

T.C.
FIRAT UNIVERSITY
GRADUATE SCHOOL OF SCIENCES

**PRODUCTION AND ELECTRICAL CHARACTERIZATION OF
ZINC OXIDE BASED COMPOSITE MATERIALS**

MASTER DEGREE THESIS

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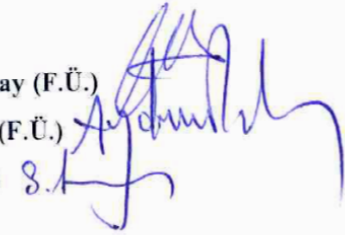
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Sevgili oğlum Ramiyar'a,
To my lovely son Ramiyar

PREFACE

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Safiye Jameel Biro
ELAZIĞ-2016

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ÖZET

ZnO ESASLI KOMPOZİT MALZEMELERİN ÜRETİLMESİ VE ELEKTRİKSEL KARAKTERİZASYONU

ZnO-CdO kompoziti hidrotermal yöntemle hazırlandı. Nanokompozitin yapısal, morfolojik, optik ve elektriksel özellikleri sırasıyla X-ışını kırınımı (XRD), FTIR, taramalı elektron mikroskobu (SEM), UV-VIS-NIR spektrofotometre ve iki prob kullanılarak gerçekleştirilmiştir. Termal kararlılığı belirlemek için TGA-DA analizi yapıldı. Bileşim analizi EDX ile yapıldı. Kompozitin bant aralığı, Zn prekürsörünün içeriğinin arttırılması ile birlikte sırasıyla 2.84, 2.78 and 3.06 eV olarak bulunmuştur.

Anahtar Kelimeler: Çinko oksit, hidrotermal metod.

SUMMARY

PRODUCTION AND ELECTRICAL CHARACTERIZATION OF ZINC OXIDE BASED COMPOSITE MATERIALS

ZnO-CdO composite was prepared with hydrothermal method. The structural, morphological, optical, and electrical properties of the nanocomposite have been carried out using X-ray diffraction (XRD), FTIR, scanning electron microscopy (SEM), UV-VIS-NIR spectrophotometer, and two probe, respectively. TG-DTA analysis was done to determine the thermal stability. The compositional analysis was done by EDX. The band gap of the composite were found to be 2.84, 2.78 and 3.06 eV respectively with increasing the content of Zn precursor.

Keywords: Zinc oxide, hydrothermal method.

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ABBREVIATIONS

CdO	: Cadmium oxide
CdO	: Cadmium oxide
CMC	: Ceramic Matrix Composite
CMNC	: Ceramic Matrix Nano-Composite
CMNC	: Metal Matrix Nano-Composite
CMNC	: Ceramic Matrix Nano-Composite
Cu₂O	: Cuprous oxide
FTIR	: Fourier Transform Infrared Spectroscopy
MMC	: Metal Matrix Composite
PMC	: Polymer Matrix Composite
PMNC	: Polymer Matrix Nano-Composite
SEM	: Scanning Electron Microscopy
ZnO	: Zinc Oxide

1. INTRODUCTION

1.1. Nanotechnology and Nanoscience

The idea of nanoscience and nanotechnology began with a famous talk “There’s Plenty of Room at the Bottom” by an American physicist Richard Feynman at the California Institute of Technology meeting on December 29, 1959. Thus he is known as father of this technology. A professor Norio Taniguchi of Tokyo University of science first time used this term of Nanotechnology [1].

Nanotechnology is the study of materials at nanoscale (1-100 nm) and application of the materials in various fields across all over the different science fields. Specially after the advancement in microscopy techniques such SEM (Scanning Electron Microscope), TEM (Tunneling Electron Microscope) and AFM (Atomic Force Microscope), huge number of research activities started to study the fantastic properties of materials at nanoscale. These properties can readily find their application in the field of medical sciences, chemistry, biology, physics, materials science and engineering. We are giving few applications of nanotechnology in various areas of life.

- We can make, antireflective, self-cleaning, anti-infrared and anti-ultraviolet, antifog, water-resistant, scratch-resistant, antimicrobial, or electrically conductive surfaces by making thin films on windows, eyeglasses, computer screens, camera displays, and other surfaces.
- Nanotechnology can be used to produce the green energy through solar cells. Reduced manufacturing costs, low temperature processing techniques such as printing can be used to produce flexible roll-to-roll printing instead traditional crystalline semiconductor solar cells.
- Batteries are very crucial parts of any electronic gadgets and even for the development of electrical cars. The surface of an electrode coated with nanoparticles can increase the electrode surface area thus allowing help flow more current between the electrode inside the battery. Ultimately we can achieve more powerful batteries, reduce charging time and less weight.
- Composite fiber synthesized with nano-sized fibers allows us to produce a lightweight water and stain repellent fabric.

- Ultra-thin material from carbon nanotubes just like few sheets together can be used in bullet proof vests.
- Nano-size transistors and integrated circuits are making our electronic devices cheaper, faster and low weight. It has brought revolution in field of electronics.
- Nanoscale reinforce additives in polymer composite materials can make lightweight, durable, stiff and resilient materials for different applications like tennis rackets, baseball bats, motorcycle helmets, automobile bumpers, and luggage bags.
- Nanoparticles are being used as catalysts to help chemical reactions increasingly everyday. These catalytic materials are necessary to control chemical reactions, saving money and reducing pollutants.
- Cosmetic industry uses nano-materials in their products like sunscreens, cleansers, shampoos, creams and lotions which provides greater clarity or coverage; cleansing; antioxidant, anti-microbial, and other health properties.
- In the field of medicine nanotechnology offers some exciting possibilities such as drug delivery and different disease treatments.

1.2. Classification of Materials

Long ago humans started learning use of different materials to make different tools which they used for hunting and for living. They even knew the use of different composite materials without fully understanding of these composites. For example, concrete is an important building material which provides us more strength to build compare to individual components. There are thousands of materials for various engineering applications. In general, all these materials can be classified into three categories metallic, ceramic and polymeric depending upon the atomic bonding forces of a particular material. Additionally, these materials can be combine together to form composites. Each class of these materials can further be divided into different groups based on chemical composition or certain physical or mechanical properties.

Composite materials are often divided into groups by the types of materials combined or the way the materials are arranged together. Below is the figure showing a commonly classification of materials within these four general groups of materials.

Metals • Ferrous metals and alloys (irons, carbon steels, alloy steels, stainless steels, tool and die steels) • Non-Ferrous metals and alloys (aluminum, copper, magnesium, nickel, titanium, precious metals, refractory metals, superalloys)	Polymeric • Thermoplastics plastics • Thermoset plastics • Elastomers
Ceramics • Glasses • Glass ceramics • Graphite • Diamond	Composites • Reinforced plastics • Metal-matrix composites • Ceramic-matrix composites • Sandwich structures • Concrete

Fig. 1.1. General classification of materials [2].

1.3. Composite Materials

With the development of technology and increasing importance and application of new materials in industry composite materials are attracting more attention. For several leading edge industries and technology applications such as spacecraft many rely on different kinds of composites. These heterogeneous materials combine the best properties of the individual constituent components.

Composite material is formed by combining two or more different materials and it results in better properties compared to the individual components used alone. In composite material, each material keeps its separate physical, chemical, and mechanical properties in contrast to metallic alloys. There are two constituents in a composite material, reinforcement and a matrix. The reinforcing component is embedded into the matrix to get

the composite. The composite materials provide us benefits like high strength and stiffness, combined with low density, weight reduction in the finished part when compared with bulk materials. The strength and stiffness is provided by the reinforcing phase. The reinforcement is stronger, and stiffer than the matrix, in most cases. The reinforcement is usually in the form of a fiber or a particulate.

Filler materials that are approximately round in all directions form particulate composite. The particulate can be of spherical form, platelets, or any other regular or irregular geometry. Particle reinforcing has disadvantage and provides less stiffness and less strength than continuous fiber composites, but on other hands it is much less expensive. These particulate reinforced composites due to processing difficulties and brittleness usually contain (up to 40 to 50 volume percent) less reinforcement [3, 4].

As mentioned above composites contain or made of matrix and doped or reinforcement part. The matrix can be a polymer, metal, or ceramic. We know that polymers have low strength and stiffness, ceramics have high strength and stiffness but are brittle, whereas metals are known for high ductility but have intermediate strength and stiffness. The matrix serves for several important functions, like proper orientation of fibers, spacing and protecting the fibers from abrasion and the environment in fibrous composites. In polymer and metal matrix composites, a strong bond between the fiber and the matrix formed and load is transmitted from the matrix to the fibers through shear loading at the interface.

In ceramic matrix composites, a low interfacial strength bond is required because the prime objective is often to increase the toughness rather than the strength. The final properties of composites are determined by type and quantity of the reinforcement [4-6].

1.4. Classification, Production and Application Fields of Composite Materials

Metal Matrix Composite (MMC) is composed with at least one metal and another material which could be another metal or ceramic or organic compound. There are three different solid, liquid and vapor phase composite manufacturing techniques.

Powder metallurgy is a well-known solid phase composite manufacturing technique. Metal powders and discontinuous reinforcement are mixed together. These powders are then bonded together and pass through different process like, degassing, and

thermo-mechanical treatment to get the composite. In foil diffusion bonding long fibers are sandwiched between the metal foils and hardly pressed to form the matrix.

There are several liquid phase methods to fabricate metal matrix composite. Electroplating and electroforming is a common method in which a solution containing metal ions loaded with reinforcing particles is co-deposited forming a composite material. In stir casting method, a discontinuous reinforcement is stirred into molten metal solution and then allowed to solidify. Spray deposition is another easy method for preparing metal matrix composites where a spray of molten metal is deposited onto a substrate with continuous fiber placed on it. Physical vapor deposition is a vapor phase technique in which pressurized metal vapors are passed through the fibers to prepare the composite.

There is a wide scope of metal matrix composites for variety of applications. These composites are expensive but the composites provide improved properties and performance which justify the cost. These MMCs offer us better properties such as improved electrical and thermal conductivity, radiation hardened, fire resistant, wider range of operating temperatures and do not absorb moisture making them effective for several applications [7].

- Cobalt matrix cemented with hard tungsten carbide particles is used for high performance tungsten carbide cutting tools [8].
- Boron nitride is a good reinforcement for steel because it is very stiff and it does not dissolve in molten steel. Steel reinforced composites can be used for tank armors.
- Automotive disc brakes are made with MMCs [7].
- Carbon fiber within a silicon carbide matrix is used in modern sport cars because of its high specific heat and thermal conductivity.
- Aluminum MMC compounds are used for bicycle frames for several years.
- Aluminum-Graphite composites are useful in power electronic modules due to the high thermal conductivity and the adjustable coefficient of thermal expansion [9].

Polymer Matrix Composite (PMC)s are made of polymer matrix with fibrous reinforcing typical glass, carbon or metal strands inside it. Polymer Matrix Composites are widely used due to their low cost and simple fabrication methods. Polymer-matrix composites are divided into two groups depending on the reinforced material used. The two kinds are reinforced plastics and advanced composites.

Reinforced plastics are made up of embedding short, flexible fiberglass in a polyester resin called E-glass, they are usually less resilient. In advanced composites fabrication we

use longer, stronger fiberglass, called S-glass, or carbon fibers inside a polymer matrix. High tensile strength, high fracture resilience, good corrosion resistance and low cost makes these composites a material of choice for several key industries such as aerospace and automobile industry [10].

Ceramic matrix composites (CMCs) are fabricated through embedding ceramic fibres into a ceramic matrix. These composites being low weight, higher thermal and chemical resistance makes them very attractive for the internal parts of gas turbines, missiles, exhaust systems for jets, aerospace, nuclear power plant and several industrial applications. The components made up of CMC composites have high thermal stability making them able to work at 1300 °C such as in gas turbines which normal composites can't withstand. The higher cost of these ceramic composite materials is a huge disadvantage but on the other hand the longer service life of CMC components compensates the higher cost of these components. Typical matrix is consisting of silicon carbide and alumina [11].

The manufacturing process usually consist of traditional fabrication processes mixing the reinforcing phase with matrix powder followed by hot pressing and sintering for composites with discontinuous reinforced phase. Composites reinforced with long fibers are manufactured by infiltrating matrix material in liquid or gaseous phase inside the lay-up fiber structure.

Nanocomposite is a multiphase material in which at least one of phase has one dimension in nanometer range. The properties of these nanocomposites not only depend on the parent individual reinforcing components. The properties of nano sized materials are greatly different from the bulk material. The fundamental properties of nano materials such as electrical, mechanical, optical and magnetic properties can be changed by controlling the morphology of these particles. Thus by controlling the morphology we can control the properties of nanocomposites without altering the chemical composition. We can combine the best properties of various nano structures possessed by each component creating a composite for advance applications. Depending on the type of nanoparticles added, the mechanical, electrical, optical, and thermal properties of polymer nanocomposites can be altered.

There are 3 types of nanocomposites according to their matrix parts :

- Ceramic-Matrix Nanocomposites (CMNC)

Ceramic materials are important for engineering applications such as in aerospace industry, turbines and automotive industry. Poor mechanical properties are big disadvantage of these ceramic materials. Several attempts were done to improve these properties by adding micron size additives or fibers to obtained ceramic composites. Recently microstructures with nanoscale size are incorporated to obtain ceramic matrix nanocomposites. This second phase dispersed in ceramic can be within the grains or at the grain boundaries. These dispersed nanoscale structures improve the mechanical properties at high temperatures of the CMNC. Sometimes a second metallic phase is dispersed in ceramics composite to improve fracture toughness.

- Metal-Matrix Nanocomposites (MMNC)

These are composites made by implanting nanoscale reinforced material into metal or alloy matrix. Carbon nanotube metal matrix composites (CNT-MMC) is an emerging new material. It has high strength, high conductivity, anti-corrosive and non-magnetic properties makes them excellent material for aerospace industry [12].

