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Investigate the Change in Structural and Optical Properties with Dopant Concentration of CUO in Alumina Nanostructured Materials

Ritu[1*], Sunil Kumar Dwivedi[2], Rajesh Sharma[3]

[1]Ph.D Scholar, Physics Department, SAS, Om Sterling Global University, Hisar (Haryana), India*
[2]Assistant Professor, Physics Department, SAS, Om sterling Global University, Hisar (Haryana), India
[3]Assistant Professor, Physics Department MNS Govt. College, Bhiwani(Haryana) 127021, India

Email: [2]drsunilkumar@osgu.ac.in, [3]rkkaushik06@gmail.com
Corresponding Email: [1]rituphy191@osgu.ac.in*

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Abstract: ***Nano sized Al2O3 and Cu doped Al2O3 nano-sized particles have been synthesized by using microwave assisted chemical co-precipitation method. The structural, morphological and optical band gap energy of the calcined samples have been examined by using X-ray diffraction techniques, FESEM, HR-TEM and UV-Vis absorption respectively. The insertion of Cu^{2+} ions into Al2O3 NPs gave significant alteration in structural and morphological properties. The Crystalline sizes of Cu-doped Al2O3 were calculated by Debye Scherrer formula and it is approximately 21 nm. The UV absorption study shows strong absorption peak around 250 nm. The FTIR spectroscopy of calcined samples were at 530 cm^{-1} and at 512 cm^{-1} proving Cu^{2+} ion in Al2O3 lattice . The FESEM images confirmed that the samples are nanosized and more or less truncated hexagonal in shape.***

Keywords: ***Microwave-Assisted Chemical Co-Precipitation, Al2O3 NSP, FESEM, HRTEM, FTIR and UV-Visible Spectroscopy.***

1. INTRODUCTION

The definition manipulation, synthesis and analysis of materials at nano scale is termed as nanoscience. The particles/materials whose dimensions lies in between 1-100nm are known as nanoparticles or nano-stuffs. In present scenario nanotechnology becomes subject of knee

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interest for researchers because of its attractive properties in various fields such as large surface to volume ratio, electrical and optical properties etc. The drastic change in properties of particulate at nano scale are mainly owing to its great surface to volume ratio (S/V), quantum confinement effect and modular optical band gap. Nanoparticles having fine ordered arrangement of atoms/ions are termed as nano- crystallites. In present times, nanoparticles have become a subject of great scientific
concern as they provide a bridge between molecules at atomic level and bulk materials. Nanotechnology deals with artificial structuresas well as natural structures on nanoscale. In this paper the authors try to explain the change in structural and morphological properties with dopant concentrations of Cu^{2+} ion in Al_2O_3NSP.

Synthesis Techniques

All chemical used in present work were purchased from HIMEDIA chemical limited and highly purified in nature (99.9% pure)/ AR grade. The authors declared that no extra purification were made at laboratory scale. All the samples i.e copper doped Alumina have been synthesized via advance chemical co-precipitation method (Microwave treated) The appropriate concentration of salts of copper and aluminium i.e $Cu(II)(NO_3)_2.3H_2O$(Copper nitrate (molecular weight(mwt.)- 241.60) and $A_2(NO_3)_3.9H_2O$ (mwt. 212.99) were mixed in 100ml doubly deionized water so that greenish colour transparent solution were obtained, The ammonium hydroxide solution was used as surfactant /base for the purpose of precipitation and added dropwise unless or until PH of solution reaches to 9.0 with formation of greenish white precipitates at the bottom of flask. The precipitates were filtered and multiply washed with dilute ethanol and doubly distilled water. The washed cake were collected in Petri dish and treated with microwave at temperature of 15^0 C for 15 minute and for two sitting and resulted dried cake were calcined with different temperature i.e 200^0C, 400^0C and 600^0C for fixed duration of 2 hrs. The finally calcined samples were crushed in agate mortar so that fine powder samples were in formation and analyzed the sample with different characterization techniques such as XRD, UV-VIS spectroscopy, IR spectroscopy , FESEM and HRTEM mechanisms.

