

## Structural and Antibacterial Activity of Copper Oxide Nano Particles

A.Pricilla Jeyakumari<sup>1</sup>, M.Ramakrishnan<sup>1</sup>, Y.Nithyanandhi<sup>1</sup>, S.Renuka<sup>2</sup>

<sup>1</sup>Thiruvalluvar Government Arts College, Rasipuram, Tamil Nadu, India

<sup>2</sup>Gnanamani Engineering College, Paachal, Namakkal, Tamil Nadu, India.

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**Abstract:** Nano particles of metal oxides are manufactured in the large scale because of their industrial applications. Copper Oxide is an important p-type semiconductor, has drawn increasing attention in the application of gas sensors because of low cost, excellent reactivity, high stability and non-toxicity, and also the secondary architectures composed of nanostructured building blocks have attracted significant interest in material synthesis and device fabrications. In the present investigation pure and Ni,Cd doped CuO nano particles have been prepared by co-precipitation method. The crystalline natures of the sample were also confirmed by X-ray diffraction pattern. The crystalline size of the pure and Cd,Ni doped nano particle were determined as 13 nm, 12 nm and 14 nm respectively. FT-IR spectra identify the functional group present in the molecular structure. There is a shift in the lower frequency region confirmed the presence of the dopants (Ni,Cd). The surface morphology of the prepared materials were also analysed by FE-SEM. It reveals the polycrystalline porous morphology with a nano flower structure. The small crystallites agglomerated to form nanoflowers and nano sheets. The energy gap value for pure and doped (Ni,Cd) CuO were also determined. Anti-bacterial studies shows that the zone of inhibition is high for the CuO and doped (Ni,Cd) CuO nano particle. CuO nanoparticle exhibited strong antibacterial activity against bacterial species.

**Keywords:** Antibacterial activity, Co-precipitation, Copper Oxide, FT-IR, Nanoflower, SEM, XRD.

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### I. Introduction

Nano materials have attracted much attention in recent years because of their outstanding properties and potential applications, exhibited neither by small molecular system nor by larger particulate matter[1]. Nano particles of metal oxides are already manufactured in large scale because of their industrial applications. Nanocopper oxide ( CuO ) is a well-known p – type narrow band gap semiconductor with lower band gap 1.2 eV ( at room temperature ) is mainly used in many application such as catalysis, photo-catalysis, humidity, gas sensors, magnetic storage media, solar energy transformation, electronics and super capacitor. Nanoparticles of copper oxide have potential to replace noble catalysts for carbon monoxide oxidation. Nano fluids of CuO nanoparticles have excellent thermal conductivity suitable for their applications as heat transfer fluids in machine tools.[2] Beit like CuO nanoparticles exhibit an enhanced sensing performance towards formaldehyde and Ethanol vapours with rapid response and high sensitivity. CuO has complex magnetic phases; forms the basis for several high T<sub>c</sub> superconductors and material with giant magneto resistance. Recently, CuO, as an important p-type semiconductor, has drawn increasing attention in the application of gas sensors because of low cost, excellent reactivity, high stability and nontoxicity, and also the secondary architectures composed of nanostructured building blocks have attracted significant interest in material synthesis and device fabrications.[3,4] This is because nanostructured semiconductors display wide applications due to the strong relationship between their geometry and their function. It is generally agreed that properties of CuO may be tuned or improved by developing materials with well-controlled characteristics such as high crystallinity, uniform size, shape and large surface area. Motivated by the application CuO nanoparticles were prepared by co-precipitation method. In the present work the prepared pure & Cd,Ni doped CuO nanomaterials were characterized by powder X-Ray diffraction method and FT-IR spectrum analysis.[5] The morphology of the nanomaterial were determined by scanning electron microscopy (FESEM). The Antibacterial activity of the pure and Ni doped CuO were also studied and presented.[6]