- Polymer-matrix nanocomposites (PMNC)

Polymer matrix nanocomposite consist of polymeric matrix with a second reinforcing phase having nanoscale dimension added into the matrix. The nanoscale reinforcing phase causes significant increase in interface to volume ratio. Adding nanoscale reinforcing into polymer matrix gives these composites multi functionality. We can improve the electrical and optical properties of polymer by adding nano particles in it. Thus we can have transparent, flexible, conducting polymer which are revolutionary for the flexible electronics.

1.5. Semiconductor Metal Oxides

In general, most oxides are good insulators, some, but some metal oxides for example CuO and Cu₂O, behave as semiconductors. Due to the less understanding of oxide semiconductors and their growth related processes, there are not many applications of these semiconductor oxides today. Zinc oxide (ZnO) is one exception, which has found application as a transducer, in solar cells and biomedical applications. However, after the discovery of superconductivity in many oxides of copper, the situation has changed lanthanum copper oxide (La₂CuO₄) is the first so-called high-T_c superconductor, discovered by Muller and Bednorz is based on the semiconductor. Lanthanum copper oxide has a bandgap of about 2 eV. Charge carriers in form of holes are created into La₂CuO₄ by replacing divalent barium (Ba) or strontium (Sr) with trivalent lanthanum (La) or when an excess of oxygen is present. When sufficient carriers are available the semiconductor exhibits properties of superconducting metal [13-22].

Metal oxide semiconductors have been extensively explored due their wide variety of applications and their interesting structural, electrical and optical properties. Among these ZnO has gained considerable attention because of its extraordinary properties, with hexagonal crystal structure and direct wide bandgap (3.37 eV at room temperature) [23]. ZnO has been applied for a variety of important practical applications such as solar cells, thin film transistors, photodiodes, chemical and gas sensors, optical and magnetic memory devices, and more [24]. There are number of techniques for the preparation of ZnO thin films like spray pyrolysis [25], plasma enhanced chemical vapor deposition [26], thermal evaporation [27], magnetron sputtering [28], and sol-gel method [29]. Recently, numerous reports on the effect of dopant concentration (Al, Cu, Ni, F, B, Ag, Na, In, Li) on the structural, optical, and electrical properties have been reported [30-33].

Among other metal oxides CdO, CuO, SnO₂ and In₂O₃ have attracted significant attention for wide applications. CdO has a cubic structure and a narrow band gap of 2.3 eV is regarded an important n-type semiconductor material for optoelectronic devices. But the main issue for CdO thin films is low band gap for wider applications. By alloying with ZnO with CdO, the band gap of composite can be red-shifted to blue, or even green light spectra range. Moreover, the incorporation of Cd into ZnO is very useful for the fabrication of ZnO/Zn_{1-x}Cd_xO heterojunction and super lattice, which is the key element in ZnO-based

light emitters and detectors [34-36]. ZnO-CdO composites have been prepared such as nanowires [37], hollow micro-nanospheres [38], nanorods [39] previously.

1.6. Aim Of The Study

The aim of this work is to produce zinc oxide semiconductor based composite materials and study the optical, structural and electrical properties of oxide composites.

2.MATERIALS AND METHODS

2.1. Energy Dispersive X-ray

Energy-dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. The main principle of spectroscopy is based on the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum [40].

A high-energy beam of charged particles such as electrons is focused onto the sample causing stimulated emission of characteristic X-rays from a specimen being studied as shown in Fig. 2.1. In the ground state of an atom within the sample electrons are in discrete energy levels or electron shells bound to the nucleus. The incident beam with appropriate energy may eject an electron from the inner shell, while creating an electron hole where the electron was. An electron from, higher-energy shell then fills the vacant position, by releasing energy equal to the difference in energy between the higher-energy shell and the lower energy shell in the form of an X-ray. The energy-dispersive spectrometer detector can measure the number and energy of the X-rays emitted from the specimen. As the energies of the X-rays are characteristic energies of the atomic structure of the elements from which it was emitted. Thus EDS provides us a tool for the elemental composition of the specimen to be measured. Four primary components of the EDS setup are

the excitation source (electron beam or x-ray beam)

- X-ray detector
- Pulse processor
- Analyzer

The EDX detector measure relative abundance of x-rays against their energy and converts into voltage signals. These voltage signals are further sent to pulse processor and analyzer for analysis and data display. Silicon drift detectors (SDD) are most common detector these days. The spectrum of x-ray energies vs. counts is used to determine the chemical composition of the sample [40].

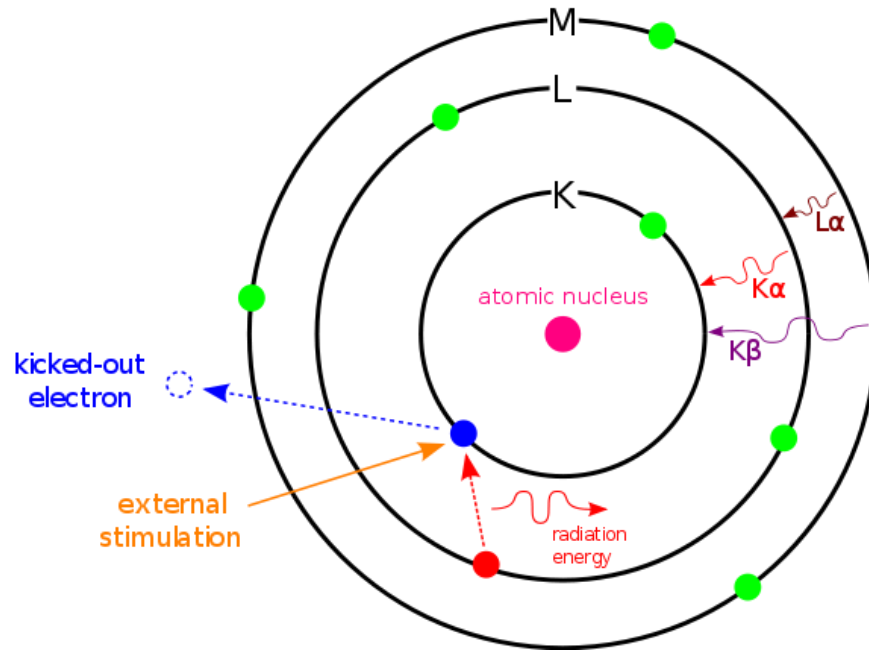


Fig. 2.1. Basic Principle of Energy Dispersive X-rays [40].

2.2. Scanning Electron Microscope

A scanning electron microscope (SEM) is a type of electron microscope widely used for surface analysis. It provides surface images of a sample by scanning it with a focused beam of electrons. High resolution images of surface topography and composition are produced by the electrons interact with atoms in the sample, producing various signals that contain information about surface. When the high energy accelerated electrons interact with the sample various signals such as secondary electrons, backscattered electrons, x-rays and heat is produced. Secondary electrons are used for showing morphology and topography on the specimen while the backscattered electrons are used for showing contrasts in composition in a multiphase specimen. The electron beam is generally scanned in a raster scan pattern, and collecting the secondary electrons using a special detector and an image displaying the topography of the surface is created. SEM can achieve resolution about 10 nanometer. Specimens can be observed in high vacuum [41].

Essential components of all SEMs include the following:

- Electron Gun
- Electromagnetic Lenses

- Sample Stage
- Detectors
- Power Supply
- Vacuum System
- Cooling system
- Vibration-free floor

The general overview of the different components of the SEM is shown in the Fig. 2.2 below.

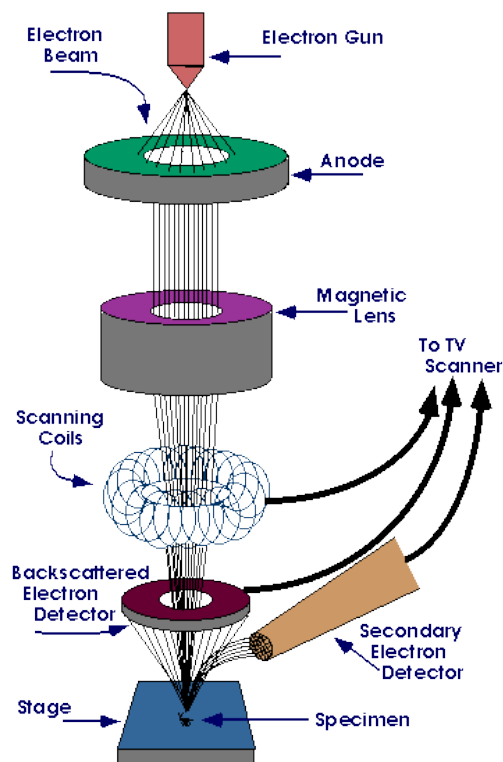


Fig. 2.2. Overview of a typical SEM [41].

2.3. Fourier Transform Infrared Spectroscopy

FTIR spectroscopy (Fourier Transform-Infrared Spectroscopy) is widely used an analytical technique to identify materials. When the infrared radiations are passed through the specimen some of the radiations are being absorbed and some are transmitted through

the sample. Using the same principle of Michelson interferometer light with different wavelength is irradiated on the sample. The spectrometer detector measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands represent the fingerprint identifying a unique molecule. The commonly used region for infrared absorption spectroscopy is $4000\text{--}400\text{ cm}^{-1}$. A mathematical technique Fourier transformation is used to convert signals from detector into the actual spectrum.

The main objective of any absorption spectroscopy such as FTIR or ultraviolet-visible (UV-Vis) spectroscopy is to measure amount of light absorbed at each wavelength. In UV-Vis spectrometers a monochromatic light is shined at the sample and the amount of absorbed light at different wavelengths is measured. Whereas, in FTIR spectroscopy a less intuitive way is used and a beam of light containing different frequencies at once is shinned at the specimen and amount of absorbed light is measured. Then a beam modified with different set of frequencies gives us a second set of data. This process is repeated several times and computer process this raw data by fourier transformation and finally a spectrum is obtained which is sometimes called an "interferogram".



Fig. 2.3. Thermo Scientific iS5 FTIR.

2.4. UV-Vis Spectrophotometer

Ultraviolet visible (UV-Vis) spectrophotometer measures the attenuation of a beam of monochromatic light according to Beer's law when passed through a sample. The ratio of intensity of light when it is passed through a specimen and same light passed through a reference sample (usually opaque) is measured. This comparison of two intensities gives us the amount of light absorbed by the sample when it passed through it. We can also measure the reflectance by taking the ratio of intensities of reflected light from the specimen and reference sample. A spectrophotometer consists of a light source, monochromator and detector.



Fig. 2.4. Shimadzu -3600 UV-VIS-NIR spectrophotometer.

2.5. Hydrothermal Synthesis

Hydrothermal synthesis is a popular technique to synthesize metal oxides and nanocomposites. Metal oxide nanoparticles are of great interest for their potential applications in the field of bio-imaging, storage devices, drug delivery and various electronic devices. There are several reports for growth of these metal oxide nanoparticles such as ZnO, TiO₂, CuO, CeO₂ and ZrO₂ etc.[42,43] using hydrothermal technique. In hydrothermal technique crystallization from the aqueous solution at high temperature and pressure takes place. This provides us an environmentally friendly method for synthesis. Water is being used as solvent and its polarity can be controlled at hydrothermal conditions. We can control different parameters like pressure, temperature and growth time in order to get various morphologies and particle sizes are possible. A reaction chamber called “Autoclave” is used for hydrothermal synthesis.

2.6. Experimental Method

Zinc nitrate hexahydrate $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Carlo Erba, Analytical grade) and Cadmium nitrate tetrahydrate $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (Carlo Erba, Analytical grade) were used as precursor materials. Zinc nitrate hexahydrate 0.5M was dissolved in 8ml distilled water and 32ml ethylene glychol. Separately Cadmium nitrate 0.8M was dissolved in 8ml distilled water and 32ml ethylene glychol. Both the zinc and cadmium solutions were mixed together in volume ratio (40ml:40ml) and so the total volume of solution was 80ml. The final solution was transferred to autoclave and heated at 150 °C for 10 hours. The second and third samples were prepared with following volume ratio as below:

sample 1- $\text{Cd}(\text{NO}_3)_2$ solution : $(\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ solution = 40ml : 40 ml

sample 2- $\text{Cd}(\text{NO}_3)_2$ solution : $(\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ solution = 20ml : 60 ml

sample 3- $\text{Cd}(\text{NO}_3)_2$ solution : $(\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ solution = 10ml : 70 ml

After the autoclave, the obtained powder was filtered and washed several times with distilled water. Then the powder was dried at 60 °C for 24 hours. Finally the powder samples were calcinated at 450 °C.

