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Variation in Structural and Magnetic Properties in $Cr_2MgO_{4\pm\Delta}$ Nanocrystallines with Mg^{2+} ion Concentration

Megha[1], Rajesh Sharma[*2], Sunita Dahiya[3]

1) Research Scholar, Baba Mastnath University. Rohtak-124001
2) Faculty in Physics, MNS Government College, Bhiwani-127021
3) Professor in Physics, Baba Mastnath University. Rohtak-124001

Corresponding Author[*]: rkkaushik06@gmail.com

ABSTRACT

In present scenario, the metal oxide nanoparticles have numerous applications in various fields of Science & Technology. Nanocrystalline Cr_2O_3 is well known for its remarkable thermal stability and other diverse applications as a colour pigment, catalyst and key bactericidal material. The microwave assisted co-precipitation method was employed in present work for the preparation of MgO (5%, 10%, 15%) doped Cr_2O_3 nanocrystallines calcined at a fixed temperature of 600^oC / 2 hrs. The XRD data of various Mg doped Cr_2O_3 shows that Cr_2O_3 parental rhombohedral structure was in formation and no phase transformation occurrence with rise of Mg^{2+} ion concentrations. However, a remarkable change was noticed in respect of FWHM and intensity of respective XRD peaks. The crystallite size gradually increases with Mg^{2+} ion concentration in MgO/ Cr_2O_3 nanocrystallines. The IR spectrum of various samples concluded that as usual water molecules broad bands found at about 3000 cm^{-1} whereas, the sharp peaks at about 423 cm^{-1}, 577 cm^{-1} and 623 cm^{-1} were attributed to Cr-O-Cr and peak at 868 cm^{-1} is the peak of O-Mg-O and O-Cr-O vibrations respectively. The *M-H* curve studies of various Mg (5%, 10%, 15%) doped Cr_2O_3 samples shows that novel materials were ferromagnetic in nature and Mg (10%) doped Cr_2O_3 calcined samples have optimum magnetic properties i.e., remnant field, coercive field and saturation applied field as 6.711 x 10^{-3} emu/g, 53.70 emu/g and 94.965 x 10^{-3} emu/g respectively. Henceforth, Mg (10%) doped Cr_2O_3 samples have superior magnetic properties with respect to others and might have futuristic applications in the fields of MRI, magnetronics and in fabrication of memory chips. The magnified view (X50,000) of samples as detected through Field Emission Scanning Electron Microscopy (FESEM) tool shows that particles were 2-D slaty stone like structures with agglomerated nature at the scale of 100 nm and their average thickness was in the range of 10 – 20 nm.

Keywords: Mg doped Cr_2O_3, MgO/ Cr_2O_3 NCs, XRD, IR studies, VSM and FESEM.

INTRODUCTION

The advanced materialistic properties of materials created a keen interest of today's researchers with assembly of novel materials at nanoscales. The functionality of materials highly influenced surface to volume ratio and quantum confinement effect at scale of 1-100 nm[1-2] .The Transition Metal Oxide Nanocrystallines (TMONs') have numerous enhanced properties because of availability of valence d-subshell electrons and among TMONs', Chromium Oxide possesses multiple Cr^{2+} and Cr^{3+} oxidation states i.e., +1, +2, +3 along with uniqueness and promising applications in the fields of photocatalysis, medicine, electronics, antibacterial and also in curing of cancerous cells etc[3-8]. The aforesaid properties of Chromia make it significant among other metal oxides. Moreover, the Magnesium Oxide nanoparticles have key features in the field of antibacterial actions, making of hydrogen storage devices and water purification[9-10]. So, therefore, the researcher aimed to synthesize Chromia and Magnesium doped Chromium samples via advanced chemical Co- precipitation techniques and thereafter studied their structural and magnetic innings of novel materials along with variation in concentration with a constant uniform heating at calcination temperature at 600^oC / 2 hours. The synthetic methodology and characterization results were discussed in details in subsequent sections of this paper.

