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Optical, Structural, Morphological Properties of Chromium (III) Oxide Nanostructure Synthesized Using Spray Pyrolysis Technique

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Abstract

Nanostructure of chromium oxide (Cr_2O_3-NPs) with rhombohedral structure were successfully prepared by spray pyrolysis technique using Aqueous solution of Chromium (III) chloride $CrCl_3$ as solution. The films were deposited on glass substrates heated to 450 °C using X-ray diffraction (XRD) shows the nature of polycrystalline samples. The calculated lattice constant value for the grown Cr_2O_3 nanostructures is a = b = 4.959 Å & c = 13.594 Å and the average crystallize size (46.3-55.6) nm calculated from diffraction peaks, Spectral analysis revealed FTIR peak characteristic vibrations of Cr-O Extended and Two sharp peaks present at 630 and 578 cm^{-1} attributed to Cr-O “stretching modes”, are clear evidence of the presence of crystalline Cr_2O_3. The energy band gap (3.4 eV) for the chromium oxide nanostructures was measured using the UV-VIS-NIR Optical Spectrophotometer. It was found that by scanning electron microscopy (SEM) and image results, there is a large amount of nanostructure with an average crystal size of 46.3-55.6 nm, which indicates that our synthesis process is a successful method for preparing Cr_2O_3 nanoparticles.

Article Info.

Keywords:
Cr_2O_3, Nanostructure, spray pyrolysis, optical properties, structure properties.

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1. Introduction

The study of microstructure and nanostructure has received increasing attention because of the new properties that materials may exhibit when reducing grain size [1]. Over the past decades, much progress has been made in the manufacture of nanostructure. "Nanomaterials", especially Transitional metal oxides have an important role in materials science, physics and chemistry as well as technological applications [2]. Metal oxides are commonly used in the manufacture of sensors, electronic circuits, fuel cells, and Coatings for corrosion-resistant surfaces and as a catalyst [3]. Nanostructure of metal oxides can have unique chemical properties due to their specific size and high density of edge surface locations [3,4]. Among the inorganic nanostructure, chromium nanostructure (III) (Cr_2O_3) has got a lot of attention due to their wide applied fields, including pigment [5],"heterogeneous catalysts" [6,7]. Coating materials for the purpose of thermal protection [8,9], biological applications [10,11], digital recording system [12], photonic and electron ic devices [13,14]. Various techniques were developed for the assembly of Cr_2O_3 nanostructure such as hydrothermal [15], Solid pyrolysis [16], combustion [17], sol-gel [18], precipitation gelation [19], oxidation chromium [20], laser-induced deposition [21], mechanochemical reaction and subsequent heat Treatment [22], and "sonochemical methods" [23]. Many preparation methods, such as: sol-gel technology, "laser induced deposition", "hydrothermal reduction", chemical-mechanical reaction, "condensation–polymerization", "gas condensation", solid pyrolysis, homogeneous precipitation with the help of urea, microwave plasma,"sono-chemical reaction", thermal treatments, nano-casting method, hydrazine reduction and solution-combustion synthesis were used to prepare Cr_2O_3 nanostructures [24]. Most of these techniques are cost-effective, complex, require high temperature, environmentally sensitive and special laboratory equipment. Therefore, for the current study, a typical hydrothermal method was used to synthesize Cr_2O_3 nanostructures due to the fact that this preparation method reduces cost and is straightforward at low temperature.

In this paper a simple and important method for the synthesis of nanosized Cr_2O_3 chromium oxide by spray pyrolysis technique in nano is described. The surface properties, size, morphology and crystallographic structure of Cr_2O_3 particles are characterized by means of X-ray diffraction (XRD), and scanning electron microscope (SEM) which will give much valuable information about these materials. In addition, optical properties of chromium (III) oxide (Cr_2O_3) nanostructure which determined using the UV-VIS-NIR Optical Spectrophotometer.

2. Experimental work

An aqueous solution of Chromium (III) chloride $CrCl_3$ (Sigma-Aldrich Labor) was prepared by dissolving 0.15M of "chromium chloride" in 100 ml in "distilled water" with constant moving for 45 minutes. In order to keep the pH value of the solution at 10, we added a few drops of "ammonium hydroxide" to the solution while moving. Chromium (III) chloride react with water to produce hydrogen chloride and chromium (III) oxide using compressed air as a carrier gas [25].

$$2CrCl_3 + 3H2O \text{ [temp]} = 6\ HCl + Cr_2O_3$$

The precursor solution was transformed into an aerosol by an ultrasonic nebulizer operating at a frequency of 1.7 MHz, which is connected to the precipitation chamber via a spray nozzle where the precipitation process takes place inside the chamber on a glass substrate at a temperature of (450 ± 5) ° C, it has been maintained, the nozzle-substrate distance at 7cm where the spraying continues for five minutes.

In this part of the experiment, thin films of chromium oxide were prepared use the aerosol generated in the ultrasonic nebulizer was injected into the stainless-steel chamber through a nozzle as shown in Fig.1with deposition time (5 min) to get required layer of Cr_2O_3 thin film. The film prepared from Cr_2O_3 was green in color and had good adhesion to glass substrates as it was tested by visual inspection.