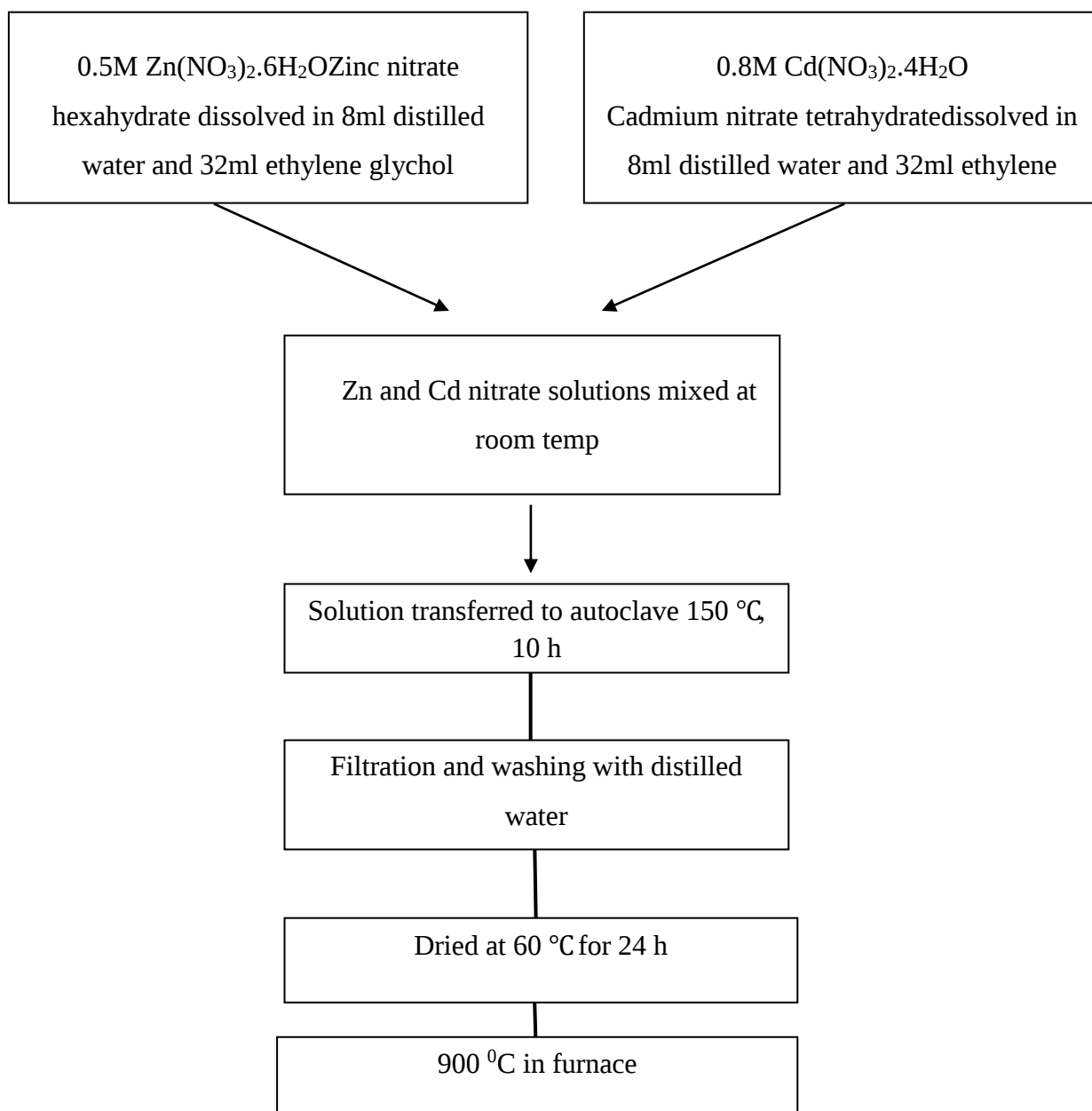


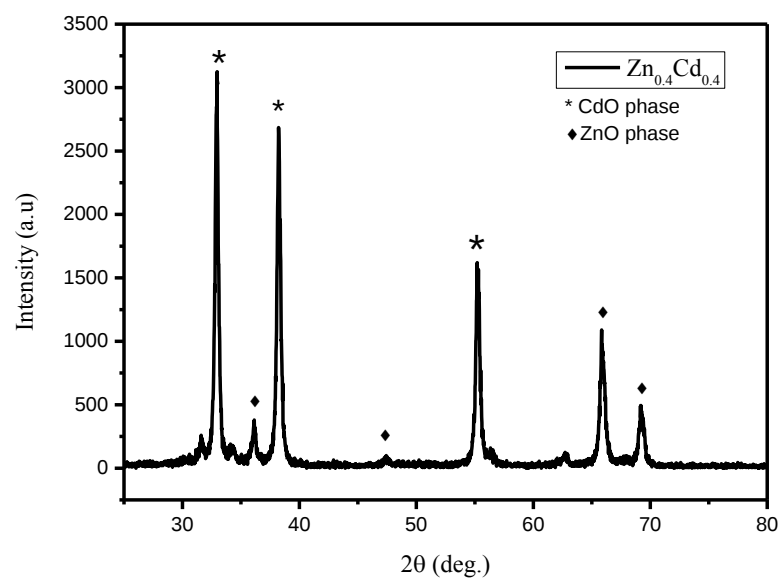
Fig. 2.5. Schematic display of steps to synthesis ZnO-CdO composite by hydrothermal method.

The crystal phase of the prepared films was investigated using Rigaku-Ultima-IV X-ray diffractometer, utilizing Cu K α radiation ($\lambda = 0.15406$ nm) operated at 40 kV, 30 mA. FTIR spectroscopy analysis was done for compositional analysis (Thermo Scientific iS5). The optical spectra were measured by UV-VIS-NIR spectrophotometer (Shimadzu - 3600PC). JEOL JSM-7001F scanning electron microscopy (SEM) were employed to study the morphology of the films.

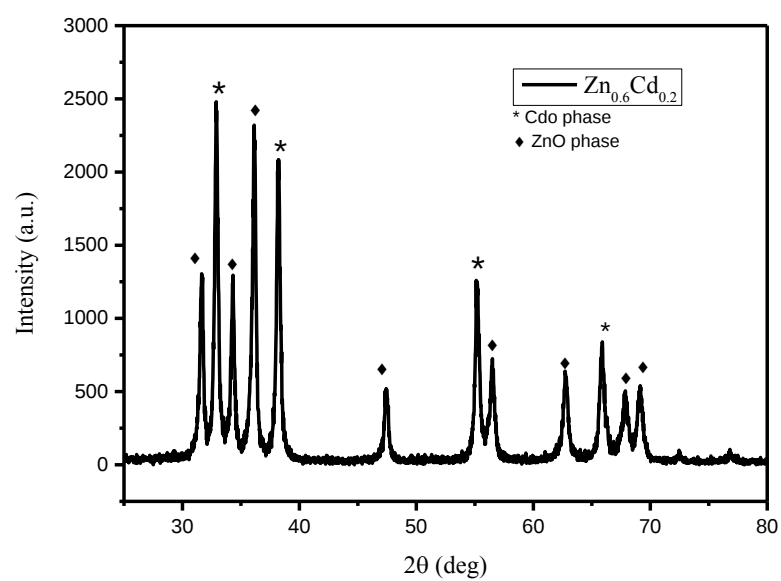
3. RESULTS AND DISCUSSION

3.1. Structural Analysis

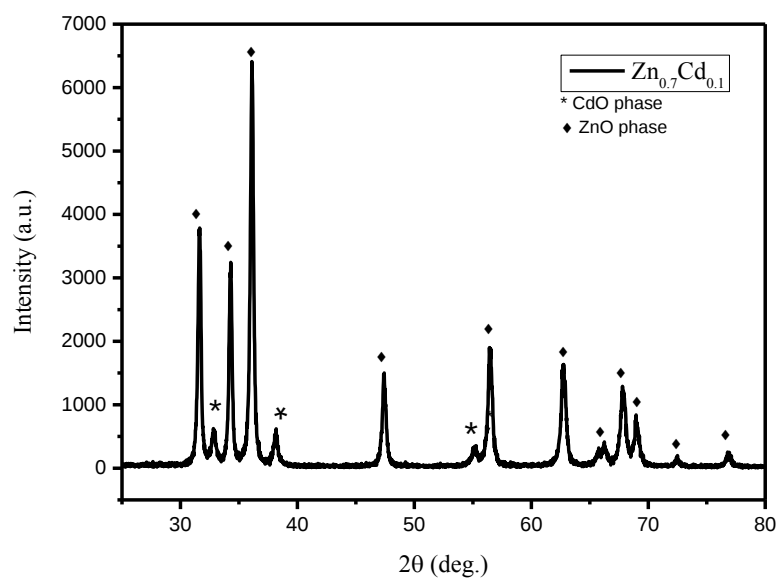
Fig. 3.1 shows the XRD pattern of ZnO-CdO nanocomposite powder through hydrothermal method. Three samples were prepared by mixing different volume ratio of $\text{Zn}(\text{NO}_3)_2$ and $\text{Cd}(\text{NO}_3)_2$ solutions. The prepared ZnO-CdO nanocomposite diffraction pattern shows both phases of hexagonal ZnO and cubic CdO. In the Fig. 3.1. (a) for sample $\text{Zn}_{0.4}\text{Cd}_{0.4}$ the major three peaks correspond to CdO phase and matches very well JCPDS Card# 005-0640. CdO phase is dominant due to presence of sharp peak in the patterns. The CdO peaks are represented by “*” sign and rest of the peaks correspond to ZnO phase matches with JCPDS Card # 036-1451 (Fig. 3.2.). In the second sample $\text{Zn}_{0.6}\text{Cd}_{0.2}$ zinc oxide phase becomes more prominent as compared to $\text{Zn}_{0.4}\text{Cd}_{0.4}$ because the volume of Zn precursor solution is increased as compared to first sample. The diffraction pattern is shown in Fig. 3.1. (b) for the $\text{Zn}_{0.6}\text{Cd}_{0.2}$ sample. As clearly seen from Fig. 3.1. (c) the diffraction pattern for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ sample, the ZnO phase is dominant as compared to CdO because volume ratio of Zn precursor is increased further. We can see from Fig 3.1. (d) that all the major peaks belong to zincite phase with hexagonal wurtzite crystal structure. The XRD diffraction pattern confirms the composition stoichiometry of the three samples prepared.



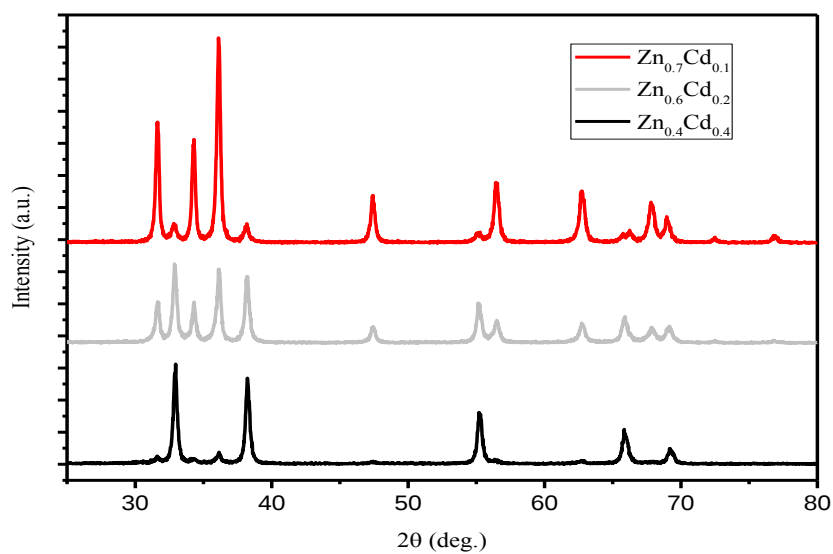
-a-



-b-

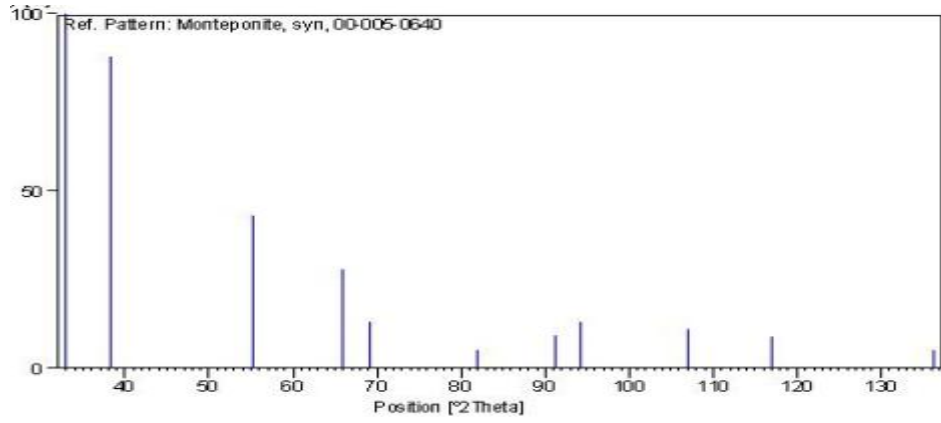


-c-

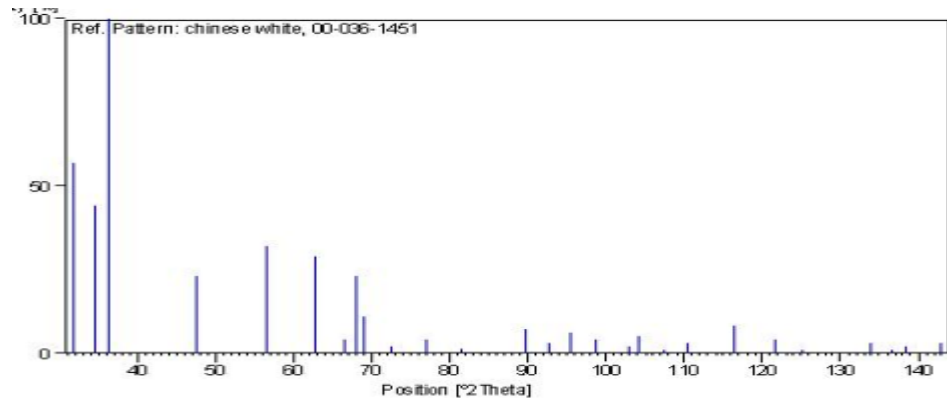


-d-

Fig. 3.1. Diffraction pattern for (a) $\text{Zn}_{0.4}\text{Cd}_{0.4}$ (b) $\text{Zn}_{0.6}\text{Cd}_{0.2}$ (c) $\text{Zn}_{0.7}\text{Cd}_{0.1}$ (d) comparison of all the three samples.



-a-



-b-

Fig. 3.2. Reference pattern(a) JCPDS Card# 005-0640 for CdO (b) JCPDS Card # 036-1451 for ZnO.

The values of d -spacing, FWHM, and relative intensity corresponding to x-ray diffraction peaks for all three samples have been tabulated in Table 3.1. It is observed from the Table 1 that characteristic peak (111) corresponding to CdO phase shifts from standard 33.002 towards lower angle. The ionic radii of Zn^{+2} (0.74) is smaller than Cd^{+2} (0.97). Considering the similar electronegativities of both Zn and Cd therefore Zn ions can easily substitute the Cd ions crystallographic positions. The replacement of Cd ions replaced by a smaller Zn ions as the Zn precursor volume is increased causes increase in d values and corresponding decrease in 2θ towards lower angle. The similar results have been reported in [44]. The value of lattice strain has been determined using the relation given below [45]:

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (3.1)$$

where β is the fullwidth (FWHM). The value of lattice strain obtained in this manner have been given Table 3.2. With the increase in volume ratio of Zn precursor the lattice strain decreases. The value of crystallite size can be evaluated from Scherrer formula [45]:

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (3.2)$$

where k is the shape factor, λ is the wavelength of x-rays, and θ is the diffracting angle. The value of crystallite size determined in this manner have been given in Table 3.2. There is slight increase in the value of crystallite size on increasing the volume of Zn precursor solution. Karthik et al [46] has also reported CdO-ZnO composite XRD diffraction pattern and calculated the microstrain values.

