

Facile Synthesis and Electrical Properties of Chromium Oxide Nanoparticles

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Abstract

The synthesis of Cr_2O_3 nanocrystal has been done by chemical precipitation method using ammonia as a precipitating agent. The hexagonal crystal structure and single crystalline nature of Cr_2O_3 powder were verified by X-ray diffraction (XRD) analysis. Debye-Scherrer formula was utilized to ascertain the average size of crystallites. The structure, shape, size, and composition of Cr_2O_3 nanoparticles were probed by SEM (Scanning Electron Microscopy) and EDX (Energy Dispersive X-ray Spectrometry). Optical band gap energy was estimated by UV-Vis spectroscopy. The various functional groups in Cr_2O_3 nanoparticles were reckoned by FTIR (Fourier transform infrared spectroscopy) findings. Inductance, Capacitance, and Resistance (LCR) meter was used to ascertain the electrical behavior of Cr_2O_3 nanoparticles. These finding suggests that the synthesized nanocrystal may be used in development of the electrical and optoelectronic devices.

Keywords: Cr_2O_3 nanoparticles, X-ray diffraction (XRD), SEM, LCR.

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Introduction

Semiconductor metal oxide nanostructures are highly enviable for nanotechnological research despite of their peculiar structural geometry because of their large surface area and smaller size. This class of compounds often exhibit new and exciting features compared to their coarse-grained equivalent [1]. Chromium (III) oxide (Cr_2O_3) is one such metal oxide that is found in nature as an Eskolaite mineral. It is also called dichromium trioxide, or chromia, or chromium sesquioxide possessing a wide band gap and comes under the category of p-type metal oxide nanostructures [2]. G. Carta et al. found that chromium oxide (Cr_2O_3) is the hardest metal oxide with low friction coefficients [3]. Cr_2O_3 crystallizes into a rhombohedral structure having space group R-3c with lattice constants $a = b = 4.953 \text{ \AA}$ and $c = 13.578 \text{ \AA}$ [2]. G. Yasmeen and Jaswal et al. found that it may also crystallize into hexagonal shape [4, 5]. M. M. Abdullah et al. showed the ac conductivity of the Cr_2O_3 nanoparticles in the high frequency region and explained it according to the frequency power law. Cr_2O_3 nanoparticles can be used for applications such as pigment production, catalysts, solar energy collectors, thermal coating materials, optical storage materials, liquid crystal

displays, and hydrogen storage [2,4]. This paper reports the synthesis and characterization of Cr_2O_3 nanoparticles via the facile chemical precipitation method. The characterizations were done by XRD, FTIR, UV-Vis spectroscopy, SEM and LCR.

Synthesis of Chromium Oxide

In a veritable synthesis process, 6g of chromium nitrate nonahydrate $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was dissolved in 100 ml of double distilled water (DDW). Drop by drop aqueous ammonia was added to the above solution along with the constant stirring. This was continued until the pH of the solution reached to 12 and the green precipitate was formed. The obtained precipitate was stirred on the magnetic stirrer at 80°C until it was burned out in ashes. The obtained residual ash was crushed and further calcined in a muffle furnace at 750°C for 4 hrs and then was again ground to attain a fine powder.

Results and Discussion

XRD Analysis

The X-ray diffraction spectrum of synthesized Cr_2O_3 nano

powder is demonstrated in Figure 1. The distinct and intense peaks reveals that the prepared Cr_2O_3 nanoparticles is of high crystalline nature. The pattern matches with the standard JCPDS card number 38-1479 [3]. The XRD pattern does not contain any extra peaks other than Cr_2O_3 nano powder peaks showing that synthesized nano powder possesses a high degree of purity. These patterns also confirm the hexagonal structure of Cr_2O_3 nano powder with crystal parameters $a = b = 4.9477 \text{ \AA}$ and $c = 13.4534 \text{ \AA}$ and matches well with earlier studies [2]. In XRD pattern the peak broadening elucidate that small nanocrystals are present in samples. The intensity of the peak, its position, width, full-width at half-maximum (FWHM), and crystallite size were ascertained by using Powder X software. The experimental values of (d) are in a good agreement with the values which is reported in the previous studies [2]. Debye-Scherrer formula was used to evaluate the mean crystallite size (D) [6] and came out to be 25.12 nm.

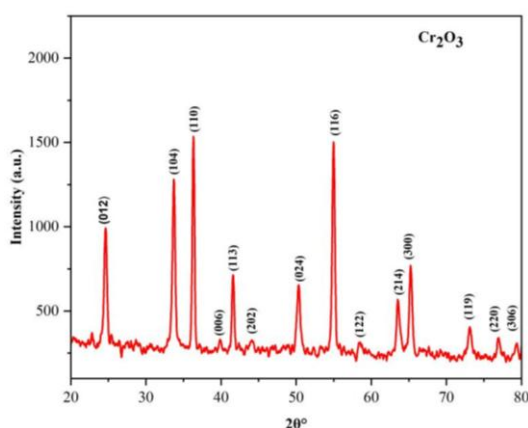


Figure 1: XRD pattern of Cr_2O_3 nanoparticles.