Characterization outcomes and Analysis

X – Ray Diffraction Analysis

The X-rays were employed to various calcined samples to study the structural changes with dopant concentrations . The samples size were determine by considering the most intense alps and by using Cu(k alpha) radiations. The grain size were calculated by Debye Scherrer approximations and the results shows that the grain size increases with increase of dopant concentration. The calculations were shown in Table-1 as under

Table 1: Crystallite size of nano-structured stuff of Copper(II)oxide concentration(5 %, 10 % & 20 %) doped in Aluminium oxide ignited at 600^oC temperature for2Hrs.

CuO doping Concentration in Al_2O_3	**2 Theta (Degrees)**	**FWHM (radians)**	**Crystallite size(nm)**

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5%	38.8	1.4	11.9
10%	38.8	0.984	17.5
20%	38.8	0.388	28.1

The change in concentration of guest material results in increased grain size of the formed nano-compositions calcined at same temperature for equal time duration.

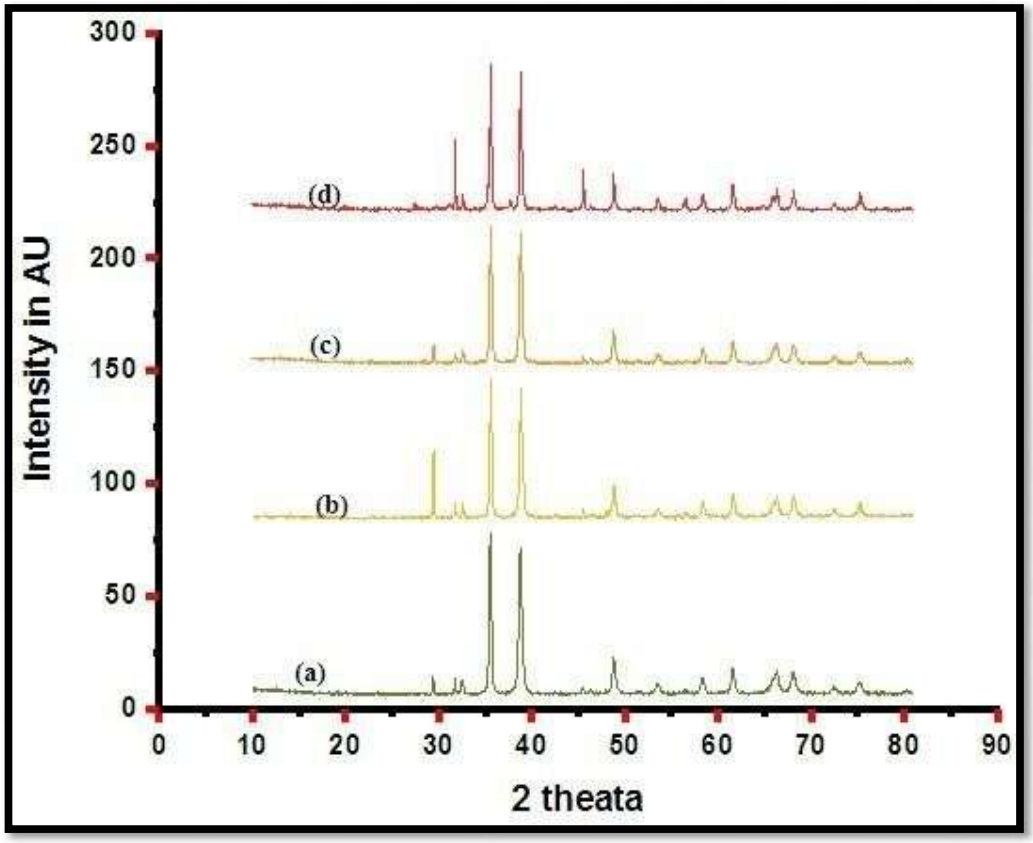

Figure 1: XRD graph of Copper oxide doped Aluminium oxide nano-structured stuffby utilizing various dopant concentrations (5 %, 10 % and 20 %) and calcined at 600^{O}C for duration of 2 hrs.

The XRD spectrum were shown in figure-1 and the perusal of images shows that the position of most intense peaks are more or less same, as analyzed by crystallographic image which clearly explored that crystalline behavior does not change with change in amount of incorporated material. However, significant changes were observed in full with half maxima in descending order.

IR Spectroscopic Investigations:
The elemental detection and contamination of other complexes have been examined through IR- spectroscopy. All the calcined samples were studied by utilizing Perkin Elmer IR spectrometer within the range of wave number (cm^{-1}) 400-4000 cm^{-1}. Figure 2 depicts the

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Fourier infrared transform spectrum of copper doped aluminium oxide heated at a certain temperature of 600^{O}C for a fixed period of 2 hours.