Table 3.1. Miller planes, 2θ , d-spacing, FWHM, percentage intensity for ZnO-CdO nanocomposite samples.

Sample	(hkl)	$2\theta(^{\circ})$	$d(A^{\circ})$	$d(A^{\circ})$ Standard	FWHM ($^{\circ}$)	Intensity (%)
Zn _{0.4} Cd _{0.4}	111*	32.94	2.7169	2.7120	0.342	100
	200*	38.22	2.3528	2.3490	0.353	90.3
	220*	55.20	1.6626	1.6610	0.382	54
Zn _{0.6} Cd _{0.2}	111*	32.90	2.7201	2.7120	0.325	100
	101	36.12	2.4846	2.4759	0.368	92.9
	200*	38.24	2.3517	2.3490	0.366	84.6
	100	31.64	2.8255	2.8143	0.388	52.7
	002	34.30	2.6122	2.6033	0.304	51.5
	220*	55.22	1.6621	1.6610	0.424	50.3
Zn _{0.7} Cd _{0.1}	101	36.12	2.4847	2.4759	0.327	100
	100	31.62	2.8272	2.8143	0.320	59.7
	002	34.30	2.6123	2.6123	0.300	49.7

Table 3.2. 2θ , d-spacing, FWHM, Crystallite size, Dislocation and Strain for ZnO-CdO nanocomposite samples.

Sample	$2\theta(^{\circ})$	$d(A^{\circ})$	FWHM ($^{\circ}$)	Crystallite size (nm)	Dislocation δ ($\cdot 10^{-4}$ nm) ²	Strain ($\cdot 10^{-3}$)
Zn _{0.4} Cd _{0.4}	32.94	2.7169	0.342	24.23406	17.0	1.430
Zn _{0.6} Cd _{0.2}	32.90	2.7201	0.325	25.49906	15.4	1.359
Zn _{0.7} Cd _{0.1}	36.12	2.4847	0.327	25.56505	15.3	1.356

3.2. FTIR Analysis

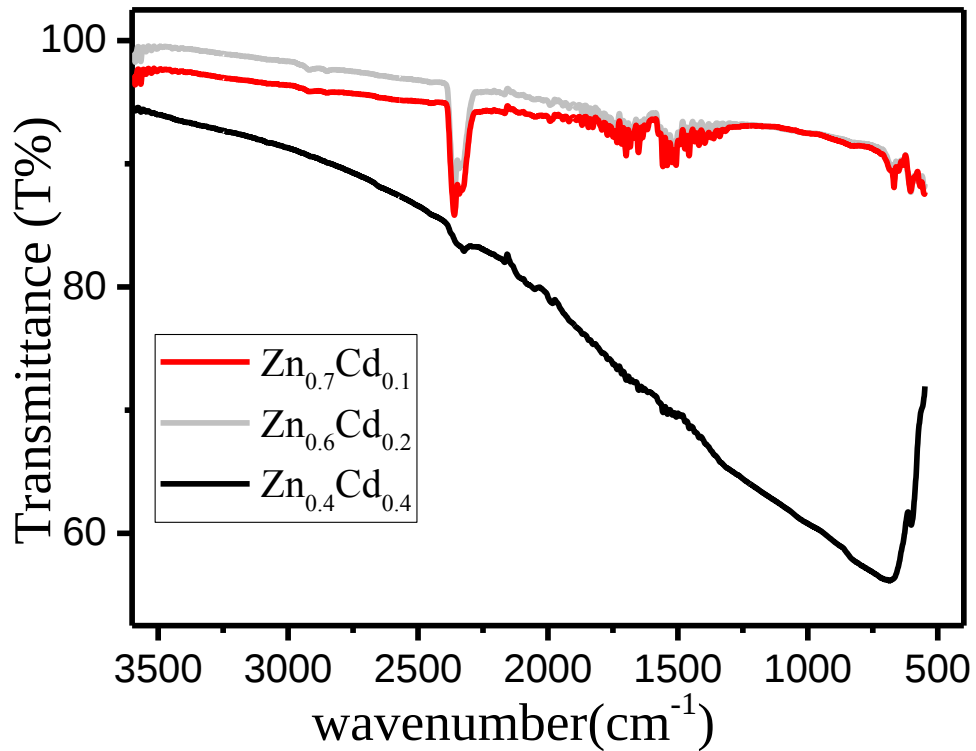


Fig. 3.3. FTIR spectra for ZnO-CdO composite for Zn_{0.4}Cd_{0.4}, Zn_{0.6}Cd_{0.2} and Zn_{0.7}Cd_{0.1} samples.

The FTIR spectra for ZnO-CdO nanocomposite has been recorded to study the various functional groups of nanocomposite shown in Fig. 3.3. The absorption band in the region of 3426 cm⁻¹ corresponds to the O-H stretching vibrations of water present in the powder sample. The band in the region 1600-1500 cm⁻¹ corresponds to the vibrations of a carboxyl group (CO). The characteristic wurtzite lattice vibrations (Zn-O) are

corresponding to the broadband in the range 400-600 cm^{-1} [47,48]. We can clearly see the peaks around 600-500 cm^{-1} in sample $\text{Zn}_{0.6}\text{Cd}_{0.2}$ and $\text{Zn}_{0.7}\text{Cd}_{0.1}$ as we increase the Zn volumetric ratio in the composite. The well known stretching mode of CdO was observed at 1420 cm^{-1} [44].

3.3. Morphological Analysis

The surface morphology of ZnO-CdO nanocomposite was studied using FESEM at various magnification and shown in Fig. (3.4-3.6). The morphology consist of spherical, non-spherical and partly cylindrical structures. The Fig. 3.4. (e) for $\text{Zn}_{0.4}\text{Cd}_{0.4}$ clearly shows at x100000 magnification the formation of typical spherical structures. When can observe at low magnification the number of rounded granular structure increases. In second sample $\text{Zn}_{0.6}\text{Cd}_{0.2}$ (Fig. 3.5. (b)) at x30000 magnification the connectivity between partially cylindrical structures is found which might be developed due to sintering necks between spherical particles. When further increasing the Zn content we can see the SEM micrographs of third sample $\text{Zn}_{0.7}\text{Cd}_{0.1}$ (Fig. 3.6. (a-e)) the morphology of the composite changes. The compositional analysis of ZnO-CdO comosite was confirmed by EDX. The EDX spectra for each sample is shown below the SEM micrographs. The spectra clearly shows the presence of Zn, O, and Cd elements along with peak of Au. The Au peak is due to the coating of Au film on the powder samples before FESEM/EDX.

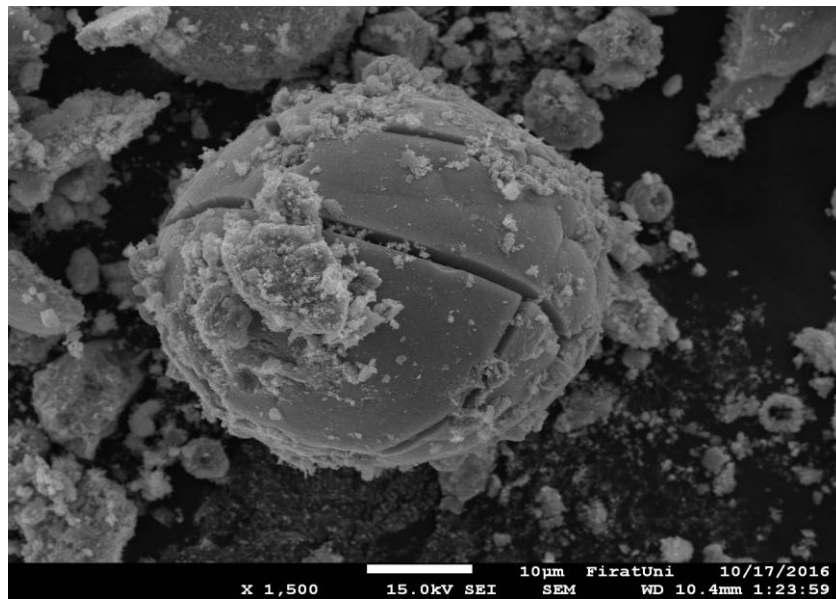


Fig.3.4. (a). SEM micrograph for $\text{Zn}_{0.4}\text{Cd}_{0.4}$ at 1500x magnification.

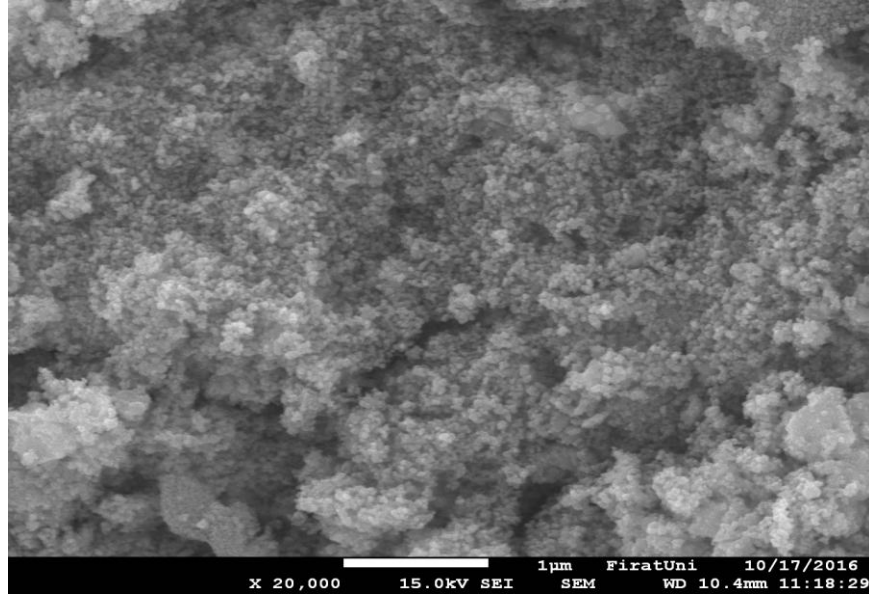


Fig.3.4. (b). SEM micrograph for Zn_{0.4}Cd_{0.4} at 20000x magnification.

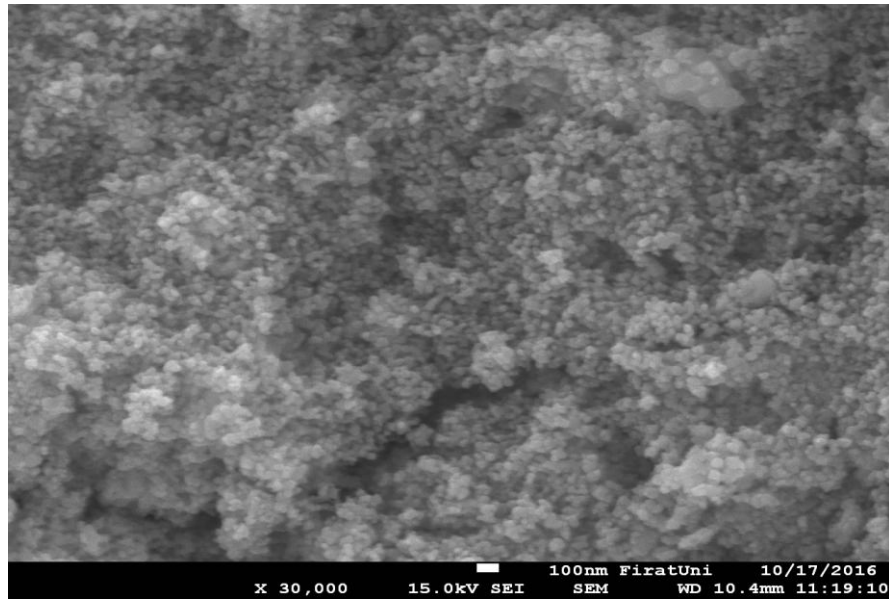


Fig.3.4. (c). SEM micrograph for Zn_{0.4}Cd_{0.4} at 30000x magnification.

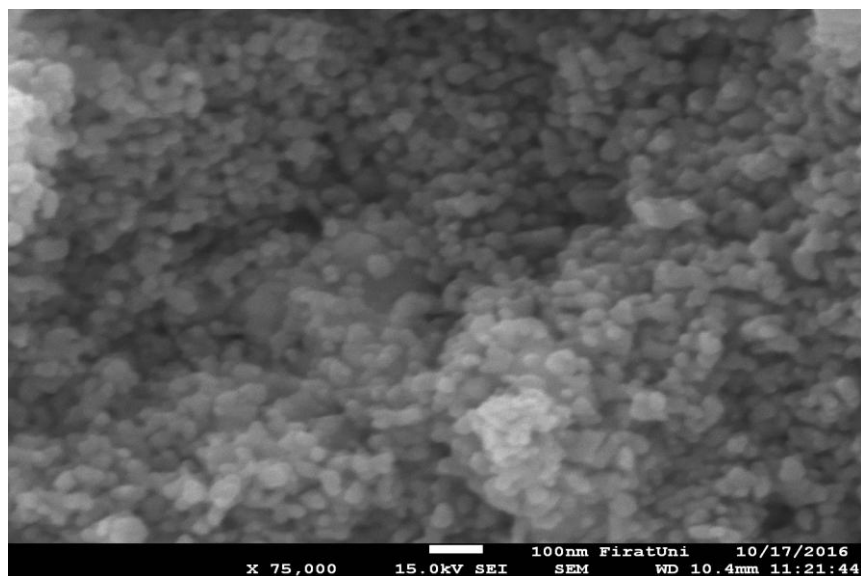


Fig.3.4. (d). SEM micrograph for $\text{Zn}_{0.4}\text{Cd}_{0.4}$ at 75000x magnification.