Experimentation

Synthesis of Cr_2O_3 (Chromia) and Mg doped Cr_2O_3 Nanocrystallines

The Mg doped Cr_2O_3 and Cr_2O_3 pure nanocrystallines were prepared by microwave assisted chemical route methods .The authors declared that all chemicals used in present study were highly pure and A.R. grade in nature and were not be further purified at laboratory scale. The appropriate amount of $CrCl_3$ $.6H_2O(s)$ and $MgCl_2.6H_2O(s)$ were taken as base salts and dissolved in 100ml doubly deionized water (DDI) with refluxing mode. The 2.0 M NaOH solution was added dropwise until or unless pH of solution rose to 9.0 with occurrence of greenish precipitates in the solution. The resultant solution with precipitates were kept for ageing process for 24 hours followed which the greenish colored precipitates got settled down at the bottom of the beaker and then collected precipitates after filtration process. Contamination in the filtered cake was removed via multiple washing with dilute Ethanol and DDI water respectively. The pure precipitates cake were kept in domestic microwave oven at 100^oC / 2hrs for two sittings of 15 minutes each and thereafter, calcined the same at 600^oC / 2hrs for better crystallinity. The calcined samples were crushed in agate mortar and used for further characterization process.

Instrumentation Used

The structural properties of pure Chromia and Mg doped Cr_2O_3 samples calcined at 600^oC/2 hrs were studied using Perkin Elmer X Ray Diffractometer having $\lambda = 1.5406$ A^o with recorded parameter on x-axis as 2θ (in degrees) and intensity on the y-axis. The technique of Vibrational Sample Magnetometer (VSM) was employed to analyze magnetic properties of samples over a wide range of magnetic field H = ±1.5 **T**. Presence of various elemental groups was assessed with IR spectroscopic tools i.e. Perkin Elmer IR spectrometer. The morphological studies of samples were examined by **JEOL-3600 FESEM** tool. The various results as shared by different labs were discussed in subsequent section of this paper.

RESULTS AND DISCUSSION

X-Rays Diffraction Data Analysis

The pure Chromia and various samples of Mg doped Cr_2O_3 powdered samples have been examined through diffraction of X rays through these samples . The recorded intensity of diffracted beams was noted over 2θ ranging between 10^o and 80^o . The 2θ versus recorded intensity of various of Cr_2O_3 and MgO/ Cr_2O_3 calcined at 600^oC/2hrs were represented in graphical form in Fig.1 and the calculated value of crystallite size, intensity and FWHM were tabulated in Table 1 as under.

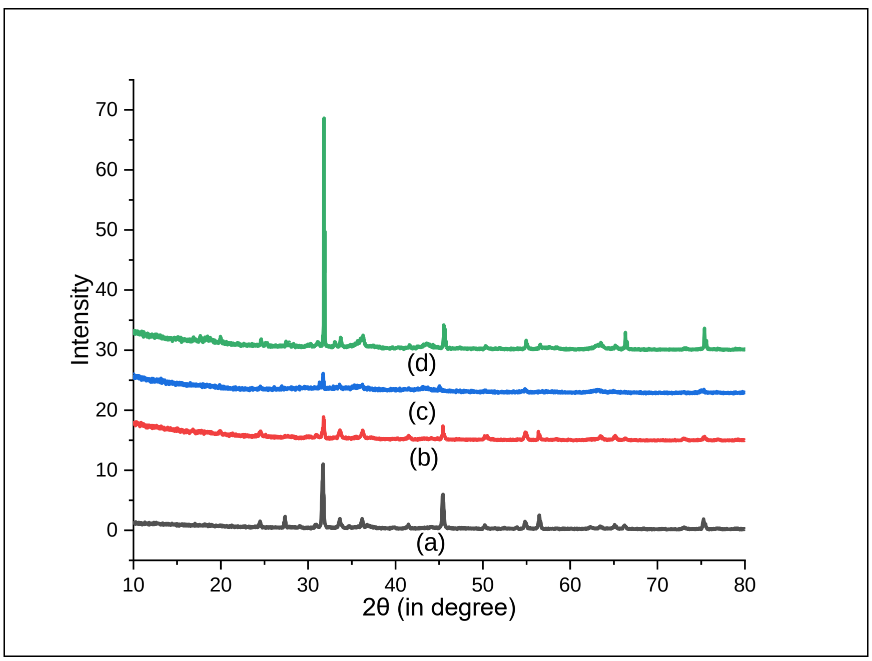


Fig. 1 — XRD Spectrum of calcined samples of (a) Pure Chromia and Mg (b) 5%, (c) 10%, (d) 15% dopedCr_2O_3 nanocrystallines calcined at 600^oC / 2 hrs respectively.

