Synthesis of Cr_2O_3 Nanoparticles by Co-Precipitation Method using Liquor Ammonia as a Precipitant and its Characterization

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Abstract - Chromium oxide (Cr_2O_3) nanoparticles have been produced by co-precipitation method using chromium chloride as the precursor and liquor ammonia as precipitating agent. The resulting sample was characterized by X-ray Diffraction (XRD) method. The average crystalline size of the prepared sample was calculated by Scherrer formula and it was found approximate 23nm. XRD characterization confirmed that chromium oxide nanoparticles were formed with hexagonal crystal structure. The obtained Cr_2O_3 can be used for various applications such as LPG gas sensors, pigments and in industrial areas.

Keywords: Chromium oxide, Co-precipitation, XRD, Hexagonal structure, LPG gas sensor.

I. INTRODUCTION

Nanotechnology delivers a wide effect on various fields like robotics, information, bio-technology, food, agriculture, cosmetics etc. with their different properties & applications [1].

Transition metal oxide exhibits unique physical and chemical properties due to their limited size and high density at corner and edge surfaces [2-7]. There are a number of transitional metal oxides present which are formed by different oxides yet chromium oxide plays a vital role in Nano science and technology. Moreover they exhibit p-type semiconducting nature with a wide band gap (~3.4eV) [3] which is extensively used in optical and electronic devices such as fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings for the passivation of surface against corrosion etc. [2-8]. Chromium oxide in its bulk form shows antiferromagnetic properties with magnetic ordering temperature of 307K [3]. But the nanoparticle of chromium oxides behaves like a ferromagnetic material due to the presence of uncompensated surface spins [8].

It has been reported that chromium oxides nanoparticles can be prepared using different synthesis techniques such as precipitation-gelation, co-precipitation, sol-gel process, sonochemical method, hydrothermal process, microwave plasma method, laser induced deposition, biological method etc.[2,4,9]. A lot of parameters such as pH, temperature, pressure etc. should be controlled to obtain nano crystalline chromium oxide. Among all the above methods precipitation found to be is easy and more accurate process since it is easy to control such parameters [2]. Crystal structure of chromium oxide nanoparticles is hexagonal in nature in which the chromium is present at the corner of surface and oxygen present on the center of surface.

Chromium oxide Nanoparticle are characterized using X-ray Diffraction (XRD), UV-Vis Spectroscopy, Scanning Electron Microscope (SEM), Energy-Dispersive X-ray Spectroscopy (EDS), Thermo Gravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Photoluminescence (PL), and Transmission Infrared Spectroscopy (TEM) to find various structural, optical, and morphological properties.

Metal oxide nanoparticles can be synthesized using many precipitating agents such as urea, NaOH, KOH, Ammonia etc. It has been reported earlier that Chromium oxide nanoparticles can be synthesized with the help of urea and ammonia [2, 5]. In this paper chromium oxide nanoparticles are been prepared by us using liquor ammonia as precipitating agent for a temperature of 50°C and for pH of 12.

II. PROPOSED METHODOLOGY

2.1 Chemicals used

Chromium (III) chloride (hexahydrate), Ammonium Hydroxide (liquor ammonia), Distilled water were used throughout the experiment.

2.2 Synthesis of chromium oxide nanoparticles

For retrieved precipitates, 0.2 M of chromium chloride was mixed in 100ml of distilled water and kept in magnetic stirrer for 30 minutes to get a homogenous solution. Liquor ammonia was added drop wise with a constant stirring until the pH of the solution reaches 12 at the temperature 50°C. The as prepared sample was washed several times (3-5) using distilled water with the help of centrifuge machine. The obtained precipitates were then dried in hot air oven at 70ºC for 24 hours and were calcinated at 550ºC

in muffle furnace about 5-6 hours. Prepared sample was meshed by mortar & pestle.

2.3 Material characterization

The prepared sample was investigated by Bruker D8 Advance XRD using Cu-Kα (0.154 Aº) radiation. The average crystalline of the prepared sample was found by Scherrer formula.

III. RESULT AND DISCUSSION

XRD was used to determine crystalline size and lattice parameter of the prepared sample. Fig 1. shows the XRD pattern of synthesized chromium oxide nanoparticle having 2□ values of 24.51º, 33.58º, 36.17º, 41.50º, 50.18º, 63.48º, 65.05º. Apart from the above values there were some other peaks which show that the sample contained some impurities.

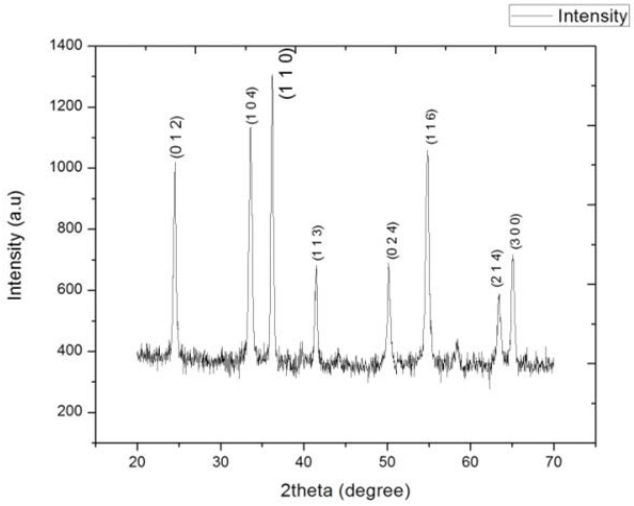

Fig. 3.1 XRD pattern of Cr_2O_3 powder synthesized at 50°C

The sharp peaks in the XRD pattern show that the obtained chromium oxide nanoparticles are of single phase. The crystalline size can be determined by using Debye-Scherrer formula [2].

$$D=\frac{\square\square}{\square\square\square\square}$$

Where, D is the average crystalline size, k is the Scherrer constant (0.89Aº), □ is the wavelength of X-Ray (0.154 A°), B is the full width half maximum of the diffraction, □ is the Bragg's angle. Thus the average crystalline size obtained is of 23nm.

Table 1 shows the miller indices of the respective 2θ values along with its crystalline size. The lattice parameters of obtained sample are a=4.94 and c=13.61 which are approximately matching with the original data of jcpds card no-850869 (a=4.95 and c=13.58). This reveals that the chromium oxide nanoparticles obtained are of hexagonal crystal structure with romobohedral phase.

Table 1. Crystalline size and lattice parameter of the prepared chromium oxide nanoparticles

θ_2	MILLER INDICES	CRYSTALLINE SIZE
24.51º	12	24nm
33.58 º	104	18nm
36.17º	110	26nm
41.5 º	113	22nm
50.18º	24	22nm
54.85 º	116	23nm
63.48º	214	27nm
65.05 º	300	49nm

IV. CONCLUSION

Hexagonal structured Chromium Oxide nanoparticle (Cr2O3) have been synthesized using Co-precipitation method. The prepared sample was then calcinated at a temperature of 500ºC for 5 hours in order to improve the phase formation and crystallinity. The prepared sample was analyzed using X –Ray Diffraction technique and its average crystalline size found was about 23nm. The obtained Chromium oxide nanoparticles can be used in many applications such as LPG gas sensor, Pigments fuel cells and other electronic devices. This study provides an easy and large production of Chromium oxide nanoparticles with fewer impurities and these impurities can be removed by calcinating the material for long time period.

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