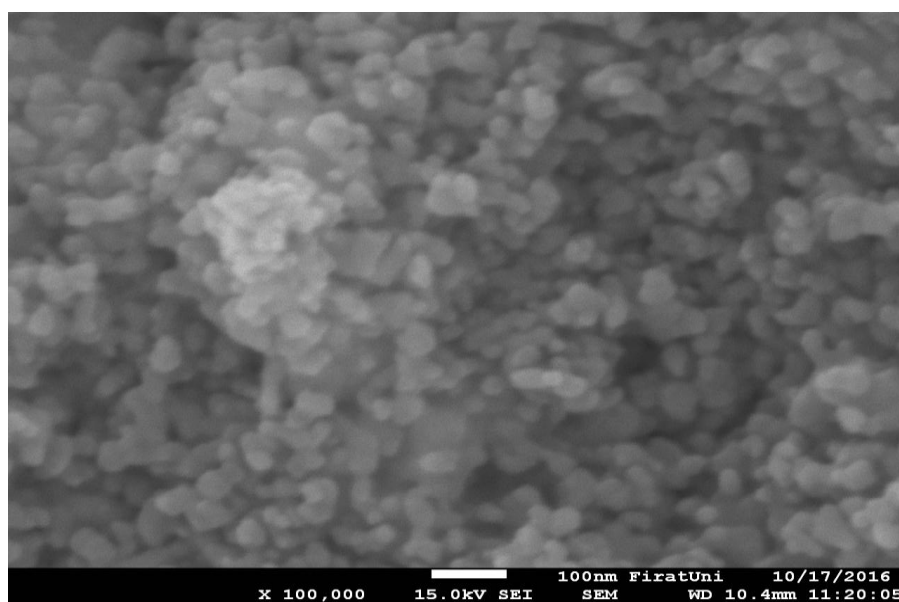
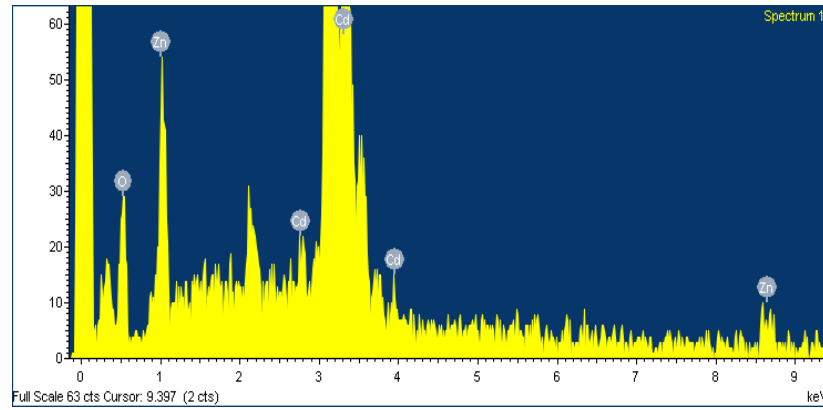


Fig.3.4. (e). SEM micrograph for $\text{Zn}_{0.4}\text{Cd}_{0.4}$ at 100000x magnification.



Element	Weight%	Atomic%
O K	14.50	52.28
Zn L	10.41	9.19
Cd L	75.09	38.54
Totals	100.00	

Fig. 3.4. (f). EDX analysis for Zn_{0.4}Cd_{0.4} sample.

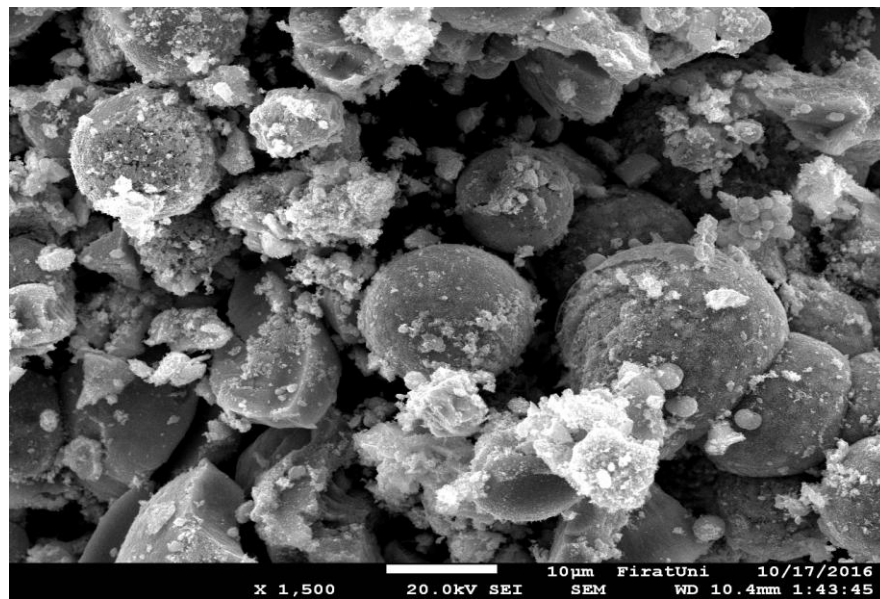


Fig.3.5. (a). SEM micrograph for Zn_{0.6}Cd_{0.2} at 1500x magnification.

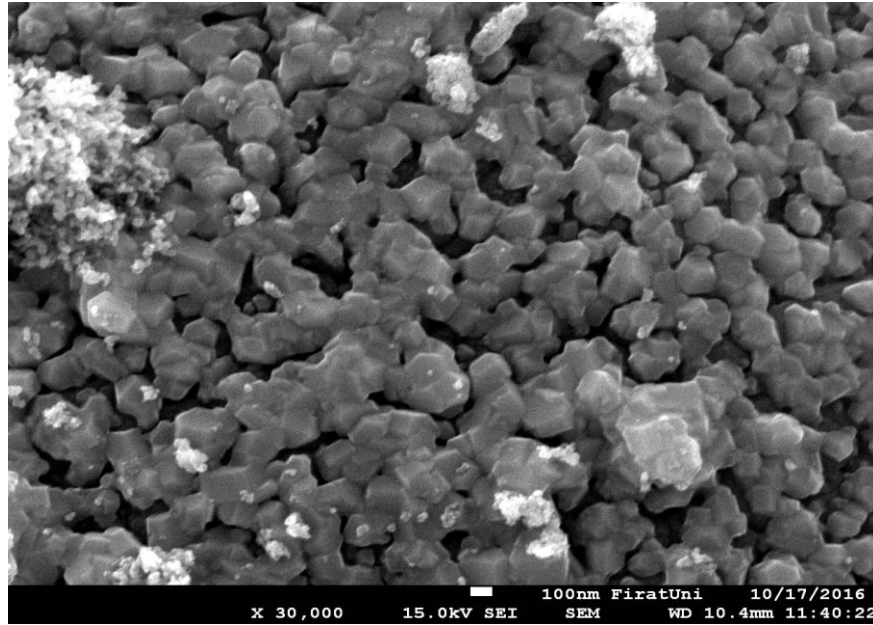


Fig.3.5. (b). SEM micrograph for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ at 30000x magnification.

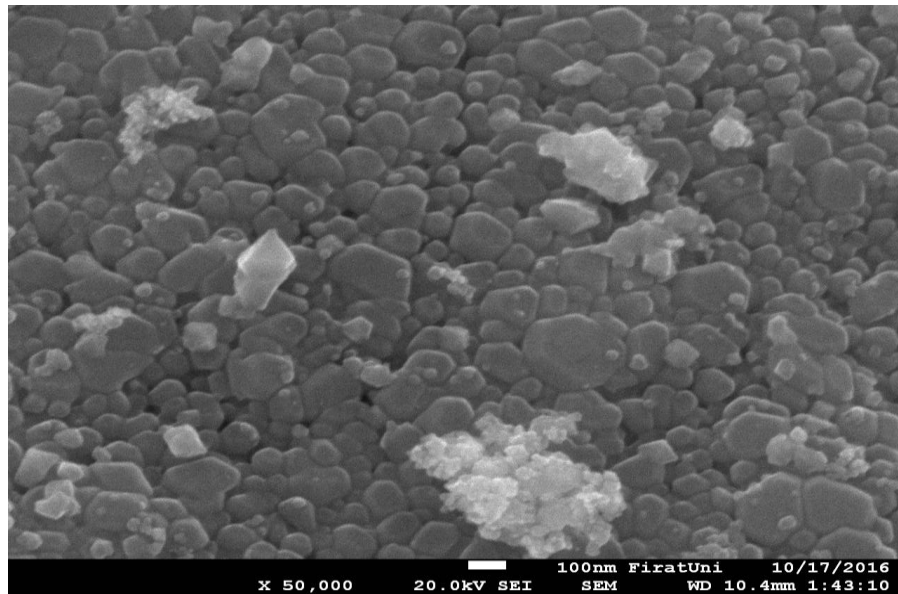


Fig.3.5. (c). SEM micrograph for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ at 50000x magnification.

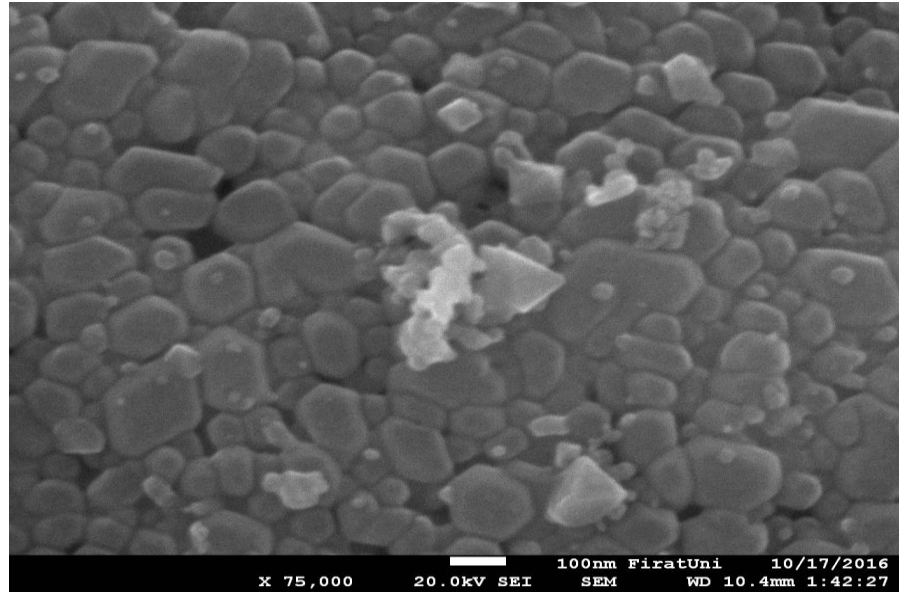


Fig.3.5. (d). SEM micrograph for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ at 75000x magnification.

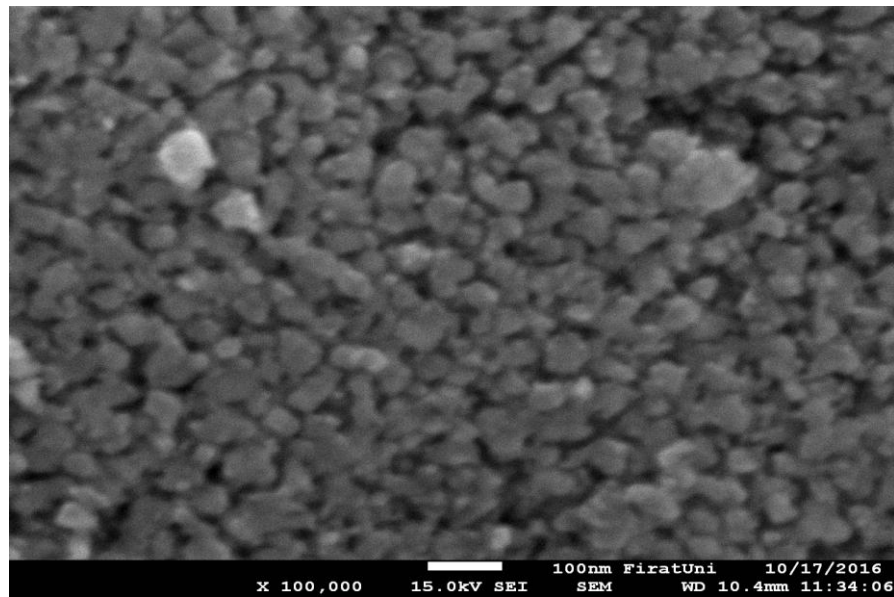
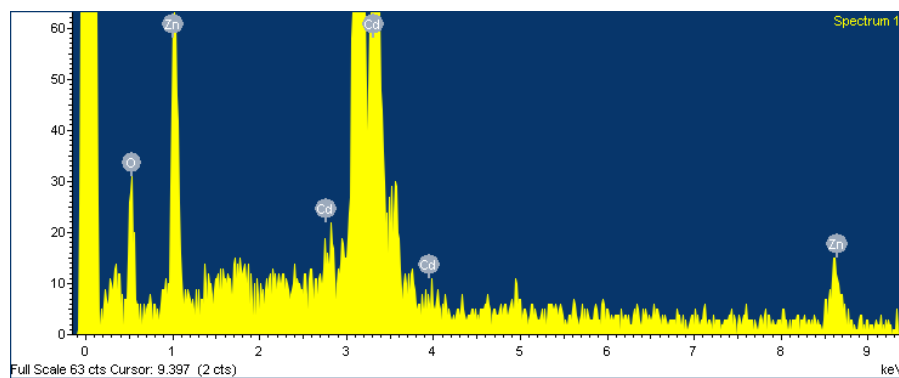


Fig.3.5. (e). SEM micrograph for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ at 100000x magnification.



Element	Weight%	Atomic%
O K	16.11	54.32
Zn L	15.69	12.95
Cd L	68.20	32.73
Totals	100.00	

Fig.3.5. (f). EDX analysis for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ sample.

