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The Structural and Magnetic Variation with Calcinations Temperature in CoO doped Al_2O_3 Nanocrystallines

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ABSTRACT

In present scenario, the technological applications of nanomaterial are at key position because of unique in physico-chemical property at this scale. The present research work explored the effect of concentration of Co element on structural and morphological modifications in Cobaltous oxide Alumina nano-crystalline. In this research work, nanosized Cobaltous oxide Alumina sample was synthesized via advanced co-precipitation chemical protocol. The specimens have been calcined at 200^0C, 400^0C, 600^0C for a time span of 2hours. The XRD results reflects that The crystalline size of newer nanocrystallines were increase in particle size with increase in calcinations temperature Cobaltous oxide Alumina hexagonal structure .The FTIR tools was used to examine the purity of samples and entities of Cobaltous oxide Alumina structural material. The magnified images of samples were seen through FESEM and HRTEM tools and exhibited structure were 2-D nanosheets were in formation.

Keywords: Cobaltous oxide, Debye-Scherrer, VSM, XRD, FTIR, FESEM and HRTEM etc.

INTRODUCTION

The superior properties of nanomaterials are widely recognized, making them a popular topic due to their numerous enhanced applications. Researchers are highly interested in these materials because they believe many possibilities remain unexplored. Nanomaterials could lead to significant advancements, as their unique characteristics at the nanoscale such as morphology, size, and agglomeration behavior—hold great potential. These nanoscale properties enable superior performance in various fields and innovations that rely heavily on nanotechnology's unique capabilities. Nanomaterials differ significantly from their larger counterparts due to a high surface-to-volume ratio and quantum effects impacting their optical properties. At the nano-scale, it was examined that the effect of quantum channels has a huge influence on the optical features of the stuff. The CoO nano-flecks are of immense significance. As we had seen before that the nano-scale composites of Cobaltous Oxide alongside Transition Metal Oxides at variant dopant concentrations showed distinct/unique properties as well as behavior at the nanoscale.

Aluminium Oxides fetched quite more attention towards itself as it possesses much significance both in technology and in science.Since, the Aluminium has various stable oxidation states; it can form the various kinds of oxides. Among other metal oxides, special surveillance has been focused on the fabrication/preparation and properties of Aluminium Oxide (Al_2O_3), which is an important role in some specifically applied usages like as in the field of liquid crystal displays, high-temperature resistant materials, corrosive resistant materials, green pigment, catalysts, etc. In this work the various samples of Cobaltous oxide Alumina were synthesized via advance chemical co-precipitation protocol and the Structural analysis of composite materials were done with the help of X-ray diffraction, Fourier transform infra-red spectroscopy, VSM, FESEM and HRTEM image magnifications.

Experimental Details

The nano stuffs of CoO doped Al_2O_3were synthesized by the Micro-wave treated co-precipitation advance synthesis techniques. The ignited stuff of various concentrations ignited at various temperatures for fixed duration.The slurry of the appropriate concentration of $Al_2(NO_3)_3.9H_2O$ (HIMEDIA, India) and $CoCl_2.6H_2O$ (HIMEDIA, India) was foregathered/mixed in the de- ionized water of 100 ml. Then the slurry of NH_3 was poured in the above said slurry at 100^0C temperature and the finalize amalgam was stirred at the temperature of 125 ^{0}C for 2 hours of time span using magnetic stirrer and then the concluding amalgam was retained for the ageing process at room temperature for at least 24 hours. On completion of reaction, the concluding yellowish-white colored precipitates were strained and then made to wash with the doubly distilled water. These precipitates were also washed then with ethanol or Merck for manifold times to get rid of the impurities or by-products. The filtered cake was then dry in the air at 100°C temperature for 4 hours of time span. The as-

synthesized specimens of the different concentrations had been ignited eventually for various time scales and at a particular temperature in the air.

The specimen was crushed in an agate mortar to obtain a fine powder of CoO-doped Al_2O_3 for further characterization.