SEM and EDX Analysis

The surface structure of the produced Cr_2O_3 nanoparticles were examined by using a SEM microscope.

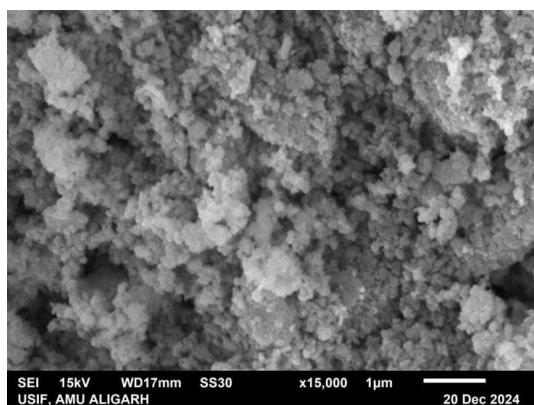


Figure 2 (a): SEM micrograph of Cr_2O_3 nanoparticles.

Figure 2 shows that all of the chromium oxide particles are having spherical shape and possesses a high degree of

agglomeration. The synthesized sample own good porosity as is clear from the micrograph. The shape of Cr_2O_3 's spherical and agglomerating behaviour is in good agreement with the earlier research article [7]. Figure 2 (b) confirms the elemental composition of Cr_2O_3 through EDX spectra.

FTIR Analysis

FTIR technique in the range of $4000\text{--}500 \text{ cm}^{-1}$ was used to ascertain the chemical composition and different functional groups (Figure 3). Generally, interatomic vibrations in metal oxides are characterized by the peaks below 1000 cm^{-1} . The broad bands at 3411 cm^{-1} and 1629 cm^{-1} owed to O-H stretching vibration because of adsorbed water from air and hydroxyl group.

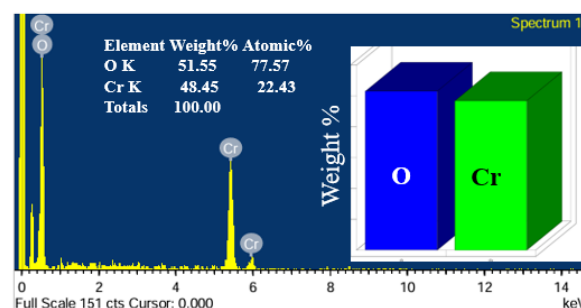


Figure 2 (b): EDX of Cr_2O_3 nanoparticles showing the elemental composition.

The C-O absorption of the Cr_2O_3 surface is depicted by the peaks at 1347 and 1182 cm^{-1} . The peaks at 1045 , 639 , and 563 cm^{-1} are the signature peaks of crystalline Cr_2O_3 nanoparticles. The band at 639 cm^{-1} is the identification of the chromium oxide nanoparticles as the Cr_2O_3 phase and the peak at 560 cm^{-1} characterizes the Cr-O distortion vibration [8]. The FTIR spectrum along with the XRD analysis confirm the successful creation of Cr_2O_3 nanoparticles.

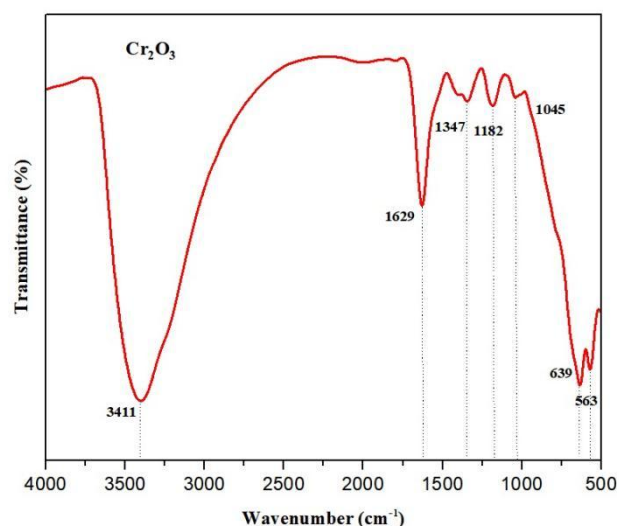


Figure 3: FTIR spectrum of Cr_2O_3 nanoparticles.

Optical Properties

Perkin Elmer Spectrophotometer was utilized to attain the absorption spectrum of Cr_2O_3 nanoparticles which reveals a strong absorption peak at 404 nm (Figure 4 (a)) confirming the d3 transition of chromium ion. A weak shoulder peak is also present at 480 nm [9]. This spectrum was further utilized to estimate the optical direct band gap (Figure 4 (b)), with the help of Tauc relation [10]. The obtained value of band gap energy with the value of 4.21 eV is in good agreement of the wide band gap nature of Cr_2O_3 nanoparticles.