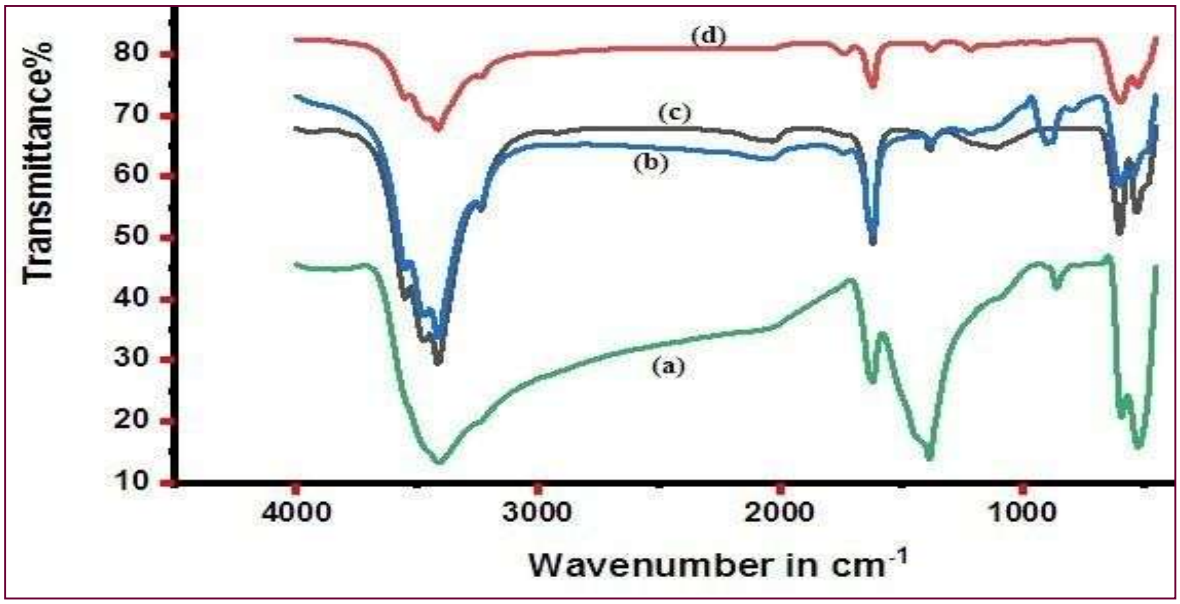

Figure 2: IR spectrum of CuO- Al_2O_3 nano-cosmposites with varying concentrations (a) pure Aluminium oxide (b) 5 % CuO- Al_2O_3 (c)10 % CuO-Al_2O_3 (d)20 % CuO- Al_2O_3 and calcined at 600^{O}C for two hrs.

The IR spectrum of various Copper(II) oxide doped Al_2O_3 have been analyzed and reflects that IR broad peaks around wave number (3410, 1717, 1394, 600 and 530) cm^{-1} and broad bands in region 3800 cm^{-1}- 2800 cm^{-1}, 1800 cm^{-1} – 1300 cm^{-1} and 800 cm^{-1} – 512 cm^{-1} have been investigated. Numerous alp/peak position presented in the graph are due to stretching mode of –OH group. The different mode of vibrations in H-O-H in linear or bending pattern or may be because of presence of water content in environment. Peak position near to wavenumber 600 cm^{-1} and 500cm^{-1} exists due to formation of different vibration modes of Al_2O_3 and confirms the formation of cuprous oxide and cupric oxide. Peak positions around wavenumber 1300 cm^{-1} exists due to CO_2 and NO_2 adsorption. The study of spectrum shows that change in dopant concentration changes the crystallite formation.

UV-Visible spectroscopy

The spectroscopic results of UV-Visible proportion of electro-magnetic (EM) radiationsof all samples were studied in the intermittent wavelength area 200 nm to 800nm. The spectrum of pure and Copper(II) oxide doped aluminium oxide nanocomposites with variant concentration (5%, 10%, 20%) heated at constant temperature of 600^{O}C and with constant concentration (10%) and a variable calcination temperature (200^{O}C,400^{O}C, 600^{O}C) is shown in figure-3(a) and 3(b) below.

