

To Explore the Antimicrobial Properties of Co-doped CdO Nanoparticles prepared by sol-gel Citrate Techniques

S.R. Gadpale¹, Dr. U. S. Khandekar²

¹Department of Industrial Chemistry Smt. Narsamma Arts Commerce and Science College, Kiran Nagar, Amravati

²Prof. Department of Industrial Chemistry Smt. Narsamma Arts Commerce and Science College, Kiran Nagar, Amravati

Abstract

This study focuses on the synthesis of Cadmium oxide (CdO) nanoparticles with doped transition metals like Cobalt (Co) of varying concentrations using sol gel citrate synthesis. Chemical and elemental characterization was carried out with X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet-Visible (UV-Vis) spectroscopy, Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray Spectroscopy (EDS). The efficacy of antimicrobial properties of the synthesized nanoparticles against a wide range of Gram-positive and Gram-negative bacterial strains and fungal strains was explored. The results demonstrate that the doping of transition metals enhances the antibacterial and antifungal properties of the CdO nanoparticles and thus it can be used as an antimicrobial agent in various industrial and medical fields due to its potential strength.

Keywords: Cadmium Oxide nanoparticles, Cobalt, EDS, SEM, , Antibacterial, Antifungal.

INTRODUCTION

With nanometer-sized materials as essential components that reveal distinctive structural, magnetic, chemical, and electronic properties because of their large surface to volume ratio and incredibly small dimensions in comparison to bulk materials, nanotechnology has emerged as an exciting field in recent years. The creation of nanomaterials for a range of uses, such as catalysts, energy storage devices, biomedicine, and environmental science, is a heavily financed field. Because of their numerous uses as catalysts, energy storage materials, and antibacterial agents, transition metal oxide (TMO) nanostructures have become particularly appealing in this respect¹⁻³. Transition metal has different structures, oxidation states, and high surface to volume ratio with high stability.

Among the different metal oxide semiconductors, n-type CdO nanostructures with 2.5 eV direct and 1.98 eV indirect bandgap have garnered a lot of interest because of their use in energy storage, advanced technology, and environmental applications like photocatalysis, transparent electrodes, antibacterial agents, and super capacitor and batteries electrodes⁴. Although several research teams have created CdO nanostructures in a variety of shapes, such as platelets, particles, rings, and wires, designing Cd-based nanostructures for energy storage and environmental applications remains extremely difficult⁵. A variety

of techniques, including structural design, mono-doping, energy band gap engineering, co-doping, coupling with functional material, and others, have also been used to improve the properties of CdO⁶. In the present work, pure and Co doped CdO nanoparticles of different concentration were prepared by using sol gel citrate method to study the antibacterial and antifungal properties along with morphological, elemental and structural analysis.

EXPERIMENTAL DETAILS

The nanocrystalline Co doped cadmium oxide nanoparticles (Co_xCd_{1-x}O) where (x= 0.2, 0.4,0.6) were prepared by using the sol-gel citrate method. For the preparation of precursor, a stoichiometric mixture of cobalt nitrate hexahydrate (Co(NO₃)₂·6H₂O) and cadmium nitrate hexahydrate (Cd(NO₃)₂· 6H₂O) in addition with citric acid and ethylene glycol was properly mixed. This mixture was then stirred on a magnetic stirrer continuously for 3 hours at 80°C to obtain a homogenous and transparent solution. The solution was further heated in a pressure vessel at 130⁰ C for about 12 hours to form gel like solution. Then the gel is subjected to heat treatment at 350⁰C in a muffle furnace to get the dried fine powder. The dried powder was then calcinated further in the range of 450⁰C to 650⁰C in order to improve the crystallinity of the synthesized Co doped CdO nanoparticles. Different concentration of Co doped CdO nanoparticles were synthesized to study the difference in the chemical, physical and elemental properties. The concentration taken for this synthesis was, Co_{0.2}Cd_(1-0.2)O, Co_{0.4}Cd_(1-0.4)O, and Co_{0.6}Cd_(1-0.6)O.

The synthesized nanoparticles was characterized using XRD which reveals that the nanoparticles calcinated at 650⁰C exhibits better crystallinity and thus for further studies these samples were used. FTIR, UV, SEM and EDS were examined and antimicrobial studies were carried out to study the biomedical applications.