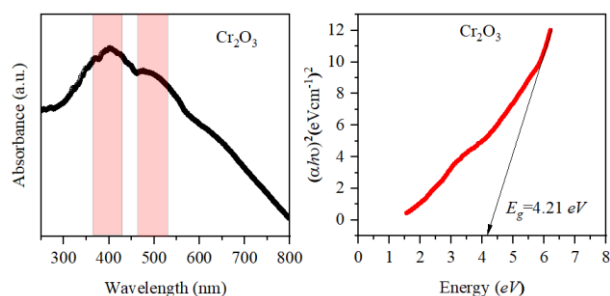


Figure 4: (a) Absorption spectrum and (b) band gap of Cr_2O_3 nanoparticles.

Electrical properties of Cr_2O_3 nanoparticles

Dielectric study was performed by LCR (Inductance Capacitance and Resistance) meter. Figures 5 (a to e) shows the frequency dependent behaviour of Cr_2O_3 nanoparticles with dielectric constant, loss, ac conductivity, and impedance. Both real and imaginary part of dielectric constant shows a decrease and a constant behaviour at high frequency value (Figures 5 (a) and (b)).

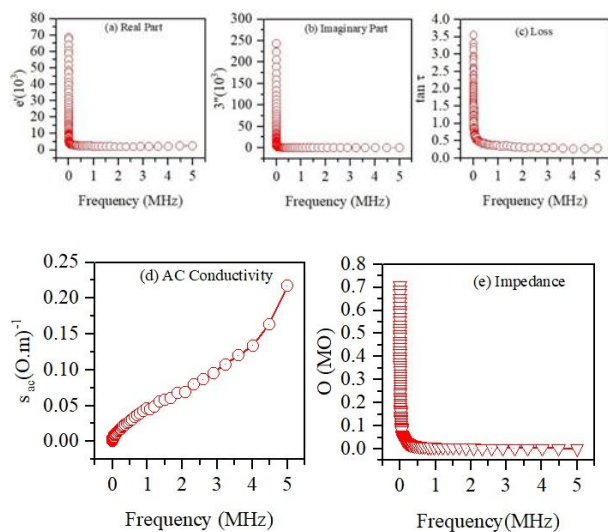


Figure 5: (a-e) Electrical behaviour of Cr_2O_3 nanoparticles.

High value at low frequency may occur due to the interfacial or space charge polarization caused by porosity and grain structure. The porosity of the prepared nanoparticles is

clearly visibly for SEM image also. Decreasing trend may be due to the dielectric relaxation whereas the constant nature may be attributed to the fact that electric dipole freezes through the relaxation process.

Figure 5 (c) shows the decrease in dielectric loss with the increasing frequency and may be due to the marginal resistance of the grain. Figure 5 (d) indicates the increasing trend with increasing frequency and clearly suggest the good ac conductivity of Cr_2O_3 nanoparticles [11]. Thus, the results show that Cr_2O_3 nanoparticles have significant dielectric dispersion at low frequencies due to polarization effects and improved conductivity with reduced impedance at higher frequencies, making them appropriate for high-frequency applications. Cr_2O_3 is showing an identical frequency-dependent dielectric behavior with other transition metal oxides, such as CoO, NiO, and ZnO [12]. The dielectric constant (ϵ') in metal oxide nanoparticles generally shows a significant drop with increasing frequency due to the inability of dipoles to keep up with rapid field oscillations. Studies on Cr_2O_3 nanoparticles have reported high dielectric values at low frequencies, primarily due to interfacial polarization, followed by a sharp decline as frequency increases, aligning with the Maxwell-Wagner polarization model [11]. Thus, the prepared Cr_2O_3 nanoparticles exhibits similar dielectric and electrical properties as to other transition metal oxides with a minor variation in values reported across different studies that may be due to the differences in synthesis methods, particle size, and processing conditions. Overall, our experimental results align well with literature reports, further validating the frequency-dependent behaviour of Cr_2O_3 nanoparticles.

Conclusions

The facile precipitation approach of creating Cr_2O_3 nanoparticles utilizing ammonia and $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ as a chromium source has been presented in this work. The high crystalline character of the Cr_2O_3 lattice with a hexagonal phase is supported by the XRD examination, which also demonstrates the notable pure synthesis of Cr_2O_3 nanoparticles. The sample's spherical shape and aggregation are shown by its morphology through SEM. EDX confirms the elemental composition. Functional groups in Cr_2O_3 nanoparticles were detected by FTIR spectra. The band gap energy of 4.21 eV shows a wide band gap nature of prepared sample. Dielectric and ac conductivity studies shows that Cr_2O_3 nanoparticles may turn up a meriting candidate in electrical and optoelectronic applications.

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