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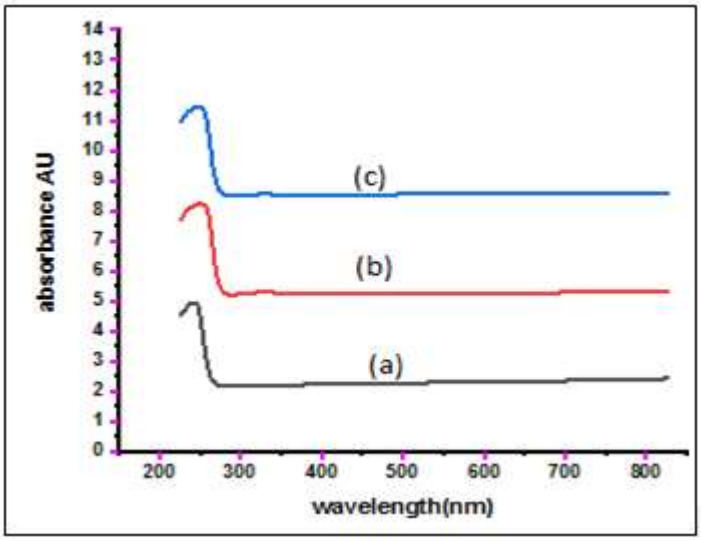

Figure 3(a): UV-Vis spectrum of Copper oxide doped Aluminium oxide nano- stuffs with varying concentration (5%, 10%, 20%) calcined at 600^0C for durationof 2 hours.

The absorbance alps look sharp near to wavelength 250nm and then become constant for further improvement in wavelength. The peaks sharpness near to 250nm explores about the crystalline behavior of the prepared nano-grains. The perusal of image showsthat the peaks position shifting towards red shift with varying dopant concentrations of copper (5 %, 10 %, 20 %) at constant temperature 600^0C for 2 hrs.

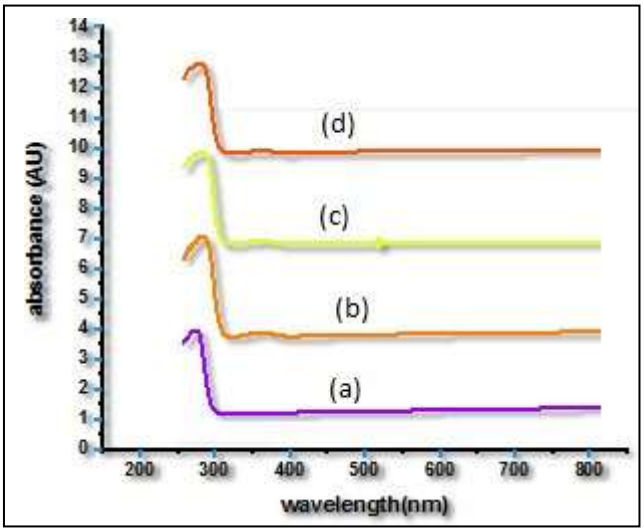

Figure 3(b): UV-Visible plot of pure and 10% Al_2O_3 doped Copper Oxide nano-stuffs ignited at temperature (200°C, 400°C & 600°C) for two hours.

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It has been observed that near the band edge of 250nm absorbance first diminishes sharply with the increase in wavelength confirming the occurrence of nano-matrix of the sample. The examined measurements of the coefficient of adsorption is more or less constant with numerous ignited temperatures and hence representing the uniformity in the crystallite size of agglutinated materials.

Field Emission Scanning Electron Microscope (FESEM) Investigations

To view the particulates which are smaller in dimension as co-related to the light energy wavelength, we required tiny sized particles so that they could be drawn back to the objective and envisaged very tiny particles and electrons plays an impressive role in it. Electron can enhance the magnification to a 10^{-6} times.