## II. Experimental

### 2.1 Synthesis of pure copper oxide and Cd, Ni doped copper oxide

0.05M of copper(II) chloride di-hydrate [ $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ] and 0.5mM of poly ethylene glycol were dissolved in 150 ml of distilled water and stirred for 15 mins [solution A]. Then 150ml of 0.6M of sodium hydroxide solution (solution B) was added drop wise into the above solution under constant stirring. Then the obtained blue precipitate of copper hydroxide was stirred and heated at  $50^\circ\text{C}$  for 1 hour to get black precipitate of CuO. The black precipitate thus formed was centrifuged and washed with distilled water, absolute ethanol and acetone and then kept in the muffle furnace at  $100^\circ\text{C}$  for one hour a black solid were obtained, and then it grained by milling process to obtained CuO nanoparticles. [3] 0.002 M cadmium sulphate were added with the solution A and the same procedure was followed to get Cd doped CuO nanoparticles. Similarly 0.002 M Nickel Chloride was added to the solution A to get Ni doped CuO nanoparticles.

## III. Characterization Techniques Structural Characterization

### 3.1. Powder X-Ray Diffraction Pattern

The x-ray diffraction analysis of pure CuO and Cd, Ni doped CuO nanoparticle were carried to identify the crystal structure as shown in fig (3.1,3.2,3.3). All diffraction peaks are index with the corresponding planes of CuO, Cd-CuO and Ni-CuO. The peaks 35.48, 53.1, 61.5, 66.04 which match well with the plane (-1 1 1), (-2 0 2), (0 2 0), (2 0 2), (0 2 2). and Ni-CuO peaks 35.3, 39.17, 49.7, 53.2, 62.2, 65.1, 67.5, 66.04 which match well with the plane (0 0 2), (1 1 1), (-2 0 2), (0 2 0), (-1 1 1), (-3 1 1), (2 2 0). All these planes of Cd, Ni doped CuO crystal structure are very close to the JCPDS9(05-0661) values of CuO. The X-ray diffraction well agree with the monoclinic phase of copper oxide. [7,8]

