations process. As a result there was formation of nanocomposites in the form of fine powder.

Ag/ZnO:

PROCESS:

The preparation of binary nano composites of Ag/Zno, Silver nitrate AR(169.872g/mol), Zinc acetate dihydrate (219.49g/mol) and Sodium hydroxide (39.99g/mol) are taken. The pH was maintained in all sets was 7-8. For 0%, 5%, 10%, 15%, 20% doping these are mix together in following manneras process chart is given below.

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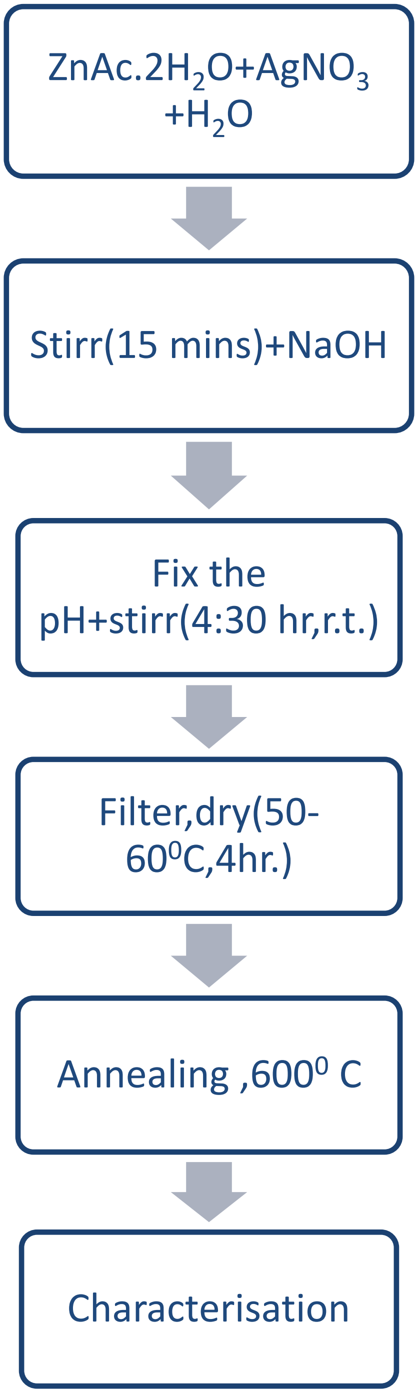

Fig 3:doping with Ag

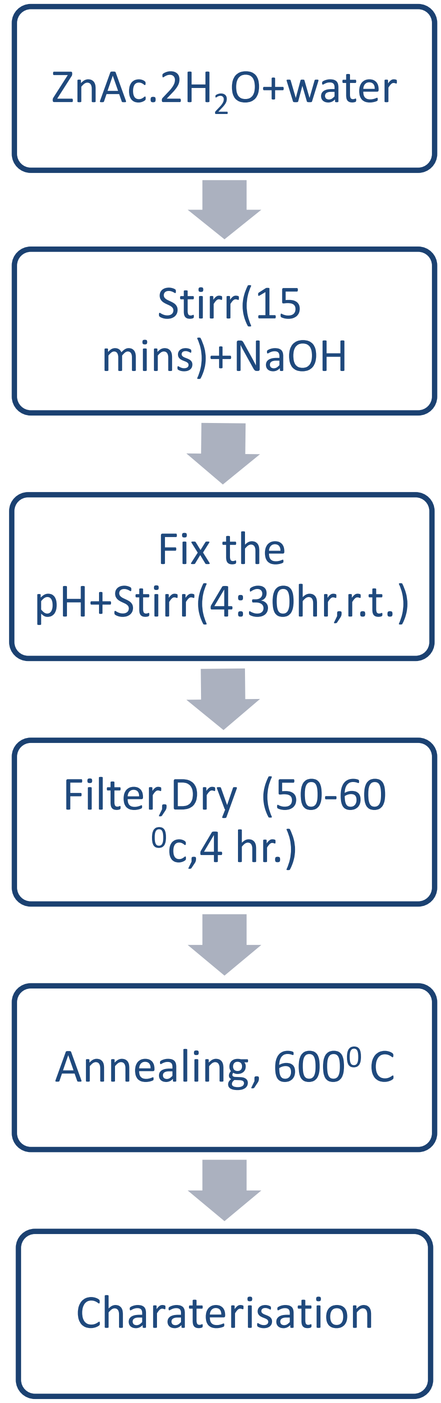

Fig 4: reference

As a result ,the solution of powder becomes thicker and for making it homogeneous solution. We kept the beaker at constant stirring for 4:30 hrs.

By doing same procedure in other sets, the difference is that we have added $AgNO_3$ with $ZnAC.2H_2O$ at a very first stage. One main thing is that we kept constants is amount of NaOH and water as given above in table. We kept these solution for constant stirring at room temperature.

After keeping every set at constant stirring and placing them for constant time (4:30 hr), we kept them at a safe place for 24 hrs. So that water and other organic layer settle down and powder accumulate on the upper part. Then we filtered it by using the whatman filter paper (normal quality) and also give washing with water for three times so that we can get fresh powder. For removal of water and other organic layer we placed all the sets on heating oven at 50^0C for 4:30 hrs. The dried powders are kept safely in a petri dish and marked them 1, 2, 3, 4, 5 respectively.

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CHAPTER 4

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Mat. Res. vol.12 no.1 São Carlos Jan./Mar. 2009

Nanocomposites: synthesis, structure, properties and new application opportunities

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