RESULT AND DISCUSSION

X-ray Diffraction

CdO and Co doped nanomaterials of different concentration obtained from the sol gel synthesis were characterized through X-ray diffraction to get information about crystal structure, crystalline size and purity of sample. XRD pattern of synthesized cadmium oxide and doped cadmium oxide were obtained at room temperature. The scanning angle 2θ was varied from 20-80 degree. X-ray diffraction data were recorded by using Cu Kα radiation (1.5406 Å⁰). The crystallite size of prepared pure CdO and transition metal doped CdO nanostructured was estimated using the full width at half maximum (FWHM) of the peaks by means of the Scherrer formula,

$$d = \frac{k \lambda}{\beta \cos \theta}$$

Where,

d is the average crystallite grain size

β is the full width half maximum(FWHM) in radian

θ is the Bragg angle

λ is the wavelength of X-rays which is 0.15406 nm for Cu target Kα radiation

k is shape factor⁷

XRD spectrum of 0.2%, 0.4% and 0.6% Co-doped CdO nanoparticles synthesized by sol-gel method and calcinated at 650⁰C is shown in figure 1 (a, b, c and d) respectively. It exhibits cubic structure when

different concentration of Co^{+2} ions is added in Cd^{+2} ions. The different diffraction peaks occurs at $2\theta = 32.98, 38.25, 55.29, 65.21$ and 69.26 . The corresponding reflection peaks of (hkl) were seen at (111), (200), (220), (311) and (222) which are consistent with the reference pattern of JCPDS card No.9006684, 1011003 and 1541392 for 0.2%, 0.4% and 0.6% Co-doped CdO nanoparticles respectively, and it reveals that Co^{+2} substituted Cd^{+2} ions without forming any Co, CoO or Co_3O_4 phase. The average particle size of Co doped CdO nano particles was calculated using Scherrer's formula and it was found to be nearly 37.70 nm for 0.2 % , 36.97nm for 0.4% and 31.47 nm for 0.6 % Co doped CdO nanoparticles. The crystalline nature of the nanoparticle was confirmed because there was a sharpness in the XRD peaks at (111) crystallographic plane of Co doped CdO nanoparticles. There is an increase in FWHM with doping due to the uniform strain in nanoparticles when the Co^{+2} is doped in it. The crystalline size of the nanoparticles decreases with increase in concentration of doping percent due to change in lattice parameters and reduction in the growth of the crystal⁸⁻⁹.

Table 1 Sizes of particles in CdO and Ni-doped CdO

Samples	Particle Size (nm)	Lattice Parameter (Å)
CdO Nanoparticles	37.70	4.6990
CdO: Co (0.2%)	37.70	4.6953
CdO: Co (0.4%)	36.97	4.6964
CdO: Co (0.6%)	31.47	4.6944