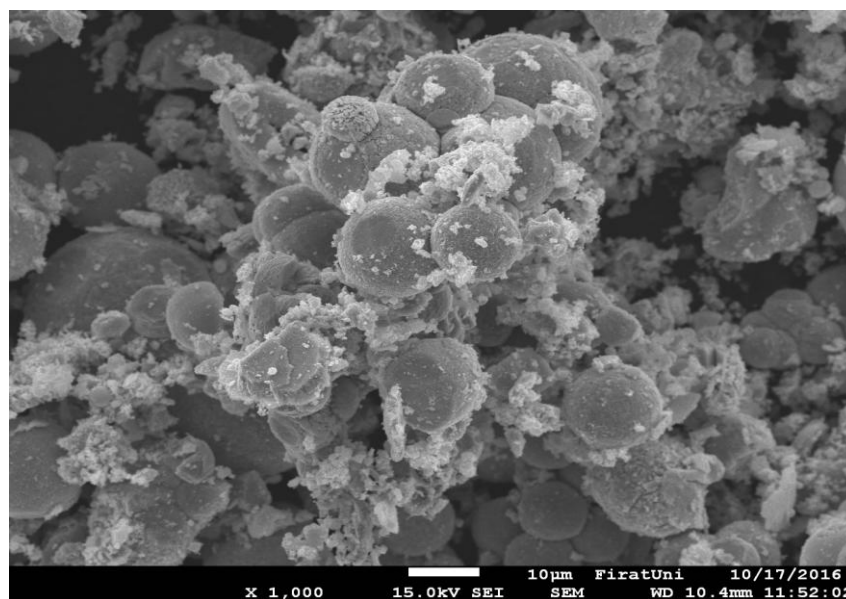


Fig.3.6. (a). SEM micrograph for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ at 1000x magnification.

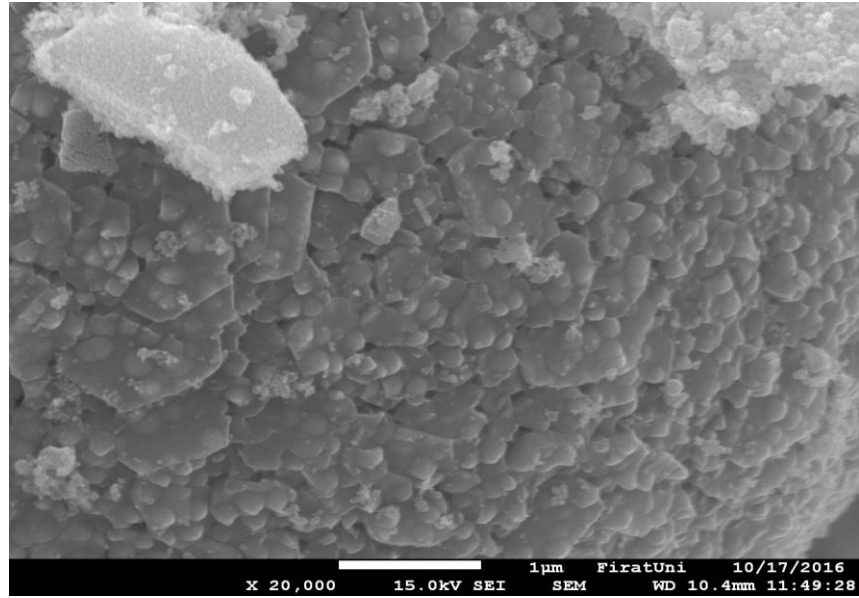


Fig.3.6. (b). SEM micrograph for Zn_{0.7}Cd_{0.1} at 20000x magnification.

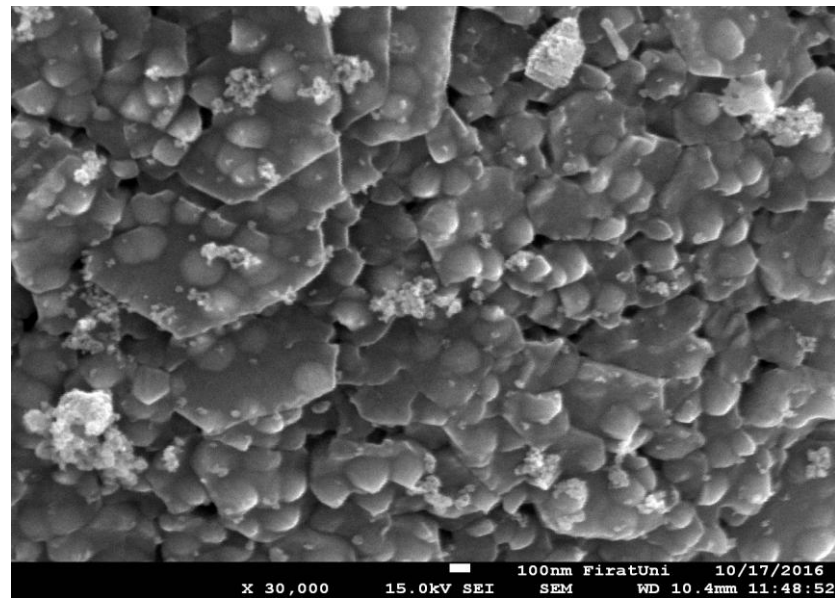


Fig.3.6. (c). SEM micrograph for Zn_{0.7}Cd_{0.1} at 30000x magnification.

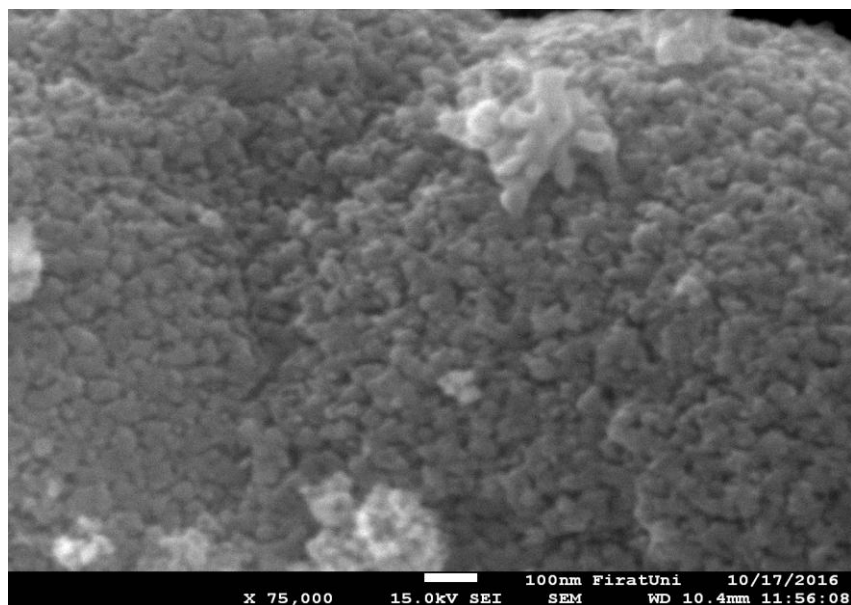


Fig.3.6. (d). SEM micrograph for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ at 75000x magnification.

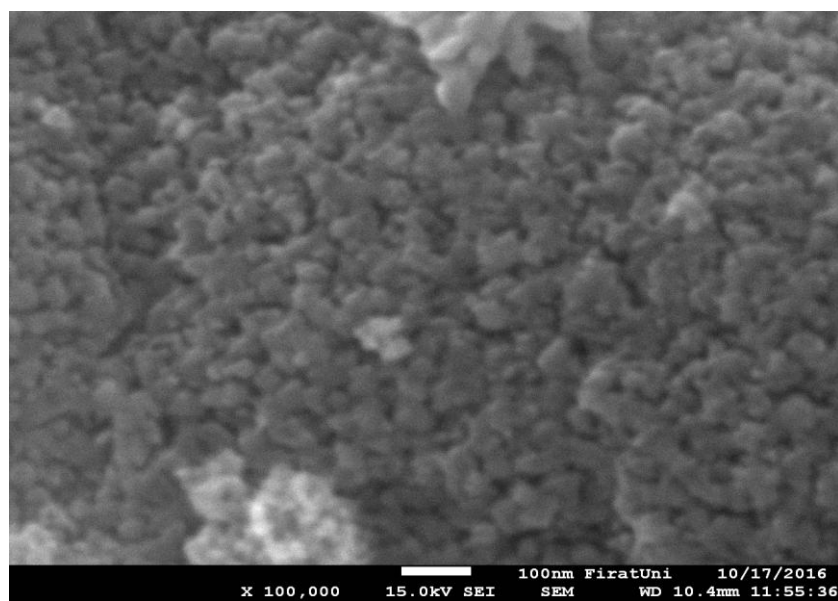
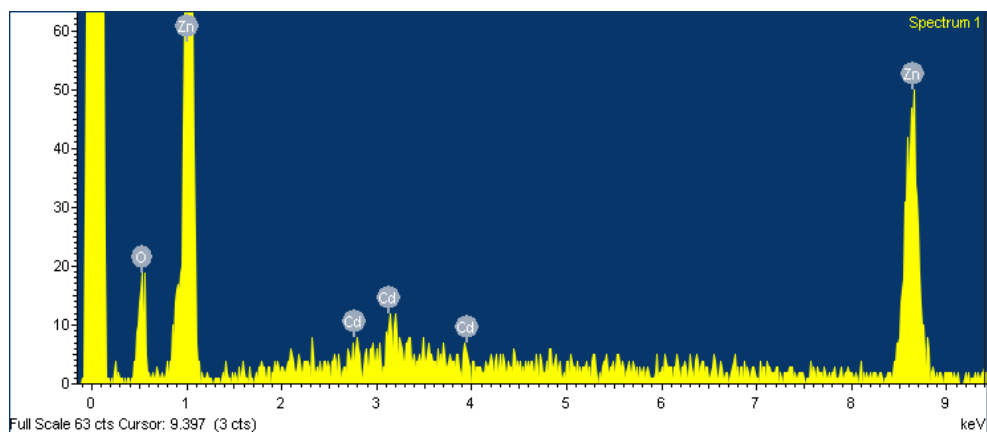


Fig.3.6. (e). SEM micrograph for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ at 100000x magnification.



Element	Weight%	Atomic%
O K	14.53	41.75
Zn L	79.15	55.66
Cd L	6.32	2.58
Totals	100.00	

Fig.3.6 (f). EDX analysis for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ sample.

3.4. Optical Analysis

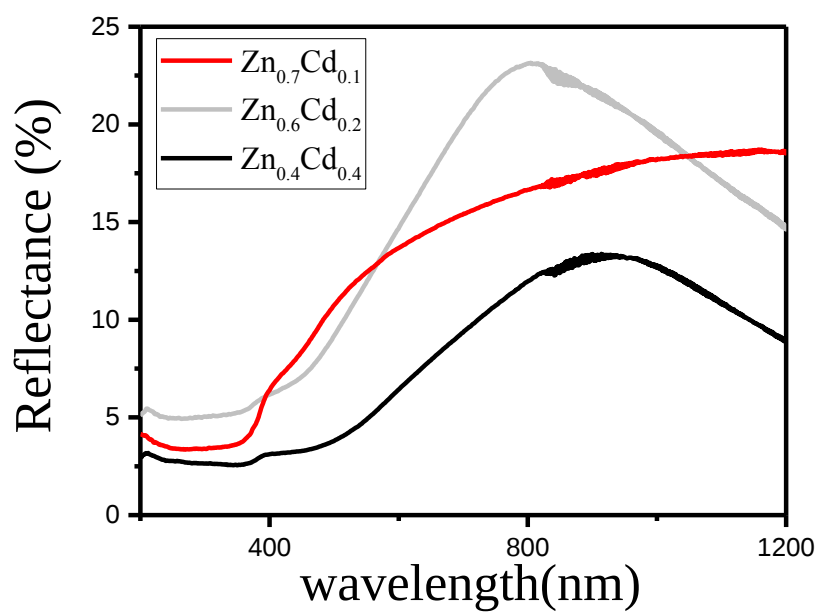


Fig.3.7. Reflectance spectra for ZnO-CdO composites with various composition.

The spectral distribution of reflectance $R(\lambda)$ at normal incident for all the samples is shown in Fig. 3.7. The light penetrates inside the sample and undergoes combination of scattering and absorption inside the sample. Some of the radiation is reflected back towards the surface. This reflected radiation contains useful information due to higher order of interaction. The reflected radiation is called Kubelka-Munk (KM) reflectance and is defined by a function. The KM function $F(R)$, can be used to approximate the optical absorbance of the sample from its reflectance and is given by [49].

$$F(R) = \frac{(1-R)^2}{2R} \quad (3.3)$$

So by replacing the absorption coefficient α in the Tauc's relation we get

$$(F(R)h\nu)^2 \propto (h\nu - E_g) \quad (3.4)$$

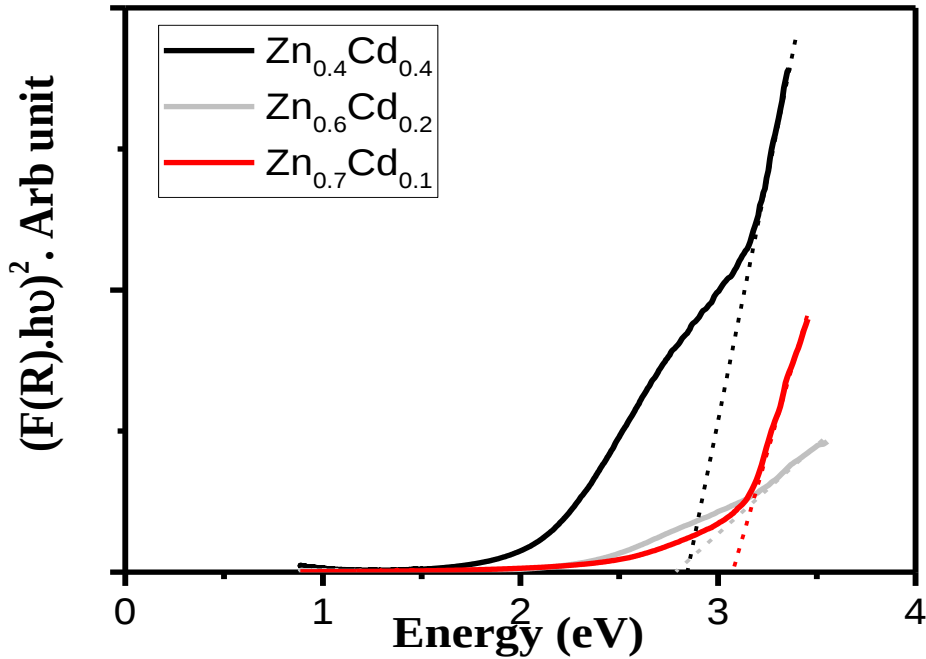


Fig 3.8. Bandgap for ZnO-CdO composites with various composition.