RESULTS AND DISCUSSION

Structural characterization:

To allocate the framework of ignited specimen by using X-ray diffractometer with copper (CuK_α)radiation (λ = 1.5408 Å) in the confine of 10^0–80^0 the powder sample studies using X-rays have been accomplished. The XRD designs of various cobaltous Oxide doped Alumina nano-sized stuff ignited at 600°C temperature for 2 hours are exhibited in **Figure-1**

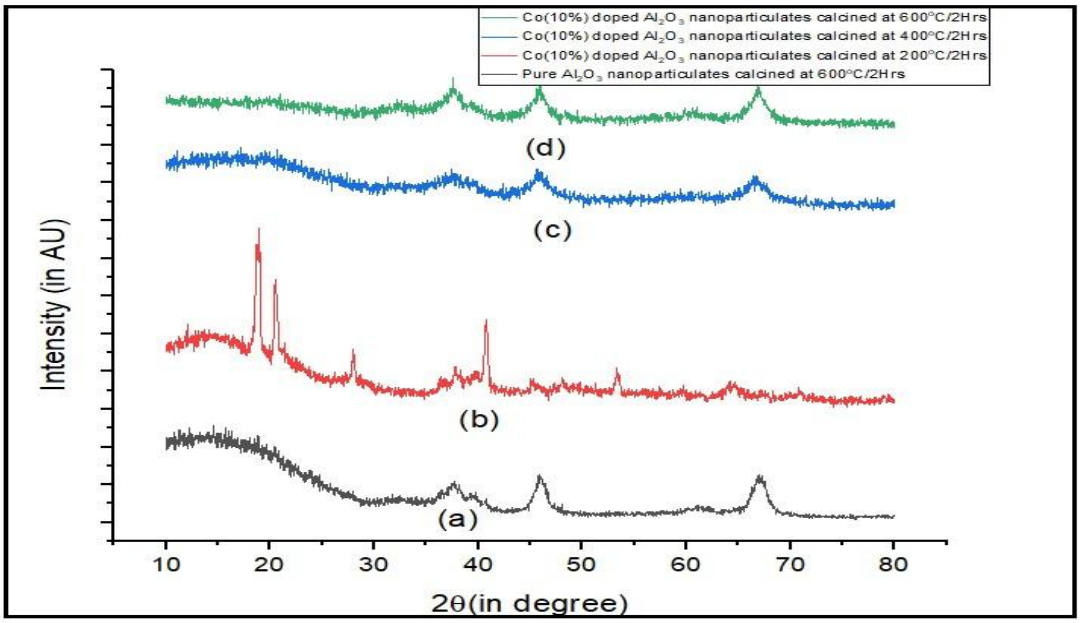


Figure-1: Cobaltous Oxide doped Al_2O_3 (10%) nano-sized stuff ignited for 2 hours at 200°C, 400°C, 600°C

The grain size for Cobaltous Oxide doped Al_2O_3(CoO, 10%) nano-sized stuff ignited at 200 ^{0}C for 2 hours is 26.76 nm, Cobaltous Oxide doped Al_2O_3(CoO, 10%) nano-sized stuff ignited at 400 ^{0}C for 2 hours is 37.44nm and for Cobaltous Oxide doped Al_2O_3(CoO, 10%) nano-sized stuff ignited at 600 ^{0}C for 2 hours is 38.17nm. It is as Co-atom having more atomic radii measure than that of Al-atom concluding in rise in the grain size of nano-sized stuff with rise the temperature of specimens of Cobaltous Oxide doped Al_2O_3 nano-sized stuff.

Table-1: XRD Cobaltous Oxide doped Al_2O_3 (10%) nano-sized stuff ignited for 2 hours at 200°C, 400°C, 600°C

Sr. No.	Calcinations temperature (°C)	2Θ (Degrees)	FWHM (Radians)	Crystallite size (nm)
1	200°C	18.79	0.52	26.76 nm
2	400°C	37.56	0.39	37.44 nm
3	600°C	45.73	0.38	38.17 nm

It was clearly observed that the location/position of XRD alps is not changed but the intensity of XRD peaks shows increasing behavior with rise of temperature and the small change in β occur with change in temperature i.e., the value of β decreases with increase of temperature. It implies that the size of grain rises with rise of temperature. It might be due to upsurge of lattice energy with upsurgences of temperature

FTIR Spectroscopy analysis

The IR spectroscopy were used to identified the group/ contamination particles/ other entities present in the samples. The transmittance rate were noted with wave number of radiation incident on samples ranging from 400-4000 cm$^-$1.The FTIR

electromagnetic spectrum of synthesized specimen of CoO doped Al_2O_3 nano-sized stuff ignited at 200^0C, 400^0C, 600^0C for 2hrs. The IR data were represented in graphs as shown in Figure-2.