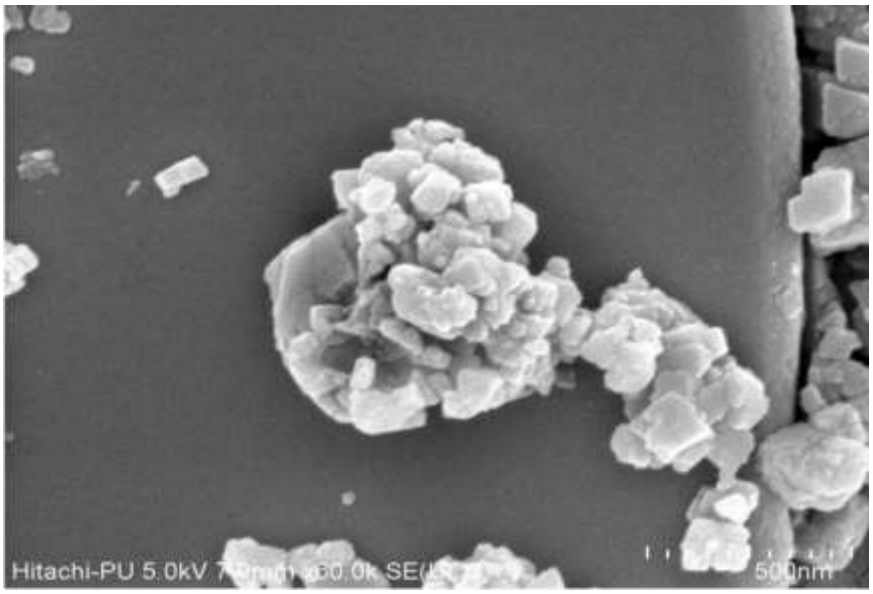

Figure 4: Field Emission Scanning Electron Microscope images of CuO doped in Al_2O_3 nanostructured materials.

The FESEM characterization was carried out by using instrument Hitachi-PU 5. established at SAIF PUNJAB UNIVERSITY, CHANDIGARH**.** The as-obtained image exhibits the creation of Copper(II) oxide doped Al_2O_3 nano-particulates. The pictures show that formed nano-composites are crispy in shape.

HRTEM Investigations

The Hitachi-PU-5 instrument at SAIF, Punjab University in Chandigarh was used for the HRTEM analysis. The high-resolution transmission electron microscopy (HRTEM) was employed to investigate the nano-composites, and the results are shown in Figure 5below.

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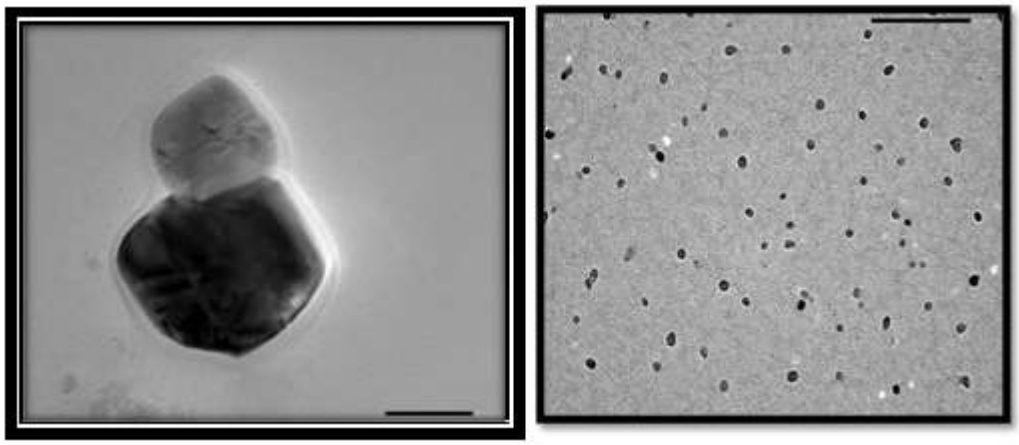

Figure 5: High Resolution Transmission Electron Microscope images of CuO doped$in\ Al_2O_3$ nanostructured materials.

The HRTEM images assured about the nano-dimension of the Copper oxide doped Aluminium Oxide particulates. From HRTEM study it becomes crystal clear that CuO crystals are truncated hexagonal in shape and homogenously distributed & have little bit agglomeration.

2. CONCLUSIONS

The CuO doped Al_2O_3 nano-particulates were successfully synthesized by micro-wave assisted chemical co-precipitation technique and grain size of samples were about 21 nm. The FESEM images insured the nanomaterial are crispy in nature. Upon conducting an HRTEM analysis, it becomes evident that CuO crystals have a characteristic truncated hexagonal shape. These crystals are evenly distributed across the sample and exhibit mild agglomeration The FTIR peaks concluded the incorporation of Cu^{2+}ion in Aluminium Oxide lattice. The UV-Visible spectroscopy showed red shift in optical bands.

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