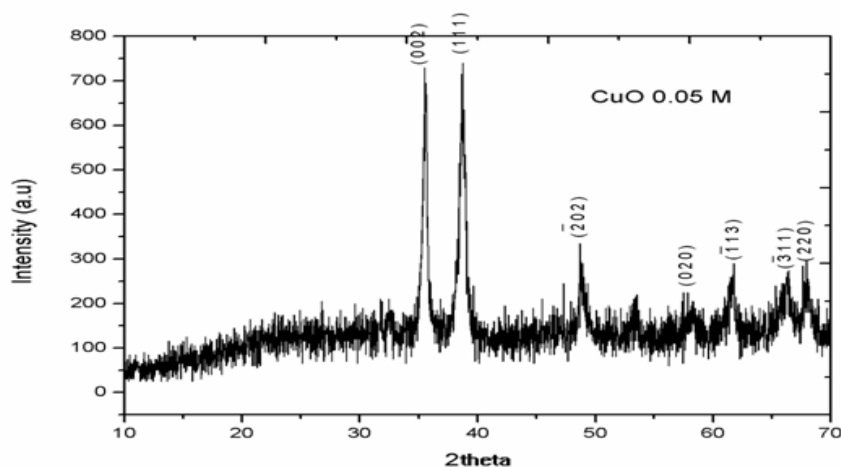


Fig 3.1 XRD Pattern of Copper Oxide

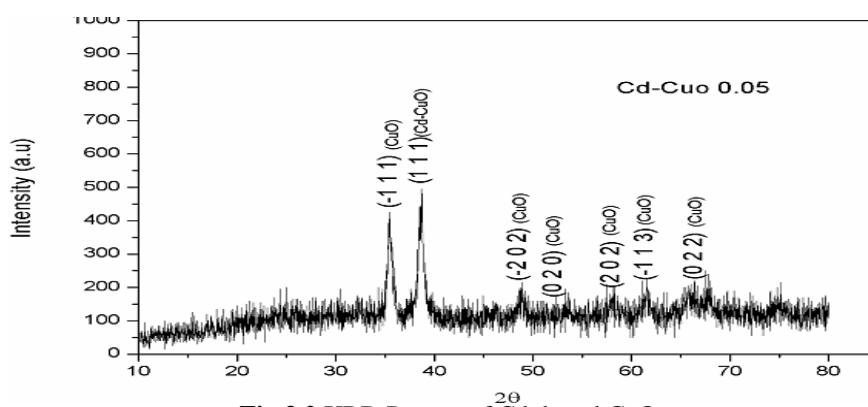


Fig 3.2 XRD Pattern of Cd doped CuO

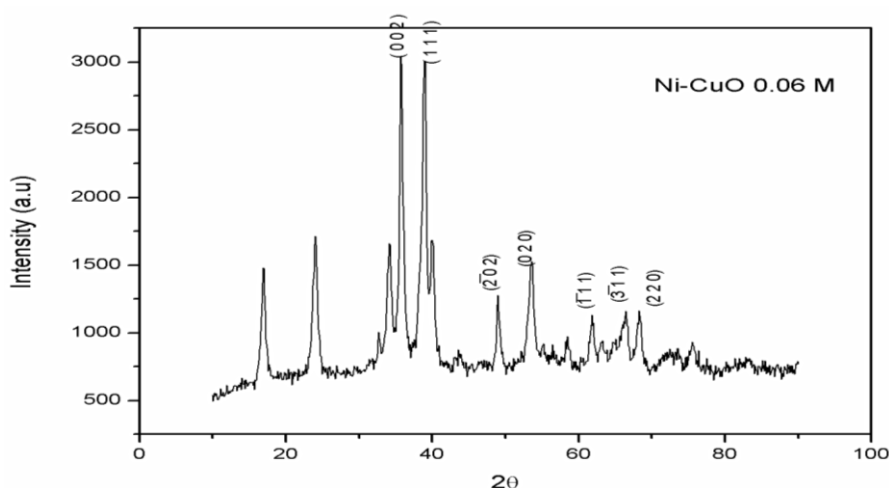


Fig 3.3 XRD Pattern of Ni doped Copper

No other diffraction peaks arising from metallic Cu or Cu<sub>2</sub>O present in the XRD pattern were observed. The crystal structure of the samples were examined by X-ray diffraction, with Cu K $\alpha$ ( $\lambda$ =1.54056) in 2 $\theta$  range. The crystallite sizes of the particles were calculated by using Sherrer's equation.

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

Where, D is the crystallite size of the particles, K is a shape factor (K=0.9 in this work),  $\lambda$  is the wavelength of the incident X-ray (1.5405 Å, CuK $\alpha$ ),  $\theta$  is the diffraction angle and  $\beta$  is the full width half maximum. The calculated particle size for pure CuO is 13 nm and for Cd-CuO is 12 and Ni-CuO is 14 nm. The addition of impurities like Cd, Ni causes a slight modification in the size of the nanoparticles.

### 3.2. FT-IR Spectroscopy

The FTIR spectrum of CuO shows the three strong peaks at 418.58 cm<sup>-1</sup>, 498.21 cm<sup>-1</sup>, and 611.31 cm<sup>-1</sup> and Cd, Ni doped CuO shows at 426.5 cm<sup>-1</sup>, 420.02 cm<sup>-1</sup> and 601.4 cm<sup>-1</sup> are associated with the Cu-O vibrations of mono clinic Copper oxide. The bands located at above 498.21 cm<sup>-1</sup> and 490.54 cm<sup>-1</sup> are due to the Cu-O stretching along the direction for both Cd, Ni-CuO similarly that at about 611.31 cm<sup>-1</sup>, 615.84 cm<sup>-1</sup> is associated with Cu-O stretching. The peaks for Cd, Ni-CuO centred at 418.58 cm<sup>-1</sup>, 420.02 cm<sup>-1</sup> and 498.21 cm<sup>-1</sup>, 490.54 cm<sup>-1</sup> show blue shift and the peak 611.31 cm<sup>-1</sup>, 615.84 cm<sup>-1</sup> show red shift compared with the reported values (429, 502 and 591 cm<sup>-1</sup> respectively). The broad peaks at about 3401.85 cm<sup>-1</sup>, 3574.69 cm<sup>-1</sup> and 3392.4 cm<sup>-1</sup>, 3329.89 cm<sup>-1</sup> are related to the O-H stretching of hydroxyl group absorbed on the surface of the sample, which is further confirmed by the band at about 1629.9 cm<sup>-1</sup>, 1709.01 cm<sup>-1</sup>. The O-H peak at 3401.85 cm<sup>-1</sup>, 3329.89 cm<sup>-1</sup> associated with defects and free carrier concentration [8,9]. The vibrational frequency of pure CuO at 865 cm<sup>-1</sup> is shifted to 993.85 cm<sup>-1</sup> in Ni doped CuO confirms the presence of Ni-O stretching. The corresponding graphs are shown in fig [3.4, 3.5, 3.6].