The Fig.1 shows that various peaks exhibited at 2θ positions **31.8^o**, 33.6^o, 37^o, 43^o, 46^o, 56.5^o, 58^o and concluded that the novel materials are crystalline in nature with the formation of rhombohedral structure. The MIP positioned at $2\theta \approx 31.8^o$ were taken for estimating the grain size of various samples. The peaks positioned at **31.8^o**, 33.6^o, 46^o and 56.5^o were analogous to ICSD File No. 00-038-1479 of Cr_2O_3 and $2\theta \approx 37^o$, 43^o and 58^o were matched with JCPDS Card No. 45-0946 of MgO. Moreover, the study reflects that no phase occurrence was found with doping and Mg^{2+} ions replaced Cr^{2+} ions against their lattice sites. The average crystallite size (d) was determined by the Debye Scherrer formula

$$D = 0.89\lambda / \beta\cos\theta,$$

where all the notations have the usual scientific meanings.

Table 1 — The calculated FWHM, MIP and crystallite size (d) of pure Cr_2O_3 and Mg (5%, 10%, 15%) doped Cr_2O_3 samples calcined at 600° C / 2 hrs.

Sr. No	Sample Name	FWHM(in rad)	MIP2θ (in °)	d (in nm)
1	Pure Cr_2O_3 NCs	0.00428	31.64	32.23
2	Cr_2O_3/MgO (5%)NCs	0.00392	31.78	35.69
3	Cr_2O_3/MgO (10%)NCs	0.00381	31.70	36.45
4	Cr_2O_3/MgO (15%) NCs	0.00379	31.80	37.65

Tabular data reflects that FWHM values gradually decreases and crystallite size continuously increases with Mg dopant concentration in Cr_2O_3 samples which might be due to more ionic radii of Mg^{2+} ions (72 pm) as compared to that of Cr^{2+} ions (66 pm) under identical conditions.

IR Data Analysis

The infrared waves were applied in FTIR spectroscopy for examination of all the calcined samples. The recorded data in terms of wavenumber and transmittance rate were taken for study and the graphical views of these data were shown in Fig. 2. as under.

