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Modifications in Structural and Magnetic Properties of MgO: Cr_2O_3 NCs with rise of Calcination Temperature

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ABSTRACT

The Transition Metal Oxide Nanocomposites' (TMON's) have found wide and elaborated uses in various technological fields due to large surface to area ratio. The Cr_2O_3 and Mg doped Cr_2O_3 nano-composites possess remarkable properties at nano scale with the technological applications in photocatalysis & water treatment, energy storage, sensors and electronic devices etc. In present work, the authors discussed the synthesis of pure Chromia and Mg doped Chromia Nanocrystallines and thereafter, various characterization techniques were employed to examine the various properties of the novel synthesized NCs. The powdered samples were prepared by advanced chemical co-precipitation method and then calcined at 200°C/ 2 hrs, 400°/ 2 hrs & 600°C/ 2 hrs respectively.

The XRD graph showed that Cr_2O_3 rhombohedral structure was in formation and Mg^{2+} ions only replaced Cr^{2+} ions in their parental structure and rise of temperature directly increased the crystallite size of powdered samples ranging from 32.23 nm to 40.38 nm respectively The IR peaks at 480 cm^{-1}, 610 cm^{-1} and 852 cm^{-1} were analogous to O-Cr-O and O-Mg-O vibrations respectively. The Vibrating Sample Magnetometer (VSM) technique was employed to analyze the magnetic properties of the nanocomposites and the M-H graphs revealed that all the calcined samples were ferromagnetic in nature with Mg (10%) doped Cr_2O_3 nanocrystallines having large value of saturation magnetization of 94.965 x 10^{-3} at higher temperatures which may be used in supercapacitors, superconductors and MRI fields.

Keywords: Cr_2O_3 nanocrystallines, calcination, XRD, FTIR, VSM etc.

INTRODUCTION

The properties of metal oxide particulates especially at nanoscales are highly dependent on size, shape and dimensionalities of particulates. Among metal oxides the transition metal oxide nanoparticulates have reported multifunctional properties because of their multi d- orbital subshell electronic configurations[1-3]. The Chromium Oxide nanoparticulates have multiple oxidation states (+2, +3 & +4 etc) depending on the method of synthesis and adhering operating conditions[4-8]. The past study shows that the Cr_2O_3 have key material with known applications in the field of catalysis, color pigment, oxidizing agent which makes it popular in industrial uses whereas, MgO nanoparticulates have enhanced applications in fertilizers, optoelectronics, catalysis and bacterial actions etc[9-10].

The aforesaid properties of Cr_2O_3 and MgO create the inclination of researcher to synthesize Mg doped Cr_2O_3 and conclusively analyze the data of various practical based studies such as XRD, FTIR, VSM and FESEM results. In present work, the versatile advanced co-precipitation techniques were used to synthesize various calcined Mg (10%) doped Cr_2O_3 MONs and then calcined at different temperatures 200° C, 400°C and 600°C for 2 hours respectively. The current study reflects that newly synthesized powdered samples have ferromagnetic behavior over a wide variety of intensity of magnetization, variable retentivity, coercive field, etc. The afiresaid material Mg (10%) doped Cr_2O_3 calcined at 600° C / 2 hrs may be a futuristic material in the technological fields of MRI, super conductors and super capacitors.

Experimentation

Synthesis Techniques

The authors initially declared that the chemicals used in present study were AR in grading and not purified further at laboratory scale. All the powdered samples were operated by domestic microwave oven at different temperatures. The 1.0 M $CrCl_3.6H_2O$ and 0.1 M $MgCl_2.6H_2O$ samples were prepared with doubly de-ionised water (DDI) in refluxing mode. The greenish color solution was highly acidic in nature and neutralizes the same withv2.0 M NaOH solution with dropwise dilution. The greenish precipitates were exhibited at about pH of reaction 9.0 and same was restored for 24 hours for ageing process. The resultant precipitates were collected with filtration process and contaminations were removed with the multiple

washes using ethanol followed by DDI water. The filtered wet cake operated was operated in domestic microwave oven at 100° C for 15 minutes / 2 seatings. The dried cake was distributed in three parts in silica crucibles and then calcined at 200° C, 400°C and 600°C for 2 hours respectively in a muffle furnace. The aggregate various calcined powdered samples were used for further characterization techniques and restored in evacuated sample holders for further futuristic applications.