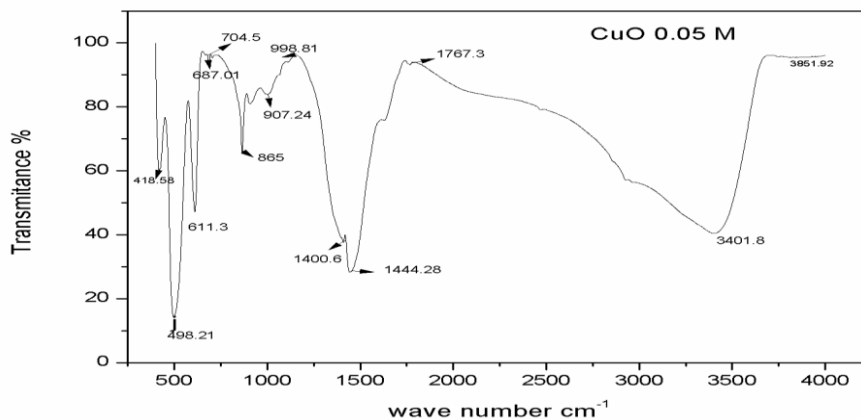


Fig 3.4 FT-IR spectrum of pure copper oxide.

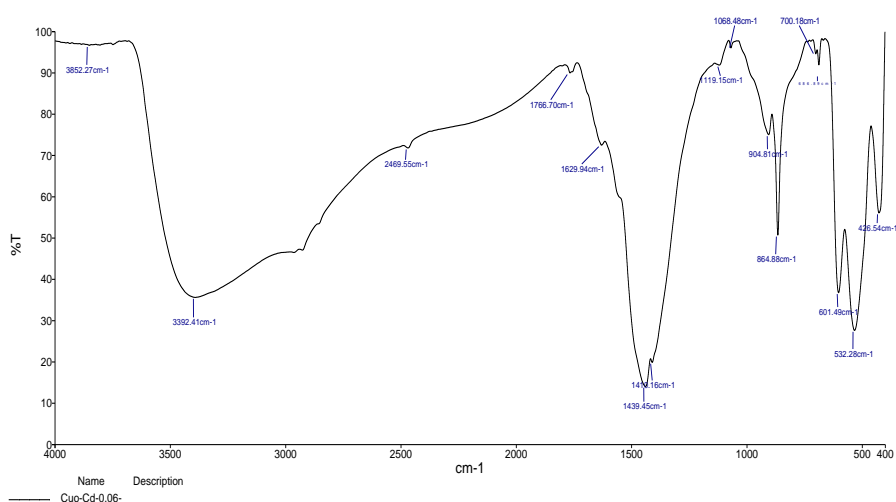


Fig 3.5 FT-IR spectrum of Cd doped Copper Oxide.