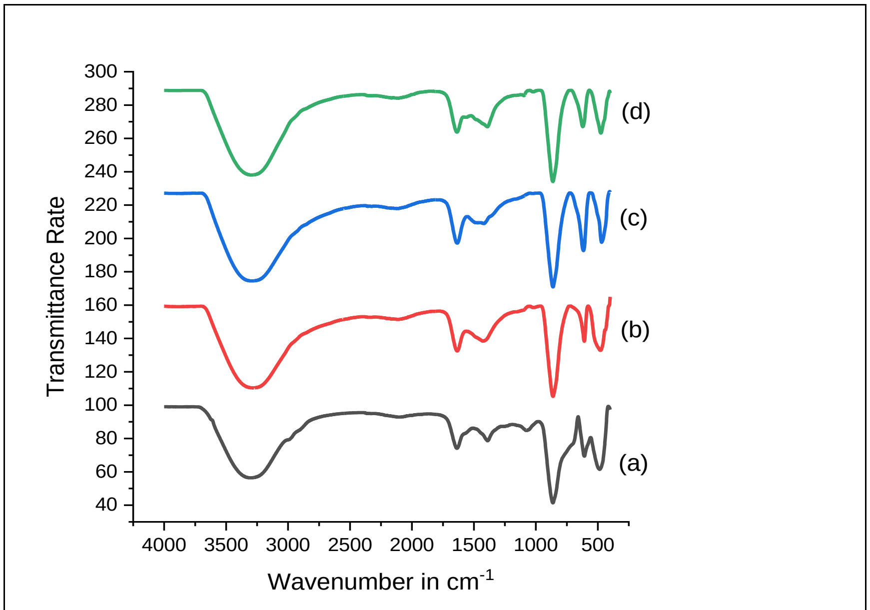
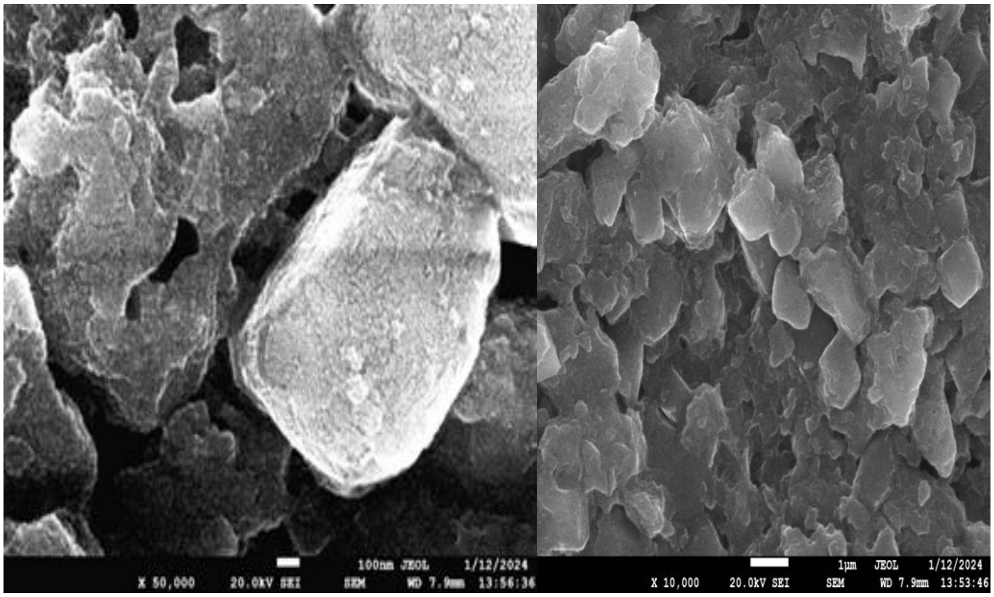


Fig. 2 — IR Spectrum of calcined samples of (a) Pure Chromia and Mg (b) 5%, (c) 10%, (d) 15% doped Chromia calcined at 600°C / 2 hrs.

The perusal of Fig. 2 shows that as usual OH ion vibration peaks of H_2O were found as broadband positioned about 3200 cm^{-1} and 1650 cm^{-1} respectively which might be due to water content present in atmospheric air of the samples. The peak positioned at about 1350 cm^{-1} was due to O = C = O of CO_2 present due to oxidation of C- atom.[8-10]. The enabled peak of O-Cr-O vibrations were found at 423 cm^{-1}, 577cm^{-1} and 623 cm^{-1} alongwith different oxidation states whereas, the peak positioned at 868 cm^{-1} were attributed to O-Mg-O vibrations and their presence reflects that Mg^{2+} ion was well incorporated in rhombohedral structure of Cr_2O_3 with the replacement of Cr^{2+} ion respectively.The IR studies support the XRD results.

FESEM Image Analysis

The pure Chromia and Mg doped Chromia samples calcined at 600°C for 2 hours were put under the FESEM (Field Emission Scanning Electron Microscopy) technique with 20.0 kV as the operating voltage with different magnification rates and scale of 50,000 and 10,000 as represented in Fig. 3 (a) and 3 (b) respectively. The image analysis concluded that the calcined samples were seen as 2D -slaty stone like structures with agglomerated nature at the scale of 100 nm with average size between 10 – 20 nm and nanoflakes in exhibition at scale of 1µm.



(a) (b)

Fig. 3 — FESEM image of (a) Pure Chromia and (b) Mg 15% doped pure Chromia calcined at 600°C / 2hrs.

VSM Data Analysis

The various magnetic properties of pure Cr_2O_3 and Mg (5%, 10%, 15%) doped Cr_2O_3 calcined at 600^{o}C / 2 hrs were explored through scanning of samples over a wide range of applied external magnetic field of ±1.5 **T** in Vibrating Sample Magnetometer. The M-H graph of foresaid samples were shown in Fig. 4 as under.