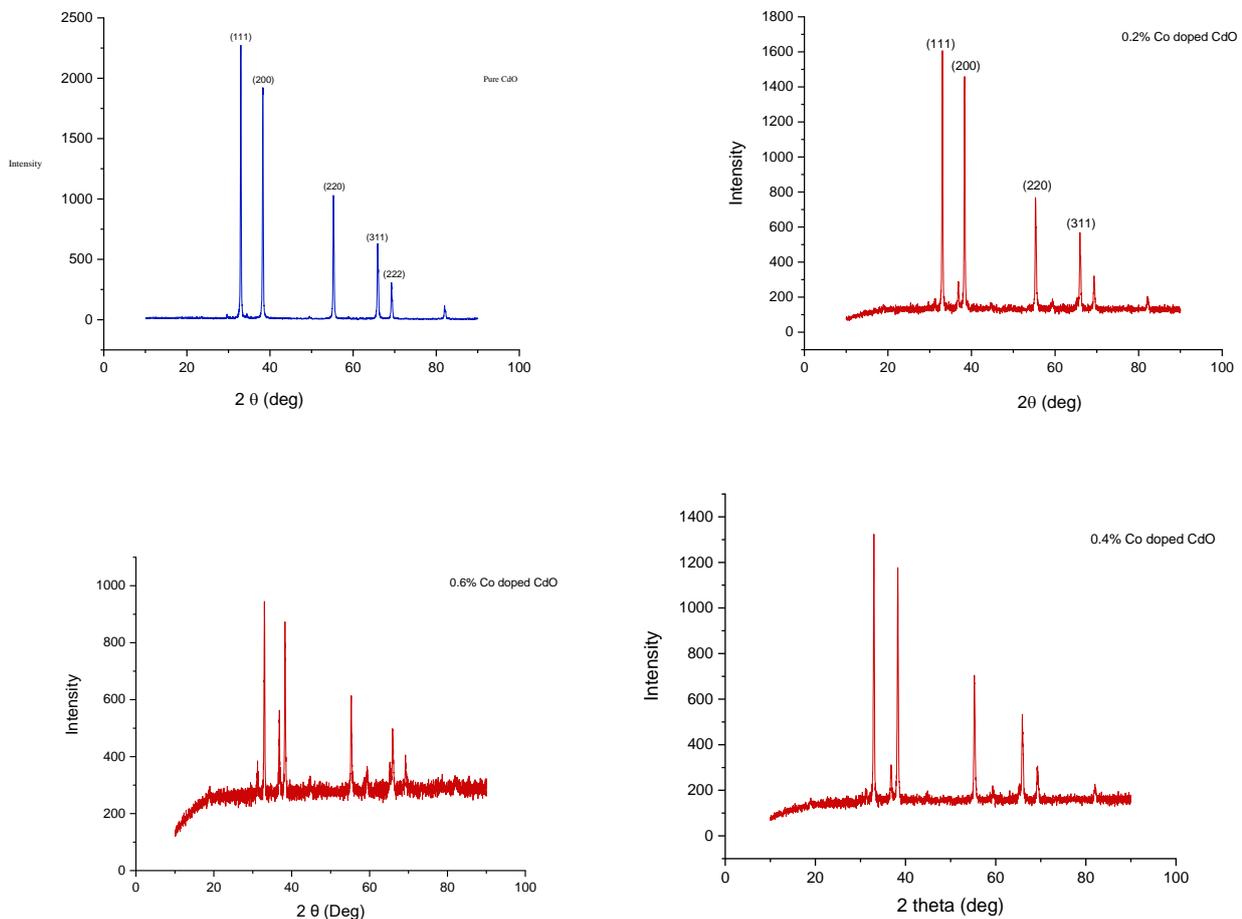


Figure 1 XRD Spectrum of pure and 0.2, 0.4 and 0.6 % Co Doped CdO nanoparticles

Fourier transmission infrared spectroscopy (FTIR) spectroscopy

Figure 2 shows the FTIR spectrum of pure and 0.6 % Co doped CdO nanoparticles. The formation of different organic functional groups and quality of materials was confirmed from the FTIR spectrum. The stretching vibrations due to Cd-O bond occurred shows the lower peak at around 578.34 cm^{-1} and 667.37 cm^{-1} . The stretching and bending vibrations of the water molecules in the sample shows the absorption peak at 3674.39 cm^{-1} . The O-H bending peaks occurs at around 3566.38 cm^{-1} . The peak at 2412.95 cm^{-1} – 2357.01 cm^{-1} due is to atmospheric vibration. Strong bond formation takes place when the Co is added to the cadmium lattice and thus, the stretching band shows high wave number in the FTIR spectrum¹⁰.