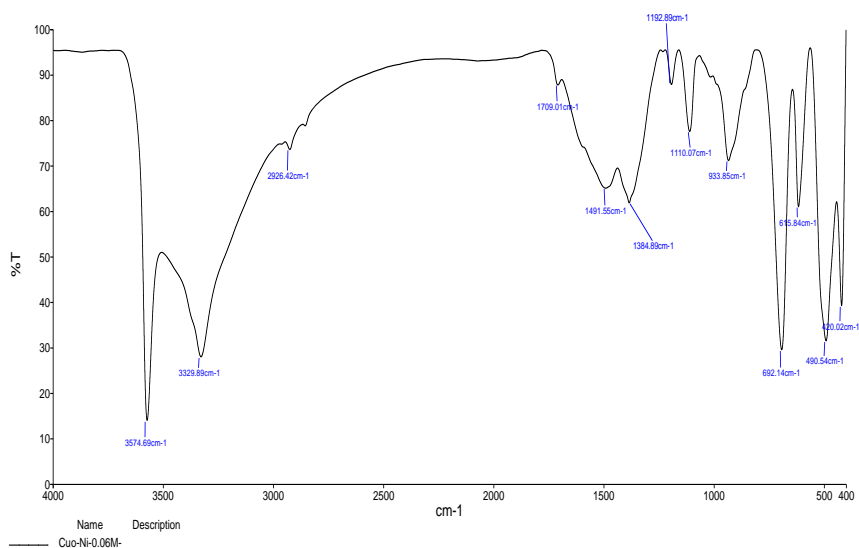
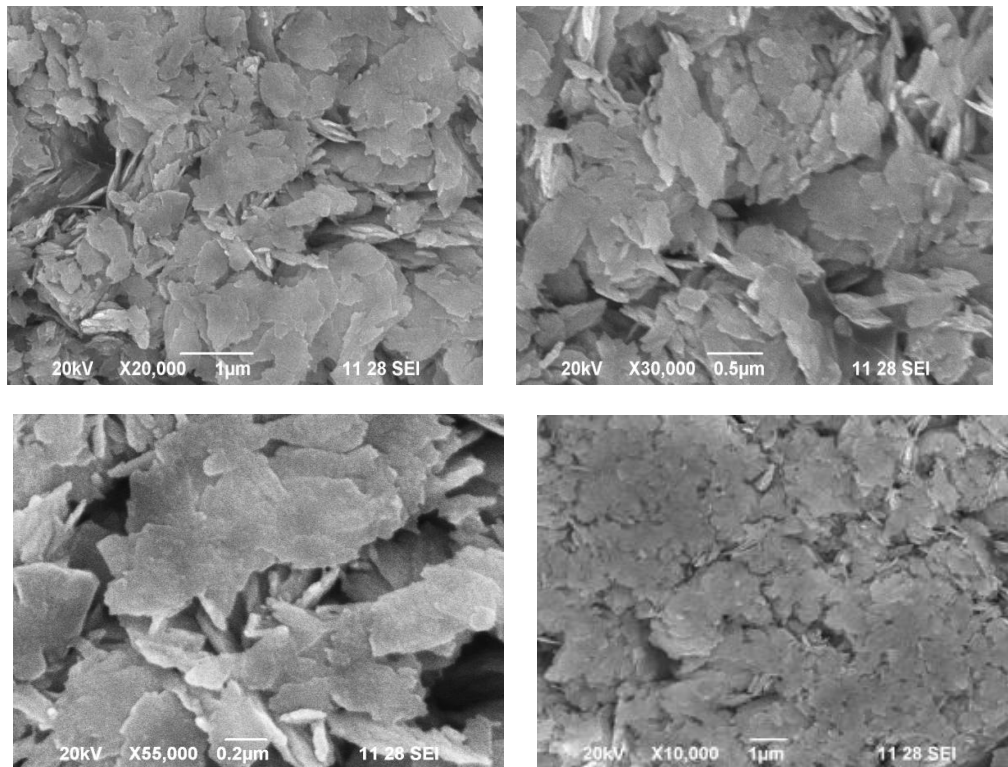


Fig 3.6 FT-IR spectrum of Ni doped Copper Oxide.