For the direct band gap, the plot between $(F(R).h\nu)^2$ and photon energy ($h\nu$) has been shown in Fig.3.8. The band gap value can be determined by extrapolating the graph of the linear region of the plots to energy axis at $(F(R).h\nu)^2 = 0$. The band gap energy of sample $Zn_{0.4}Cd_{0.4}$ is found to be 2.84 eV, then as the volume of Zn precursor solution

increases a small decrease in bangap value is observed, for sample $\text{Zn}_{0.6}\text{Cd}_{0.2}$ bandgap energy is found to be 2.78 eV. The bandgap value for $\text{Zn}_{0.7}\text{Cd}_{0.1}$ increases to 3.06 eV with further increase of volume concentration of Zn. Blue shift has been observed on increase in Zn precursor solution volume. The bandgap value for pure ZnO and CdO are 3.3 ev and 2.5 ev [50,51]. The increase in Zn content causes the lower states in conduction band to be filled and hence leading the blue shift in bandgap energies. The similar trends in bandgap energies has been reported by Jule at al and R. Saravanan et al [44,52].

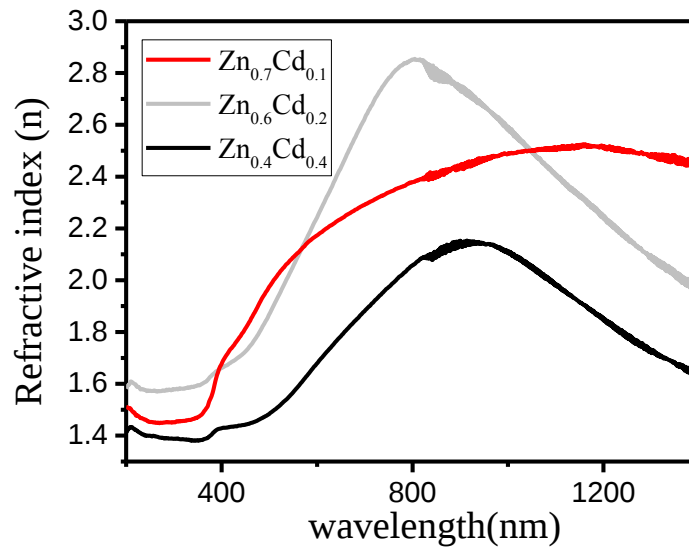


Fig 3.9. Refractive index for ZnO-CdO composites with various composition.

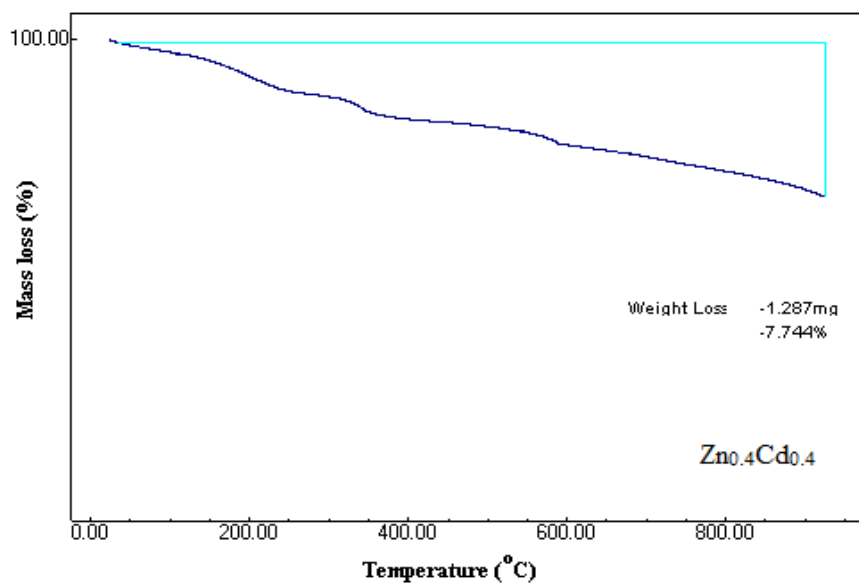
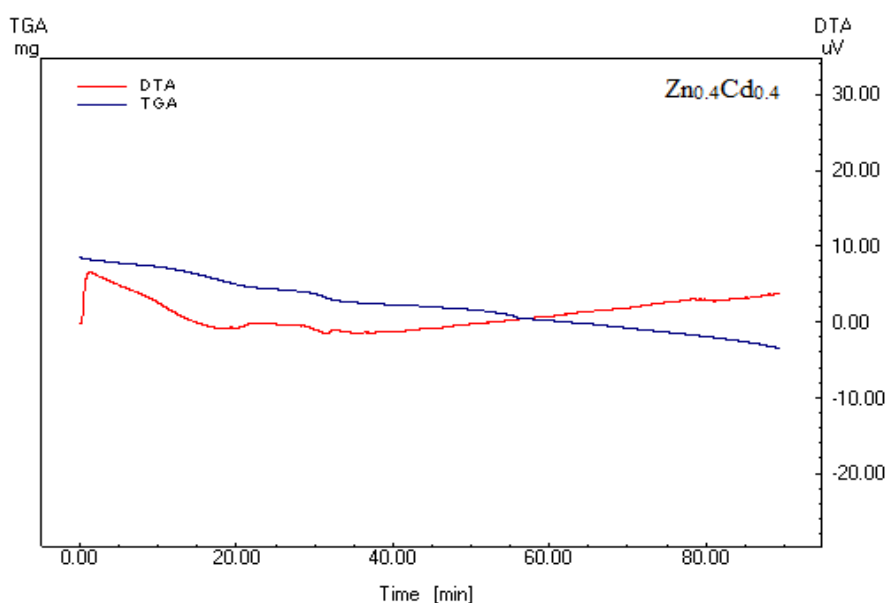
The study of dispersion is crucial for the application of any material in the field of integrated optical devices and device design for optical communication and spectral dispersion. The refractive index of the film was determined by the following relation [53].

$$n = \left[\frac{1+R}{1-R} \right] + \sqrt{\frac{4R}{(1-R)^2} - K^2} \quad (3.5)$$

Where $K = \alpha\lambda/4\pi$ is the extinction coefficient. The varition in refractive index have been shown in Figure 3.9.

3.5. Thermal Analysis

Thermal measurements such as TG/DTA were made to determine the weight loss of the samples, quality of the product, calcination temperature and phase transitions from room temperature to high temperature. The weight loss of sample $\text{Zn}_{0.4}\text{Cd}_{0.4}$ is 7.74% , for sample $\text{Zn}_{0.6}\text{Cd}_{0.2}$ is 4.71% and for sample $\text{Zn}_{0.7}\text{Cd}_{0.1}$ is 2.57%. For all samples Fig. 3.10 (a-c) we observe a small endothermic peak in DTA measurements this peak belongs to residues of the samples.



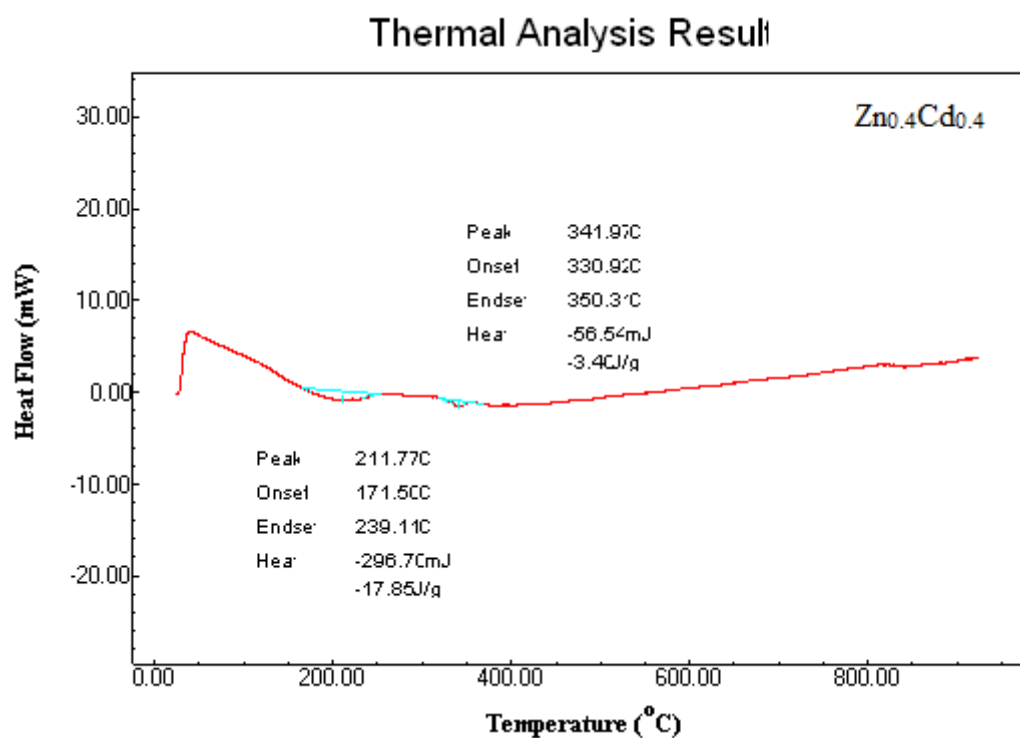
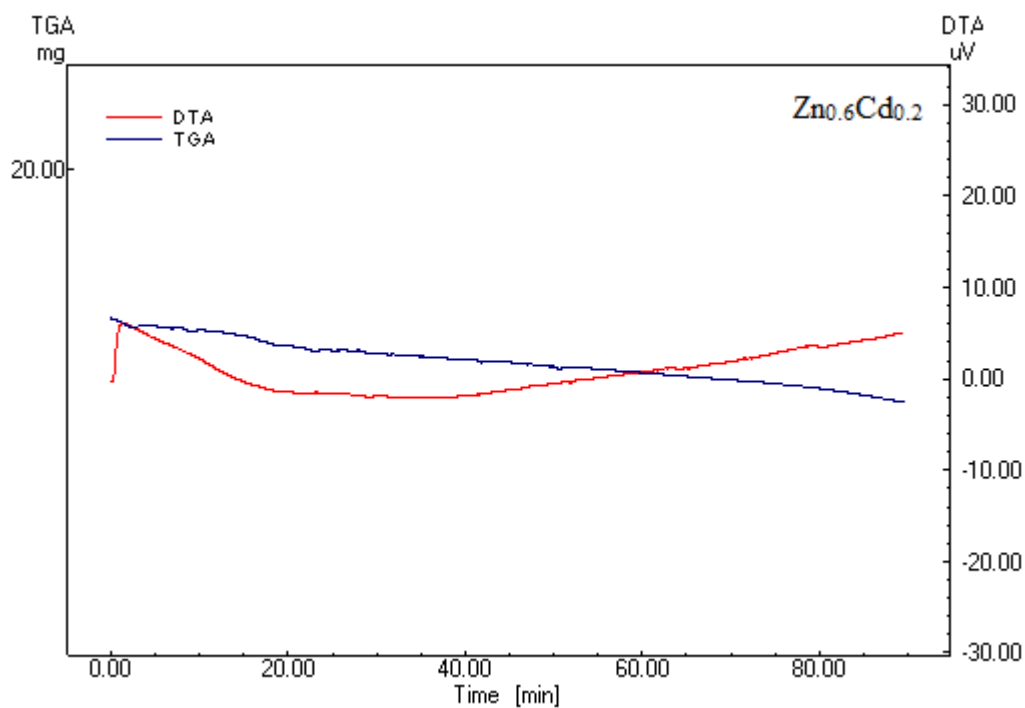


Fig.3.10 (a).TG-TDA analysis for Zn_{0.4}Cd_{0.4} sample.