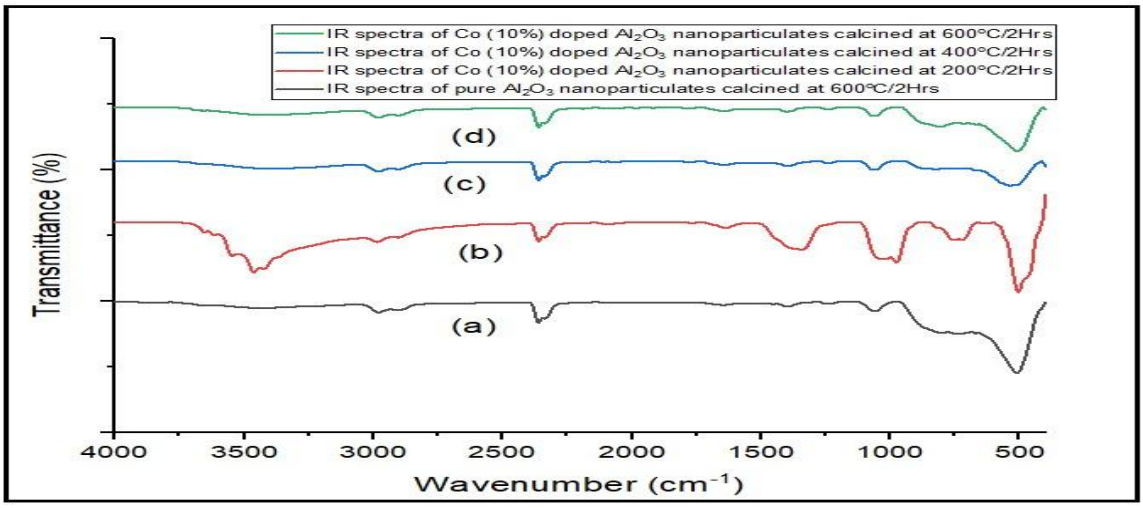


Figure - 2: FTIR Spectra of Cobaltous Oxide doped Al_2O_3(10%) nano-sized stuff ignited for 2 hours for distinct temperature (a)200°C (b) 400°C (c) 600°C

The FTIR analysis of the synthesized Alumina nanoparticles revealed several characteristic peaks, indicating the presence of various functional groups. At peak positions 516 cm^{-1} and 837 cm^{-1}, Al-O-Al and Co-O-Co vibrations were identified. A peak at 1300 cm^{-1} corresponds to Co-O-Al bonding. Additionally, peaks at 1546 cm^{-1}, 2330 cm^{-1}, and 3402 cm^{-1} indicate the presence of -OH groups. These -OH group vibrations are due to water molecules present in the atmosphere, which interact with the nanoparticles. This detailed analysis helps in understanding the chemical structure and bonding of the synthesized Alumina nanoparticles. FTIR spectra of the Cobaltous Oxide doped Al_2O_3(10%) nano-composites ignited for 2 hours at 200 ^{0}**C**, 400 ^{0}**C**, 600 ^{0}**C** of assembled specimen are exhibited. The perusal of the image exhibits that consign of the ignited specimens rises with rise in calcinations temperatures. It might be due to rise of the condensation of the oxygen throughout calcinations course of action.

Sr. No.	**Sample Name**	**Saturation Magnetization (10-3 emu/g)**	**Coercive Field (in Hc Oe)**	**Maximum Permeability (in10^{-6} emu/g/Oe)**	**Max. energy loss(inMGsOe)**	**Remanent Magnetization (10^{-3}emu/g)**
1	Pure Al 600°C/2hrs	74.653	105.673	97.703	14.215	10.220
2	Co doped Al_2O_3 200°C/2hrs	1.769	71.402	2.400	7.353	170.462
3	Co doped Al_2O_3 400°C/2hrs	4.004	68.121	5.733	8.762	393.382
4	Co doped Al_2O_3 600°C/2hrs	1.489	74.222	2.023	7.183	151.559

Vibrating Sample Magnetometer Results:-

The magnetic properties of various samples were explored through deploying vibrating sample magnetometer tools and the applied field ranges were taken from 0-10,000 Os. In present work the various calcined samples of Cobalt (Co) 10% doped alumina nanocrystalline were studied through VSM tool and result were compare with pure alumina sampled calcined at 600℃ for 2 hrs.

The data received from lab CEERI Pilani were represented in various graph. The perusal of graphically representation show that the particles are more or less ferromagnetic in nature with small hysteresis
loss.