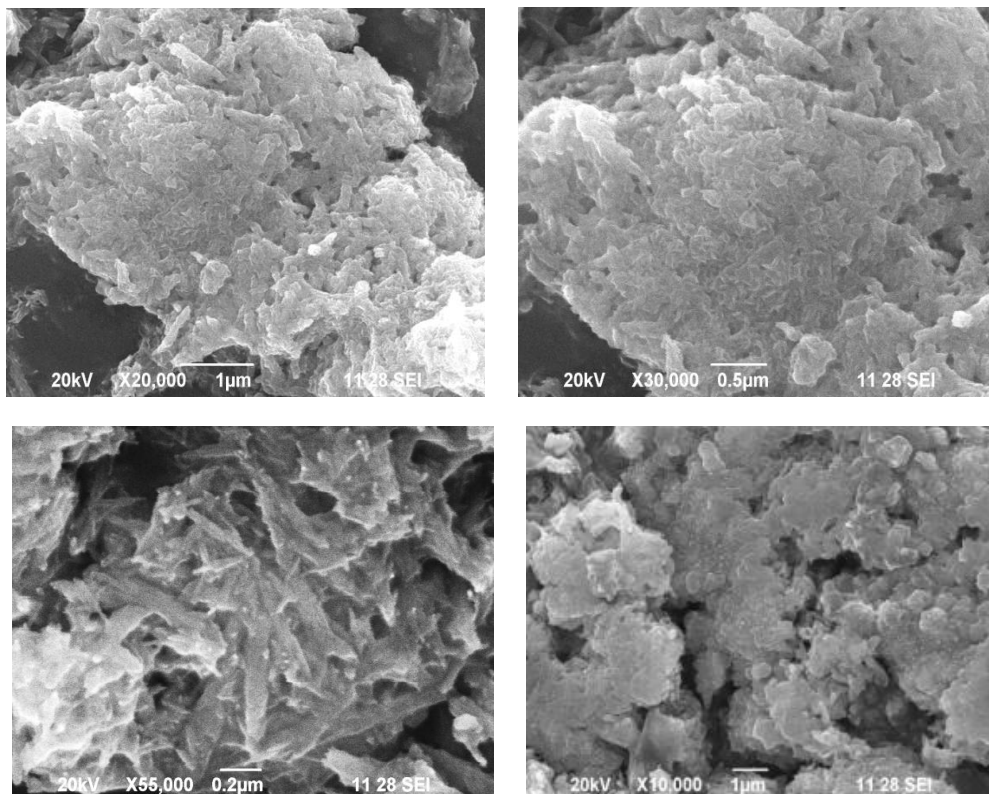
### 3.3 Surface Morphology

#### 3.3.1 Scanning Electron Microscope

Figure[3.7] shows pure CuO scanning electron microscopy (SEM) micrograph of prepared nano particle scanned by 20KV electron beam at different X20,000,X30,000 and X10,000,X55,000 magnification. The SEM image reveals the polycrystalline, porous morphology with the nano fused surface. The small crystallites agglomerated to form nano cluster elliptical nano particle and nano sheets. Fig[3.8] shows the scanning electron microscopy (SEM) micrograph of prepared Cd-doped CuO nano particle scanned by 20KV electron beam at X20,000,X30,000 and X10,000,X55,000 different magnification. The SEM image reveals the crystalline, porous morphology with the inter-connected grain presented on the nano surface. The small crystallites agglomerated to form nanoflower with stem, rice like structure nano particle may provide novel platform for photovoltaic sensor and other device applications.[10,11]