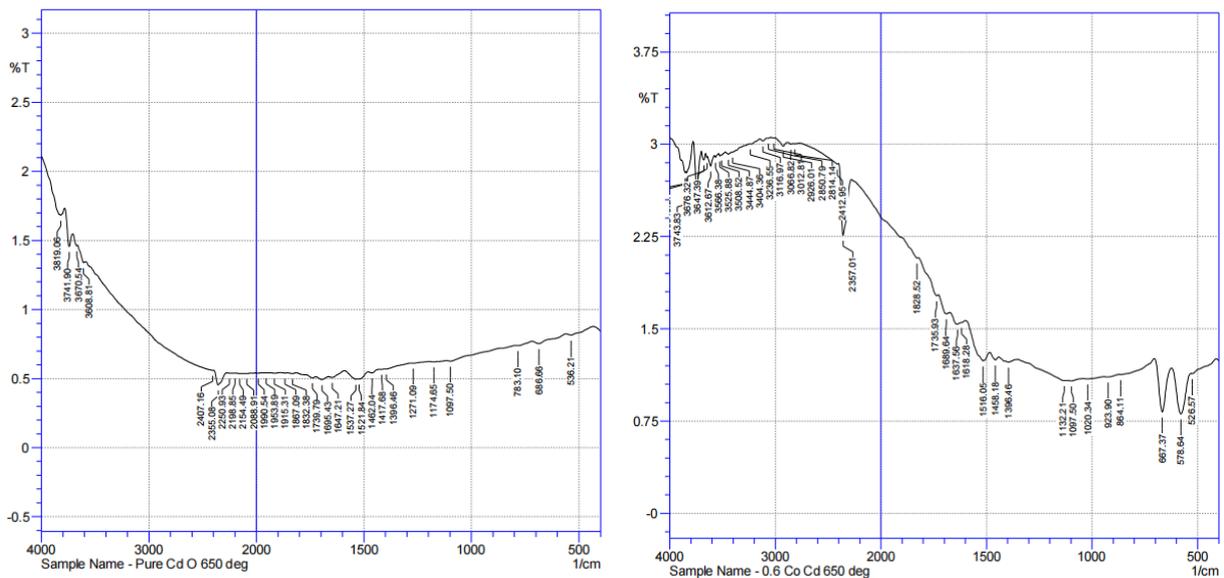


Figure 2 FIR Spectrum of Pure and 0.6% Co Doped CdO Nanoparticles

UV- visible spectroscopy

Figure 3 shows the absorption spectra of pure and 0.6% Co doped CdO nanoparticles. The synthesized nanoparticles exhibits absorption bands at around 200-400nm. The doped CdO nanoparticles because of quantum confinement show blue shift. The band gap decreases as the concentration of Mn as dopants increases and it is found to be 3.00 eV, 2.87 eV and 2.71 eV for 0.2%, 0.4% and 0.6% Mn-doped CDO nanoparticles respectively¹¹.

Data Set: Pure Cd O 650 deg - RawData

Data Set: 0.6 - Co Cd 650 deg - RawData

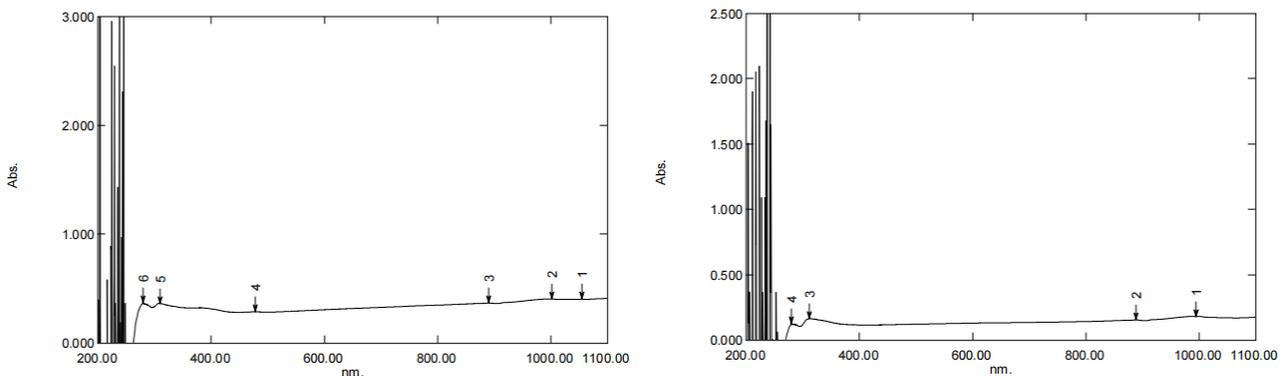
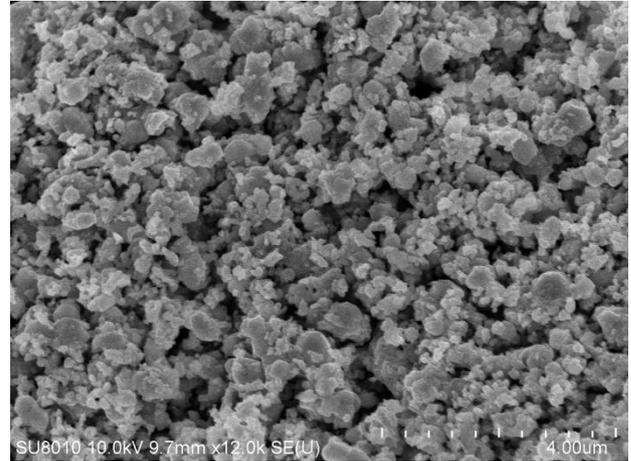
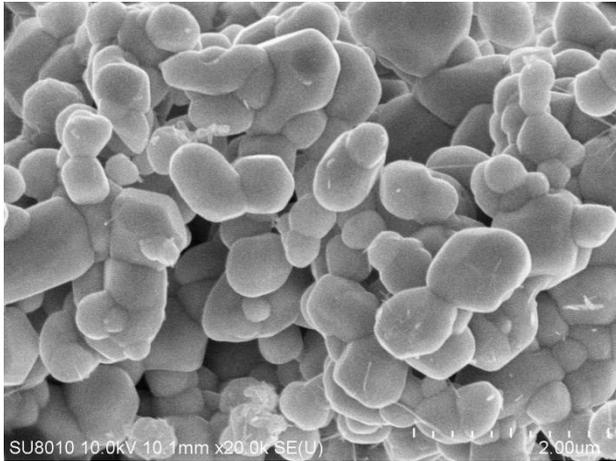