Table:-

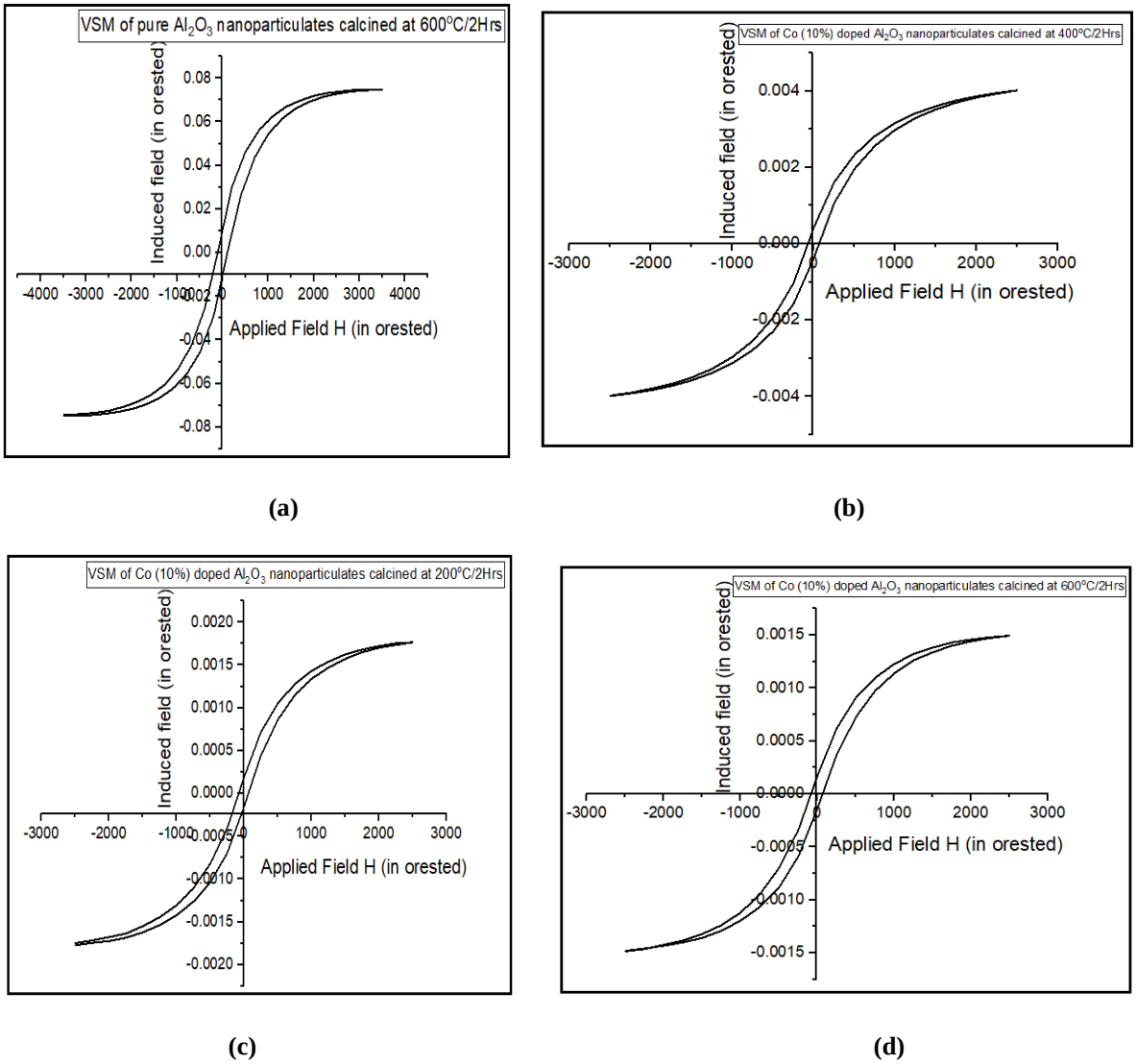


Fig. 7.7. VSM graph of pure Al2O3 at (600°C) Fe doped nano-particulates calcined at (a)200°C (b) 400°C (c) 600°C

High Resolution Transmission Electron Microscopy (HRTEM)

Investigation
HRTEM micro-graphs of Cobaltous oxide doped Al_2O_3 nano-composites with concentration 10% ignited at 600 ^{0}C for 2 hours were exhibited in figure-3**.**