**Fig 3.7** SEM image of pure CuO

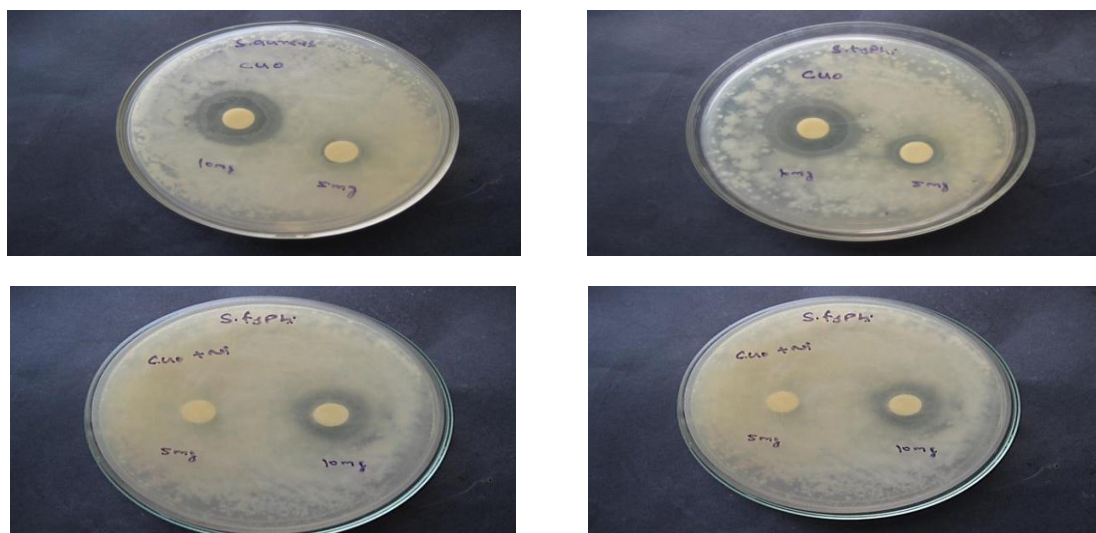


**Fig 3.8** SEM image of Cd doped Copper Oxide



### 3.6antibacterial Activity

Many antibacterial studies were made using different nanoparticles. The reason for anti-bacterial studies is due to the presence of reactive oxygen species generated by different nanoparticles[12-16 ]. Chemical interaction between hydrogen peroxide and membrane proteins or between the chemical produced in the presence of CuO, Ni CuOnano particles and the outer bilayer hydrogen peroxide to medium. In fig 3.12 we clearly see the antibacterial activityof the bacteria is completely destroyed and anti-bacterial activity still active. The result revealed that the antibacterial activity are sensitive to the test samples. The Antibacterial strains is summarized in table shows that the CuOshowed good antibacterial activity against the various bacterium like Staphylococcus aureus (Gram +ve), Salmonella typhi (Gram -ve), Bacillus subtilis, Escherichia coli their interaction with CuO are shown in fig (3.12) and also zone of inhibition is given in table.1.As the diameter of the zone of inhibition is high, we can conclude that CuO is very effective antibacterial agent.



**Fig 3.12** Antibacterial activity of Copper Oxide on Staphylococcus aureus, salmonella typhi

**Table1** Zone of inhibition of CuO and Ni doped copper oxide

Zone of inhibition in mm				
Name of the sample	Copper Oxide		Ni-Copper Oxide	
Name of the microorganisms	5 mg	10 mg	5 mg	10 mg
Staphylococcus aureus	11mm	26mm	11 mm	26 mm
Salmonella typhi	10mm	25mm	11 mm	25 mm

### IV. Conclusion

CuO nanomaterial was synthesized by co-precipitation method at room temperature. The material investigated here are the oxide of earth-abundant material. The powder X-ray diffraction pattern of the synthesized CuO confirm that the material is a nanomaterial of the particle size CuO and Cd, Ni doped CuO are 12.6 nm and 11.8 nm, 14.17 nm respectively. The functional groups in the Copper oxide and Cd, Ni doped copper oxide nanomaterial has been confirmed by FT-IR spectral analysis..Low cost production is the main advantage for any product to compete in the market and CuOnano structures being solution process able provide much potential to become the choice for cheap devices. The surface morphology of the samples were determined and structure confirmed by SEM. The CuOnano structure is of great importance because it enables fabrication of humidity sensor with improved performance at low cost, which may be breakthrough for the development of an efficient humidity sensor.

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