Figure 3 UV spectrum of Pure and 0.6% Co doped CdO nanoparticles

SEM with EDS Analysis

The surface morphology of the samples was studied using Scanning Electron Microscopy. The morphology of undoped CdO samples along with 0.6% Co doped CdO is shown in Figure 4 which gives an idea that there is an agglomeration of grains with spherical structure while 0.6% Co doped CdO nanoparticles due to slight agglomeration of nanoparticles also exhibits spherical morphology¹².



FESEM image of Pure CdO nanoparticles FESEM image of 0.6% Ni doped CdO nanoparticles

The EDS of Undoped CdO nanoparticles shows the presence of Cd and O element and Al as an impurity whereas 0.6% Co doped EDS shows the presence of Ni and no other impurities.

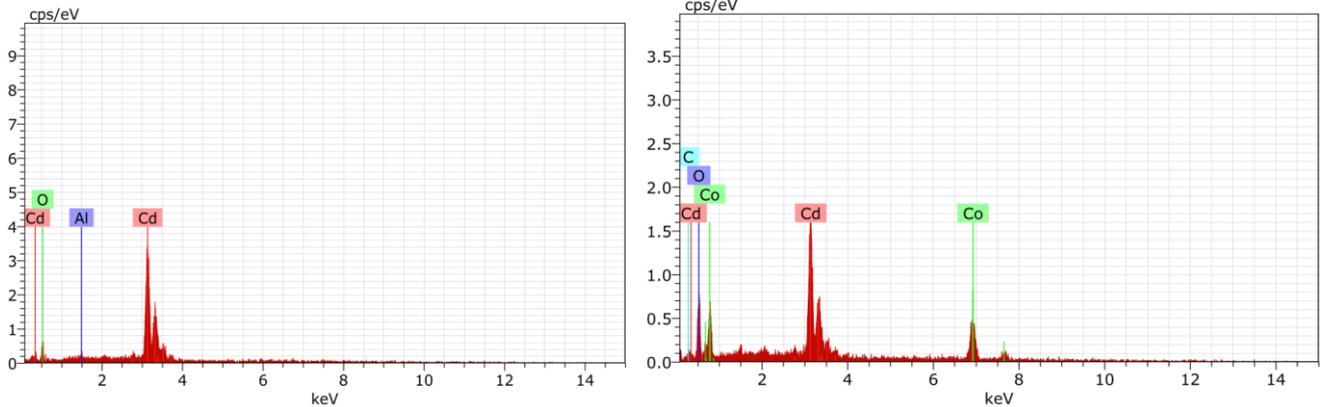


Figure EDS spectrum of Pure and 0.6% Co doped CdO nanoparticles

ANTIMICROBIAL APPLICATION

Nanoparticles are being researched extensively because of their potential uses in a variety of sectors, such as medicine and antibacterial chemotherapy. Certain bacteria are able to endure and grow on the surface of various objects. Microorganisms can be employed to produce consumer goods like bread, curd, and wine because of this ability, but many of them also cause food materials to degrade and are bad for cellular life because they can spread illness. Nanoparticles are being researched extensively because of their potential uses in a variety of sectors, such as medicine and antibacterial chemotherapy. Certain bacteria are able to endure and grow on the surface of various objects. Microorganisms can be employed to produce consumer goods like bread, curd, and wine because of this ability, but many of them also cause food materials to degrade and are bad for cellular life because they can spread illness.

Antibacterial Activity

Nanocrystalline undoped CdO and doped $\text{Co}_x\text{Cd}_{1-x}\text{O}$, (where $x = 0.2\%$, 0.4% and 0.6%) synthesized by sol gel method calcinated at 650°C were subjected to antibacterial analysis by using Agar well disc diffusion method against different gram positive and gram negative bacterial strains. In the disc diffusion test sterile disc (Himedia – SD067 different samples were impregnated at $20\mu\text{l/ml}$ for 24 hours at 37°C .