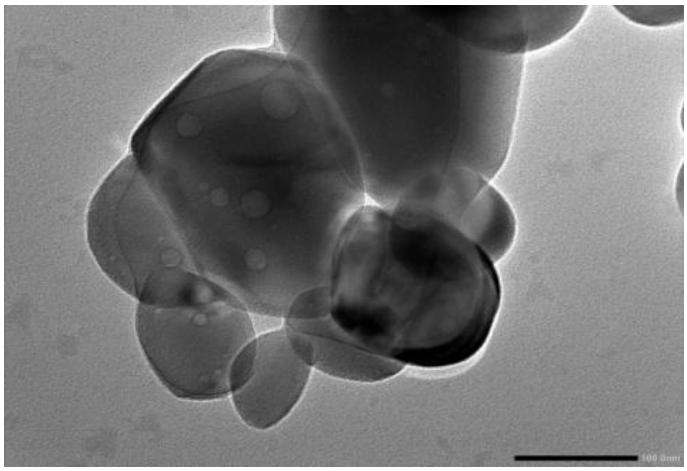
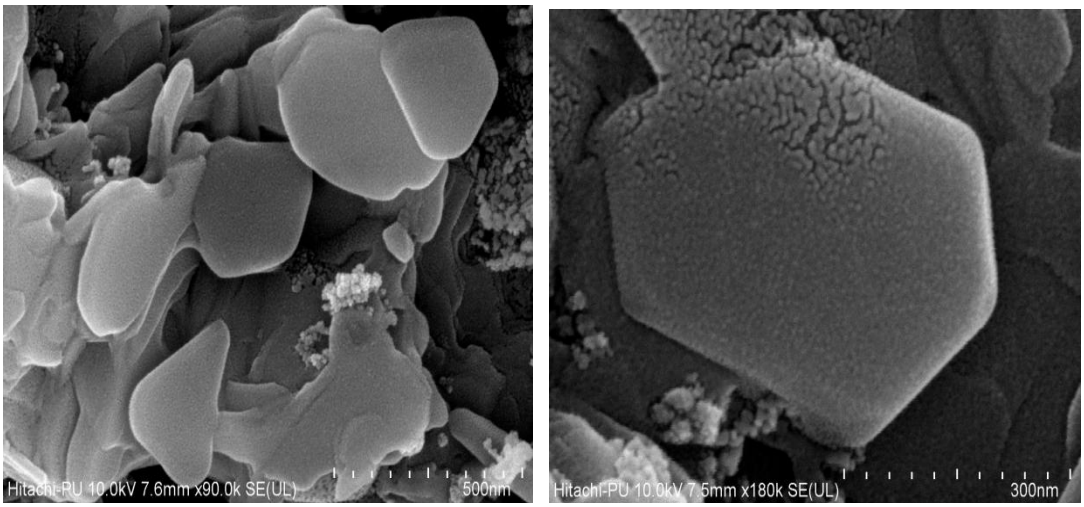


Figure-3: .HRTEM images of CoO dopedAl_2O_3(10%)nano-sized stuff ignited at 600 ^{0}C for 2 hours

Examination of the Image exhibits that diameter of the nano-sized stuff ranging from 26 to 38 nm and intermediate grain size estimated to be 32nm. The micrographs of TEM concluded that grain size results resemble with XRD results and clarified that grain size rises with doping molar concentration. From the micro-graph, it was inspected that the nano-fleck are polycrystalline kind and spherical in contour.

Field Emission Scanning Electron Microscopy (FESEM) study

The scanning of sample through electron microscopy images of Cobaltous oxide doped Al_2O_3nano-sized stuff ignited at 600 ^{0}C for 2 hours were more or less similar to typical scanning of sample through electron microscopy. Micrograph of Cobaltous oxide doped Al_2O_3 (10%) nano-composites ignited at 600^{0}C for 2 hours is exhibited in Figure-4**.**



(a) **(b)**

Figure -4: FESEM images of CoO (10%) doped Al_2O_3nano-sized stuff ignited for 2 hours (a)400^{o}C(b) 600^{o}C

Examination of Image exhibits that flecks are polycrystalline, cluttered in style and
2D nano thin film in formation with flecks like structure.SE

CONCLUSIONS

The nano-sized stuff of Cobaltous oxide doped Al_2O_3 having various calcinations temperature of the Cobaltous oxide has been assembled by Micro-wave treated co-precipitation advance synthesis protocols. The conclusions of HRTEM assist the XRD sequels grain size approximate to 26 to 38 nm. The Perusal of HRTEM images exhibit that the size of all ignited

Cobaltous oxide doped Al_2O_3 nano-networked specimen lies in a confine of 26 nm to 38 nm and 2-D nanosheets were in formation. The FTIR Spectra of the ignited nano networked stuff of Cobaltous oxide doped Al_2O_3 containing calcinations temperature was exhibiting the Alps(peaks) at positions 516 cm^{-1} and 837 cm^{-1}, Al-O-Al and Co-O-Co vibrations were identified. A peak at 1300 cm^{-1} corresponds to Co-O-Al bonding. Additionally, peaks at 1546 cm^{-1}, 2330 cm^{-1}, and 3402 cm^{-1} indicate the presence of -OH groups. These -OH group vibrations are due to water molecules present in the atmosphere, which interact with the nanoparticles. which are similar to the alps(peaks) as appeared in Cobaltous oxide nano-flecks.

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