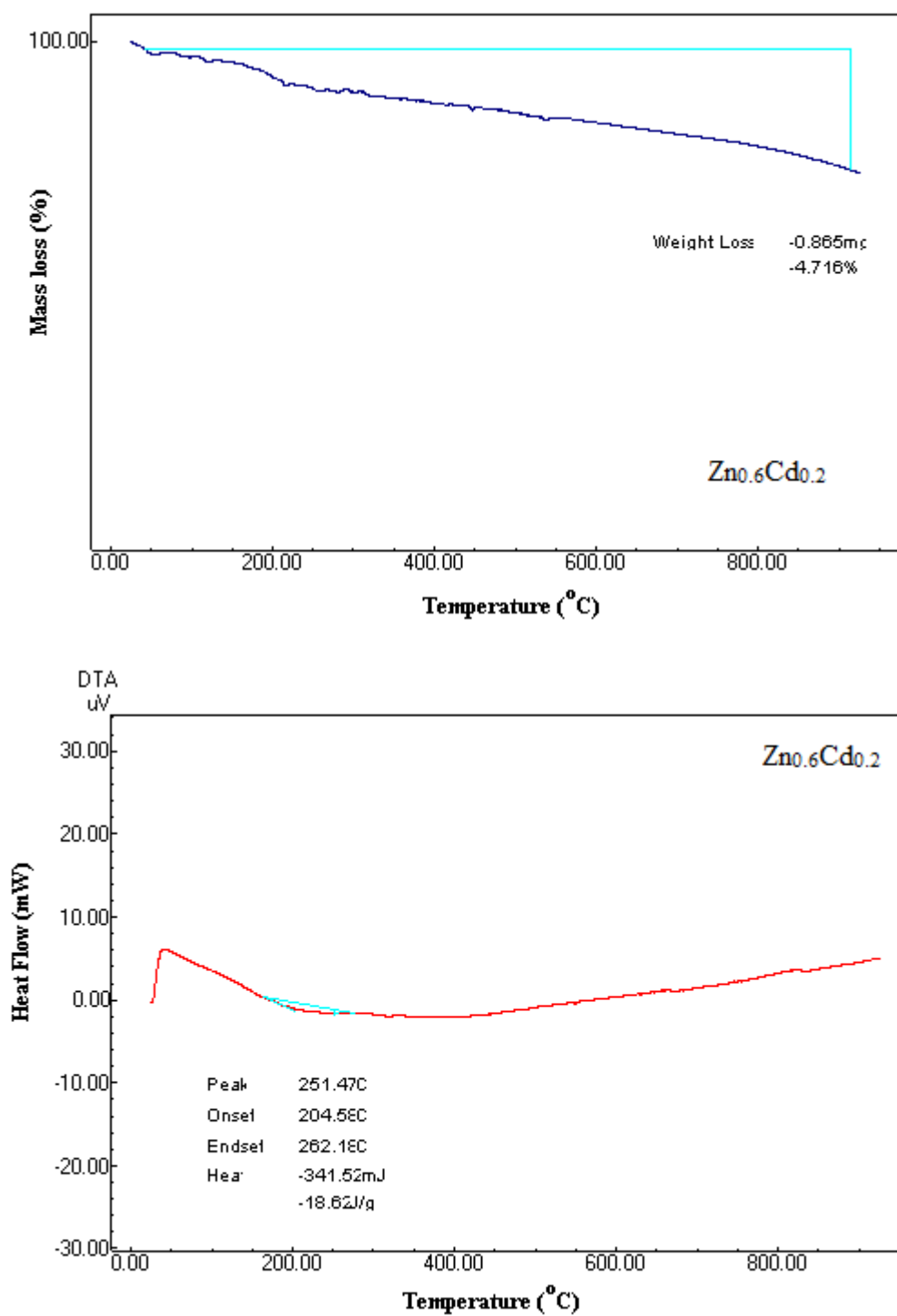
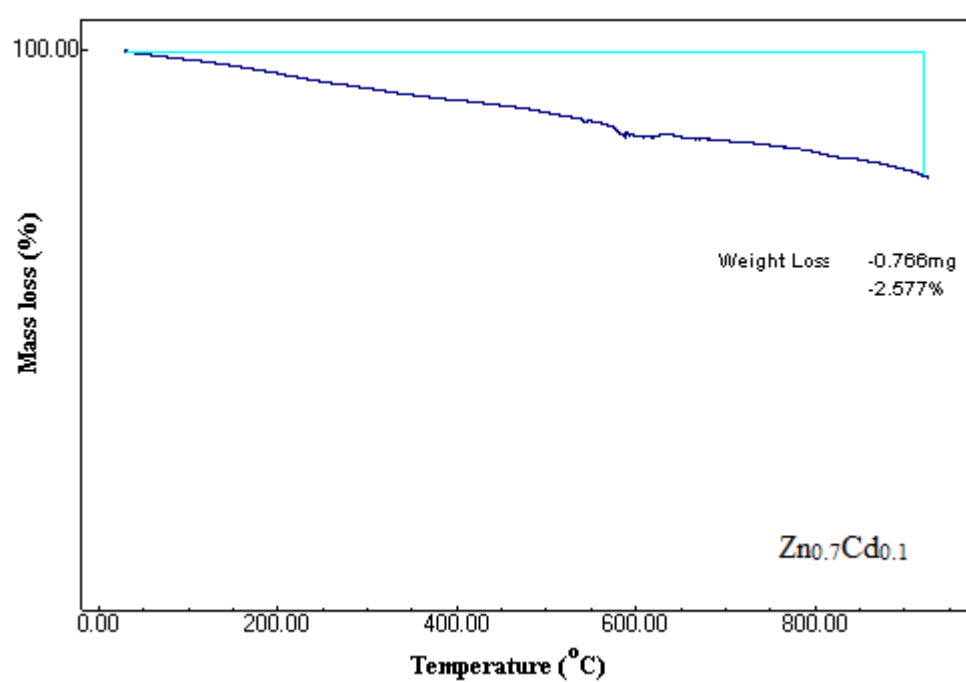
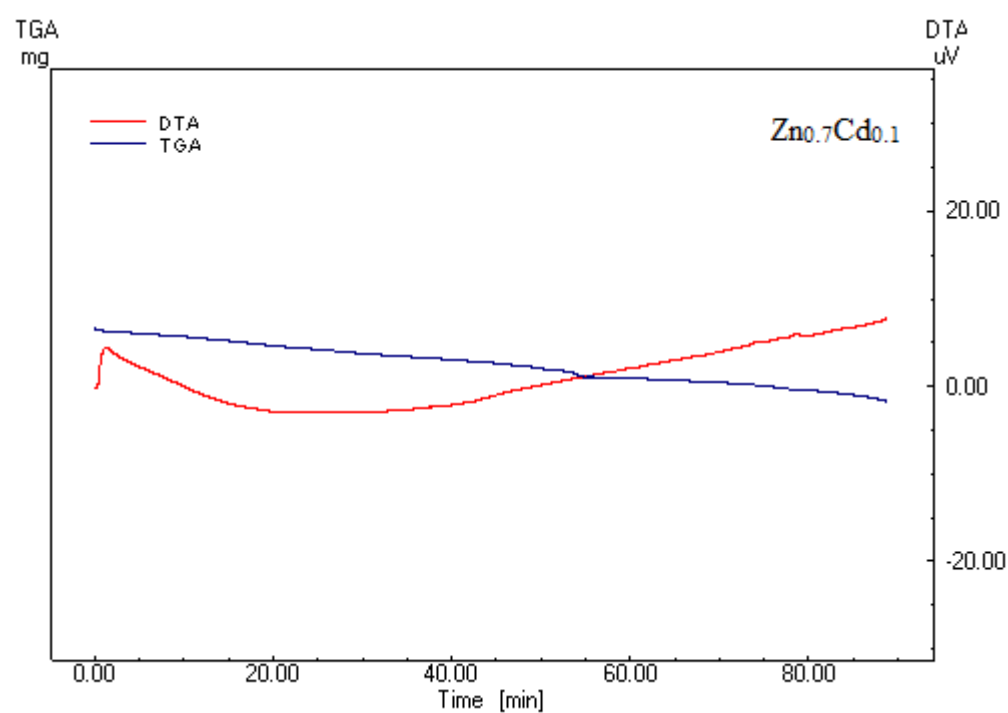


Fig.3.10 (b).TG-TDA analysis for $\text{Zn}_{0.6}\text{Cd}_{0.2}$ sample.



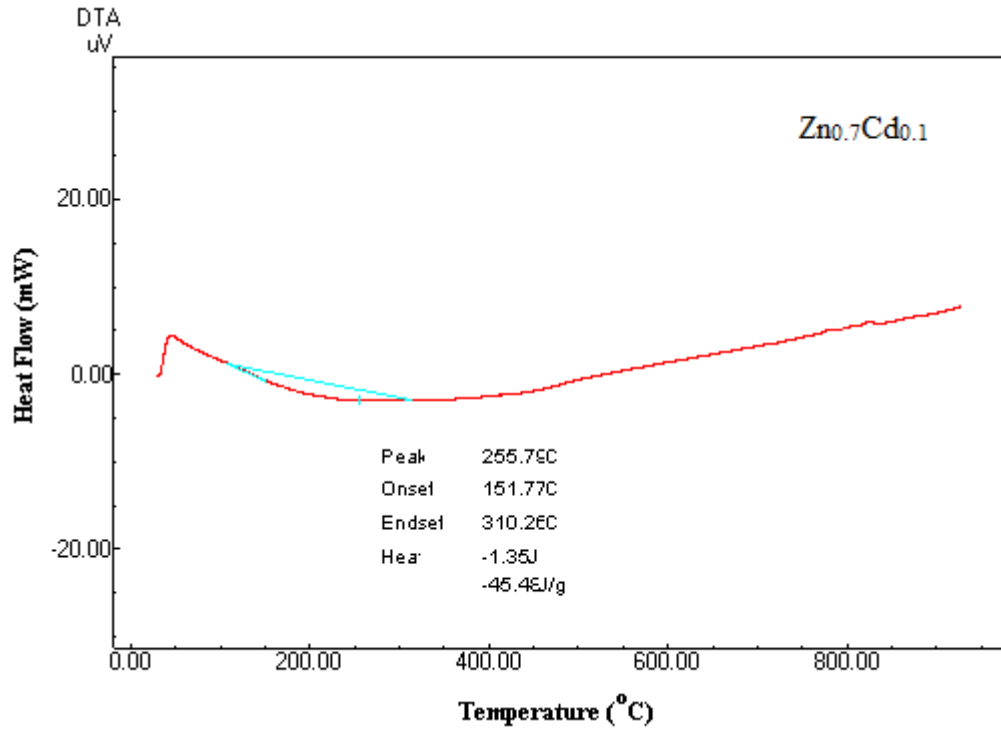
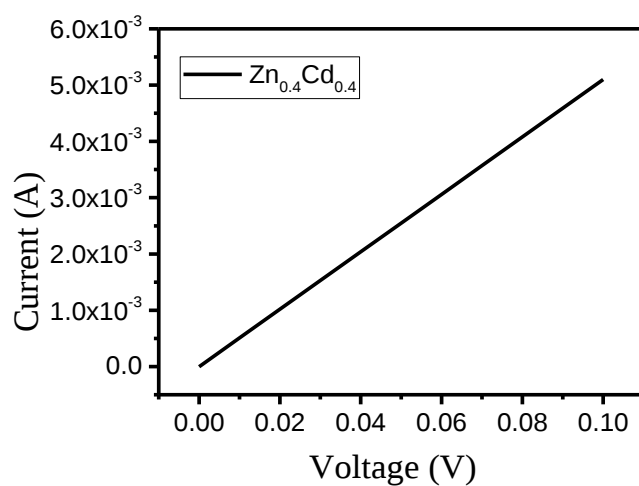


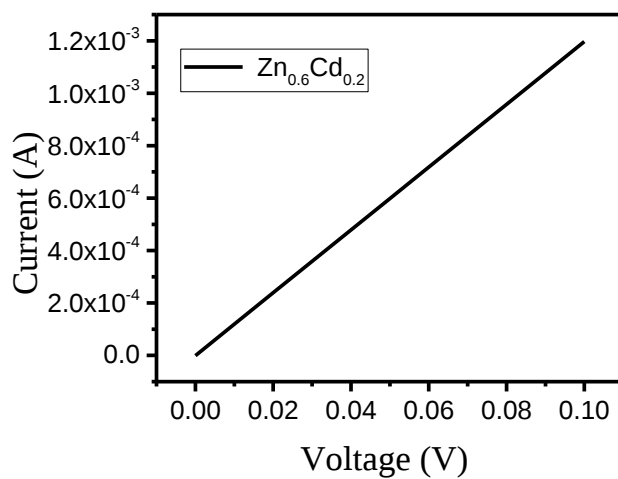
Fig.3.10 (c).TG-TDA analysis for Zn_{0.7}Cd_{0.1} sample.

3.6. Electrical Conductivity

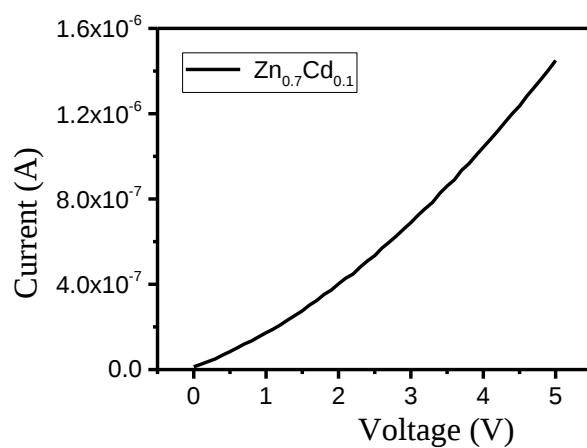
In order to investigate the electrical properties of the samples the dc conductivity of the samples was measured using two probe method. The I-V graph for the three samples has been shown in Fig. 3.11.(a-c). The electrical conductivity of the samples was found 4.6×10^{-3} , 1.3×10^{-3} and 4.4×10^{-8} S/cm, respectively for the three samples. We can clearly see from the graphs below that the conductivity is decreasing with increase in Zn content. K. Ocakoglu et. al. [54] has reported the electrical conductivity of ZnO nanorods at room temperature is 6.7×10^{-8} S/cm. The reported value is very close to the sample Zn_{0.7}Cd_{0.1} conductivity value. In the ZnO-CdO composite, the ZnO have hexagonal wurtzite and CdO have cubic crystal structure that causes the phase segregation. Thus, this phase segregation, crystal strain, grain boundary barrier effects may enhance the electron scattering and cause the deterioration in conductivity.



-a-



-b-



-c-

Fig. 3.11. DC Conductivity of ZnO-CdO compsites for (a) $\text{Zn}_{0.4}\text{Cd}_{0.4}$ (b) $\text{Zn}_{0.6}\text{Cd}_{0.2}$ (c) $\text{Zn}_{0.7}\text{Cd}_{0.1}$.

4. CONCLUSION

The ZnO-CdO composite was synthesized by using the hydrothermal technique. The structural analysis was done using the X-ray diffraction and particle size was calculated by scherrer formula. The morphological properties were investigated by using FESEM and we can see some spherical and non spherical particles. The bandgap values of the three samples was 2.84, 2.78 and 3.06 eV respectively which are in agreement with previously reported values. The thermal analysis graphs shows the confirms the formation of stable composites. The dc conductivity of the pellets was measured and value of conductivity decreases with increase in Zn volume concentration. Finally, the dielectric constant behavior was studied with change in frequency.

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