Test organisms used:

1. *E. coli* (MTCC 118)
2. *K. pneumoniae* (MTCC 109)
3. *S. aureus* (MTCC 1430)
4. *E. faecalis* (MTCC 2729)

Sensitivity Result

S.N.	Samples	Zone of Inhibition (mm)			
		<i>E. coli</i> (MTCC 118)	<i>K. pneumoniae</i> (MTCC 109)	<i>S. aureus</i> (MTCC 1430)	<i>E. faecalis</i> (MTCC 2729)
1	Sample A	14	14	15	14
5	Sample B	15	16	15	14
6	Sample C	14	13	15	13
7	Sample D	15	12	11	12



Figure 4.1 Zone Of inhibition for *E.coli* , *K. pneumonia*, *S. aureus*, *E. faecalis*

Antifungal Activity

Antifungal antibiotic control with DMSO was used during analysis. The compound was dissolved in DMSO

Test organisms (fungus) used:

1. *Candida albicans*
2. *Trichophyton rubrum*

S.N.	Sample	Antifungal Activity Zone of Inhibition (mm)	
		<i>Candida albicans</i>	<i>Trichophyton rubrum</i>
1	Sample A	18 mm	18 mm
5	Sample B	8 mm	15 mm
6	Sample C	15 mm	18 mm
7	Sample D	18 mm	20 mm



Figure 4.2 Zone of inhibition for *Candida albicans* and *Trichophyton rubrum*

REFERENCES

- O.M. Ikumapayi, Akinlabi E. T. , Adeoye A, Fatoba S.O., Micro fabrication and nanotechnology in manufacturing system- an overview, Materials Today Proceedings, 44, 2021, 1154-1162.
- Almeida L, Felzenszwalb I, Marques M, Cruz C, Nanotechnology activities: environmental protection regulatory issues data, Heliyon, 6, 2020, 053303.
- Padil V.V.T, Waclawek S, Cernik M, Varma R.S, Tree gum-based renewable materials: sustainable applications in nanotechnology, biomedical and environmental fields, Biotechnol Adv., 36, 2018, 1984-2016.
- Kumar P. S, Selvakumar M, Bhagabati P, Bharathi B, Karuthapandian S, Balakumar S, Cdo/ZnO Nanohybrids: facile synthesis and morphologically enhanced photocatalytic performance , RSC Adv., 4, 2014, 32977-32986.
- Khairy M, Ayoub H, A, Banks C, E, Large-scale production of CdO/Cd(OH)² nanocomposites for non-enzyme sensing and supercapacitor applications, RSC Adv., 8, 2018, 921-930.
- Sivakumar S, Venkatesan A, Soundhirarajan P, Khatiwada C, P, Synthesis, characterization and anti-bacterial activities of pure and Ag doped CdO nanoparticles by chemical precipitation method, Spectrochim. Acta - Part A Mol. Biomol. Spectroscopy, 136 ,2015, 1751–1759.
- Allahverdikhani T, Barvestani J, Meshginqaalam B, “The effect of different transition metal dopants on the magnetic properties of armchair antimonene, phosphorene, and their binary nanoribbons”, Journal of Magnetism and Magnetic Materials, 600, 2024, 1-24.
- Khayati G, Dalvand H, Darezereshki E, Irannejad A, “A facile method to synthesis of CdO nanoparticles from spent Ni–Cd Batteries”, Materials Letters, 115, 2014, 272-274.
- Subramanian M, Vijayalakshami S, venkataraj S, Jayavel R, “Effect of cobalt doping on the structure and optical properties of TiO₂ films prepared by sol-gel process”, Thin Solid Films, 516, 2008, 3776– 3782.

10. Tharmar T, George A, Kumar A, “Optical Properties and FTIR Studies of Cobalt Doped ZnO Nanoparticles by Simple Solution Method”, Indian Journal of Science and Technology, 9 (1), 2014, 1-4.
11. Vindhya P, Suresh S, Kunjikannan R, “Antimicrobial, antioxidant, cytotoxicity and photocatalytic performance of Co doped ZnO nanoparticles biosynthesized using *Annona Muricata* leaf extract”, Iranian Journal of Environmental Health Science & Engineering, 21(1), 2023, 167-185.
12. Akbari B, Tavandashti M, Zandrahimi M, Particle size characterization of nanoparticles –a practical approach, Iranian journal of materials science and engineering , 8(2), **2011**, 48- 56.