Instrumentation Used

The structural properties of Chromia and Magnesium doped Chromia samples calcined at various temperatures were studied with the help of **Perkin Elmer X- Ray Diffractometer** having λ = 1.5406 A° with recorded parameter on X-axis as 2θ (in degrees) and intensity on the Y- axis respectively. The magnetic properties of the various samples were analyzed with the Vibrational Sample Magnetometer (VSM) over a wide range of magnetic field H = ±1.5 **T** whereas elemental groups present in the samples were assessed with **Perkin Elmer IR spectrometer**. The morphological study of samples was examined by **JEOL 3600 FESEM microscope**. The comparative analysis and results of Mg doped Cr_2O_3 nanocrystallines calcined at 200°C, 400°C & 600°C for 2 hours respectively and pure Cr_2O_3 calcined at 600°C / 2 hrs have been derived in this paper.

RESULTS AND DISCUSSION

X-Rays Diffraction Data Analysis

The X – Ray diffractometer was used to examine the crystallographic innings of the various calcined samples of Mg doped Cr_2O_3 and pure Cr_2O_3 nanoparticles. The X- Rays generated through CuK_α (λ = 1.5406 A°) were incident over a range of 2θ ≈ 10° to 80° and intensity of transmitted beams were studied on photographic plate in the parameters of 2θ position and the recorded intensity. The analysis of various parameters and results were represented in Table 1 and calculated graphical representation of data was shown in Fig. 1 as under.

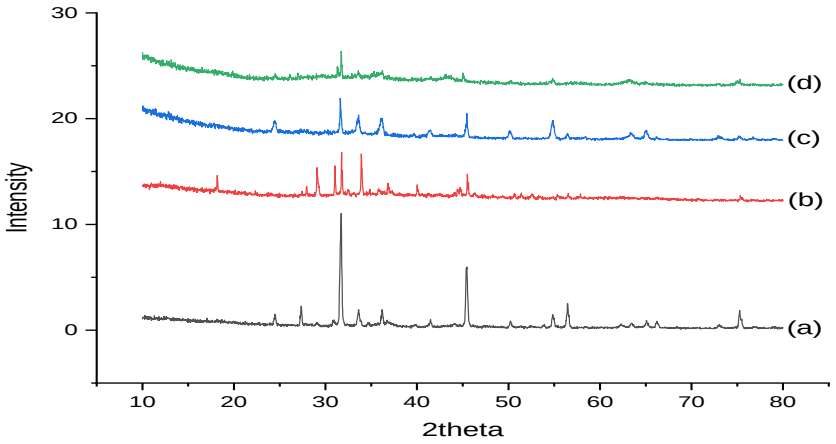


Fig. 1 — XRD Spectrum of calcined samples of (a) Pure Chromia calcined at 600°C and Mg (10%) Nanocrystallites calcined at (b) 200°C , (c) 400°C and (d) 600°C for 2 hours respectively.

The perusal of Fig. 1 shows that peaks were seen at 2θ ≈ 24.5° , 27.9°, 29°, 31.4°, **31.8°**, 33.92°, 36.5°, 45.4°, 54.8° ,56.5°. Among these peaks the bold position represents the Most Intense Peak (MIP) that was taken for further study to calculate the crystallite size of the crystallines. The MIP at 2θ position 31.8° and peaks at positions 24.5°, 33.9° were analogous to **JCPDS card no. 38-1479** and peaks at 2θ positions 36.5°, 45° and 57° were matched with **JCPDS Card No. 45-0946** signifying the formation of single phase parental rhombohedral structure. The average crystallite size (D) was determined by the Debye Scherrer formula, $\mathbf{D = 0.89\lambda / \beta\cos\theta}$,
where all the symbols have their usual meanings.

Table 1 — The Crystallite size of pure Cr_2O_3 and Mg (10%) doped Cr_2O_3 NCs calcined at different temperatures.