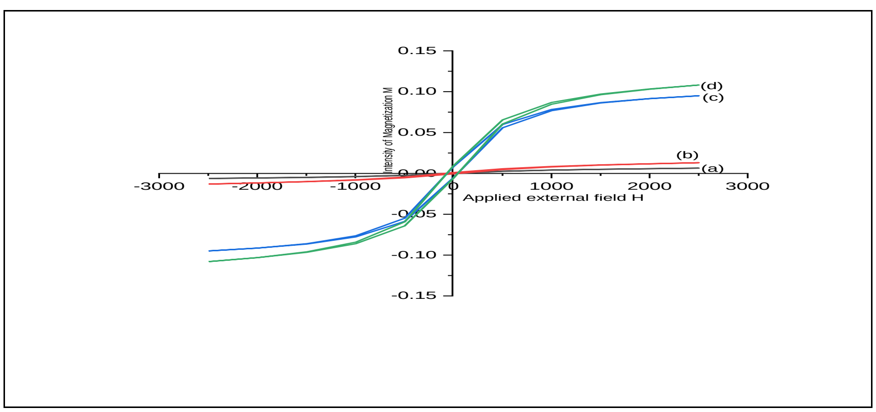


Fig. 4 — The VSM spectrum of (a) Cr_2O_3 pure, (b)Mg 5%, (c) Mg 10% and (d)Mg 15% doped Cr_2O_3 samples calcined at 600^{o}C / 2 hrs.

The VSM graph of various Cr_2O_3 pure and Mg (5%, 10%, 15%) doped Cr_2O_3 samples calcined at 600°C / 2hrs reflects that all the samples appeared as ferromagnetic in domain and magnetic character increases with increase of Mg concentration in Cr_2O_3 / MgO NCs. The Chromia and 5 % Mg doped Chromia have lesser magnetic behavior whereas, Mg (15%) doped Chromia NCs have better response at higher externally applied magnetic field. The conclusively different parametric analysis were shown in Table 2 as under.

Table 2 — The Hysteresis parametric study of pure Chromia and Magnesium (5%, 10%, 15%) doped Chromia at 600^o / 2 hours.

Sr. No	Sample	Ms. Saturation (emu/g)	Coercive (Hc Oe)	Remnant (emu/g)	Squareness factor	M at Max field (emu/g)	Energy Loss(Oe)	Saturation Applied H_s(Oe)
1	Pure Cr_2O_3 (600^oC)	6.340×10^{-3}	61.764	336.761×10^{-6}	0.053	6.340×10^{-3}	$7.057E^{+0}$	2304.36
2	Mg(5%) Cr_2O_3 (600^oC)	13.113×10^{-3}	**76.008**	815.354×10^{-6}	0.062	13.113×10^{-3}	$7.890E^{+0}$	2296.23
3	Mg(10%) Cr_2O_3 (600^oC)	**94.965×10^{-3}**	53.703	**6.711×10^{-3}**	0.071	94.95×10^{-3}	$20.508E^{+0}$	**1889.18**
4	Mg(15%) Cr_2O_3 (600^oC)	12.778×10^{-3}	49.704	489.532×10^{-6}	0.038	12.698×10^{-3}	$7.845E^{+0}$	2281.10

CONCLUSION

The pure Cr_2O_3 and Magnesium (5%, 10%, 15%) doped Cr_2O_3 samples were successfully prepared via microwave irradiated chemical co-precipitation method and thereafter, the powdered samples were calcined at 600^oC for 2 hours. The various calcined samples have been analyzed with X-Ray diffraction and data reflects that parental Cr_2O_3 single phase rhombohedral structure was conformed. Moreover,the crystallite size increases with increase in dopant concentration. The FESEM microimages of samples calcined at 600^oC / 2 hours exhibited the formation of slaty stone like structures with agglomerated nature. The VSM analysis classified the samples to be ferromagnetic in domain and magnetic character increases with increase of Mg concentration in Cr_2O_3 / MgO NCs. The Chromia and 5 % Mg doped Chromia have lesser magnetic behavior whereas, Mg (15%) doped Chromia NCs have better response at higher externally applied magnetic field.

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