Sr. No	Sample Name	FWHM (in rad)	MIP 2θ (in °)	Crystallite size (in nm)
1	Pure Cr NCs at 600° C	0.00428	31.64	32.23
2	Cr/Mg NCs at 200° C	0.00384	31.72	36.12
3	Cr/Mg NCs at 400° C	0.00385	31.84	36.43
4	Cr/Mg NCs at 600° C	0.00343	31.70	40.38

The study of Table 1 depicts an increasing trend in the crystallite size of the pure Chromia and Magnesium doped Chromia with rise in the calcination temperatures (32.23 nm to 40.38 nm) i.e., at for pure Chromia at 600°C the crystallite size was lesser as compared to that at 600°C for Magnesium (10%) doped Chromia. This increase in size accounts to the quantum confinement effect and crystallinity occurred at higher temperatures.

IR Data Analysis

The Infra Red Spectroscopy was used to detect the presence of metal oxides or any other groups present in the calcined samples. The recorded Transmittance Rate was plotted against Wavenumber that ranged from 400 cm^{-1} to 4000 cm^{-1}. The presence of two broad bands at 3306 cm^{-1} and one broad band at 2214 cm^{-1} in spectrum might be due to the hydroxyl group vibration. The sharp peaks seen at 480cm^{-1} , 610 cm^{-1} and 852 cm^{-1} were due to the stretching and bending vibrations in Cr –O –Cr due to temperature increment.

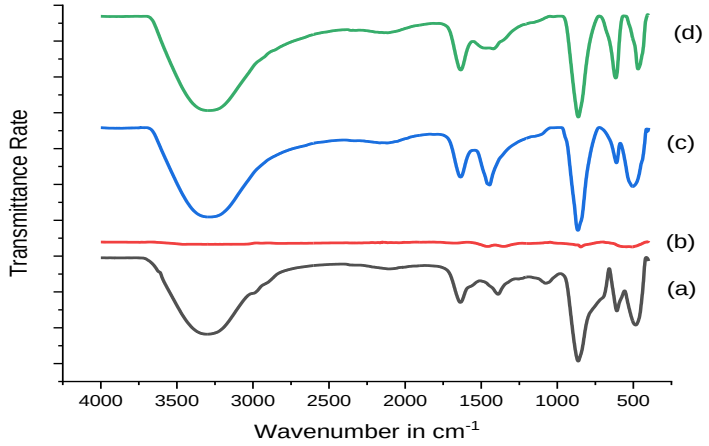
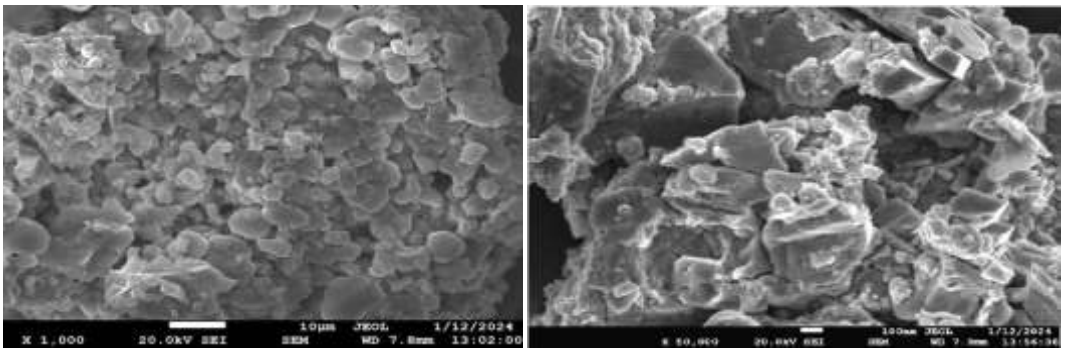


Fig. 2 — IR Spectrum of calcined samples of (a) Pure Chromia at 600° C and Mg (10%) calcined at (b) 200°C, (c) 400°C and (d) 600°C for 2 hours.

FESEM Image Analysis

The pure Chromia sample that was calcined at 600°C for 2 hours was put under the FESEM (Field Emission Scanning Electron Microscopy) technique using 20.0 kV as the operating voltage with a) magnification rate of 1,000 on 1µm and with b) magnification rate of 50,000 on 100 nm micrographic scale as represented in Fig. 3(a) and 3 (b) respectively. The perusal of images concluded that the calcined Mg doped Cr_2O_3 nanoparticulates were 2-D nanoflakes information and irregular lamina like structure.



(a) (b)

Fig. 3 — FESEM image of (a) Mg (10%) doped Chromia and (b) Pure Chromia calcined at 600 °C /2hours

VSM Data Analysis

The magnetic properties of Cr_2O_3 and MgO doped nanoparticulates were examined with the Vibrating Sample Magnetometer (VSM) technique and the data as received from the lab was presented in graphical form in Fig.3 as shown under. The applied field strength was in the range of ± 1.5 **T**.

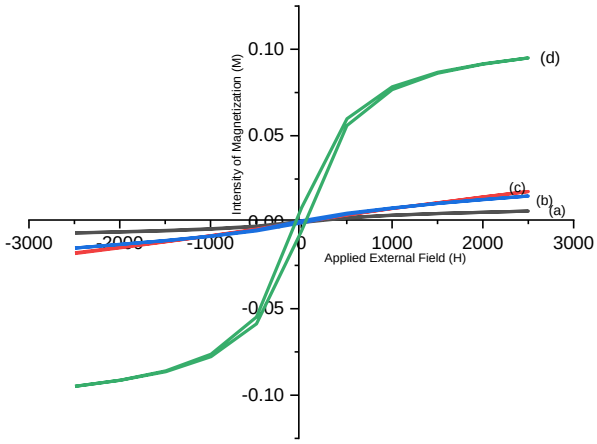


Fig. 4 — The VSM spectrum of (a) Pure Cr_2O_3 and Mg(10%) doped Cr_2O_3 nanoparticles calcined at (b) 200°C, (b) 400°C & (c) 600°C for 2 hours respectively.

The analysis of the VSM graphs shows that the novel material might have ferromagnetic nature with saturation magnetization decreases with increase of calcination temperature. The novel Mg (10%) doped Cr_2O_3 calcined at 600° C / 2 hrs has remarkable enhanced magnetic properties which might be due to the removal of imperfections / crystalline defects at temperature such as 600° C / 2hrs and may have futuristic applications in fields of MRI, super capacitors and super conductors.The parametric evaluation was tabulated in table 2.

Table 2 — The Hysteresis parametric study of (a) pure Chromia calcined at 600°C and Mg (10%) (b) 200°C, (c) 400° & (d) 600° C for 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°C)	6.340 x 10^{-3}	61.764	336.761 x 10^{-6}	0.053	6.340 x 10^{-3}	7.057E^{+0}	2304.36
2	Mg(10%)/ Cr_2O_3 (200°C)	17.939 x10^{-3}	17.942	152.000 x 10^{-6}	0.008	17.939 x 10^{-3}	8.700E^{+0}	2389.54
3	Mg(10%)/ Cr_2O_3 (400°C)	15.046 x 10^{-3}	59.875	631.171 x 10^{-6}	0.042	15.039 x 10^{-3}	8.332E^{+0}	2316.55
4	Mg(10%)/Cr_2O_3 (600°C)	**94.965 x 10^{-3}**	53.703	6.711 x 10^{-3}	0.071	94.956 x 10^{-3}	20.508 E^{+0}	1889.18

CONCLUSION

The various samples of pure Cr_2O_3 and Mg doped Chromia Nanocrystallines have been successfully prepared with microwave irradiated chemical co-precipitation technique and then calcined at different temperatures. The XRD studies of various calcined samples concluded that crystallite size increases with rise of calcination temperature (36.12 nm at 200°C and 40.38 at 600°C) with single phase formation of rhombohedral Cr_2O_3 structures. The IR peaks exhibited at positions 480 cm^{-1}, 610 cm^{-1} and 852 cm^{-1} were used to confirm the O-Cr-O and O-Mg-O lattice vibrations of molecules. The novel Mg (10%) doped Cr_2O_3 calcined at 600° C exhibited superior properties that may be used in MRI, super capacitors and super conductors. FESEM images showed the 2 D nanoflake and irregular lamina like structures of the samples calcined at 600° C / 2 hrs..

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