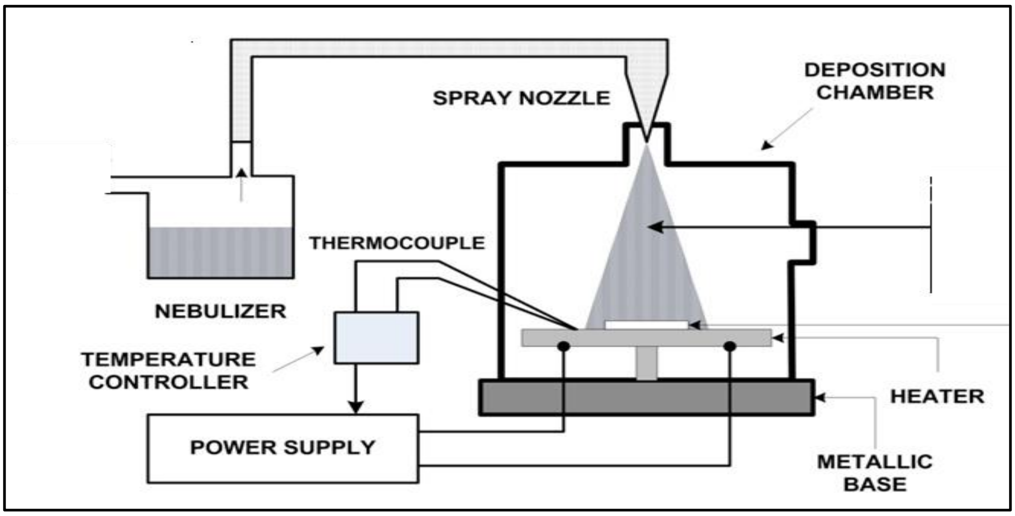

Figure 1: Schematic diagram for system set up.

3. Results and discussion

3.1. X-Ray and FTIR studies

The X-ray diffraction was carried out on "a Philips Analytical XPERT". Using a diffractometer "Cu Kα radiation (λ = 1.54056Å) with a "MINIPROP detector", It works at 40 Kv and 30 mA. It was recorded X-ray diffraction patterns between 2θ =10◦ to 80◦. shows the XRD pattern (Fig. 2), it can be observed peaks that belong to the crystalline phases of Cr_2O_3 (012), (104), (110), (113), (024), (116) and (300). All peaks can be set to Cr_2O_3 stage according to data released by the "ICDD (International Center for Diffraction Data) cards", No. card (00-038-1479). The average crystallite sizes were calculated using the Scherrer equation $D=K\lambda/(\beta\cos\theta)$ where, λ = 1.54056 Å is the wavelength of an X-ray, K = 0.9 is the "Scherer constant" β is (FWHM) and "θ" is the "Bragg diffraction angle". The calculated lattice constant value for the grown Cr_2O_3 nanostructures is a = b = 4.959 Å & c = 13.594 Å whereas reported [26]value for lattice constant is a=b=4.953 Å & c=13.578 Å. The recorded and calculated values of the lattice constants agree well. Table (1) shows the size of the grains for the prepared sample, which is agreed with the report [27].

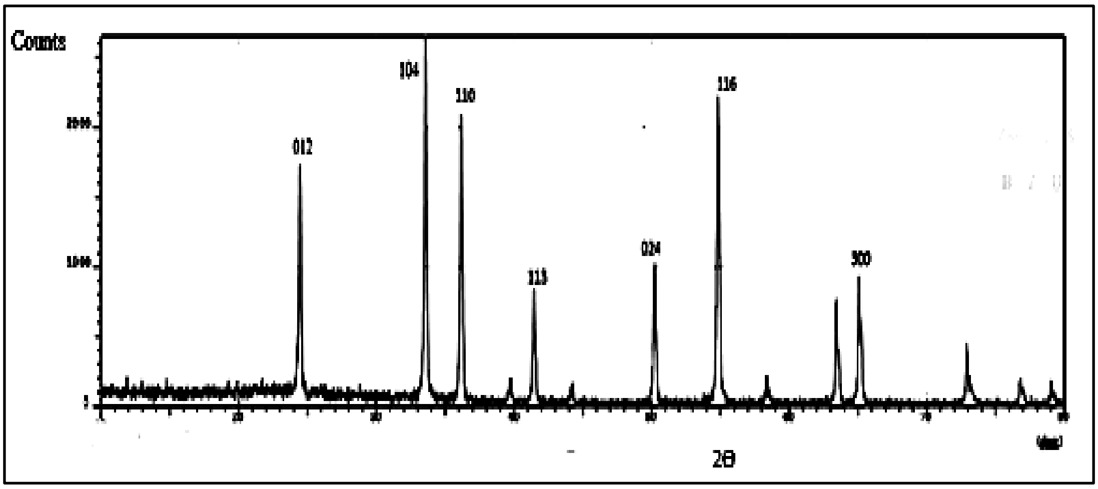

Figure 2: XRD model of Cr_2O_3 nanostructure prepared by spray pyrolysis.

Table 1: Experimental and standard values of peaks, and grain size of Cr_2O_3 sample prepared with SP technique.

2θ (deg)	*dhkl (Exp.) (Å)*	*dhkl (Std.) (Å)*	*hkl*	*FWHM (deg)*	*G.S (nm)*
24.5055	3.62965	3.63132	012	0.167	46.354
33.5954	2.6656	2.66533	104	0.1651	47.958
36.1976	2.47959	2.4796	110	0.1575	51.524
41.4719	2.17561	2.1752	113	0.6950	50.4
50.2167	1.81533	1.81521	024	0.1609	49.677
55.2476	1.66133	1.67237	116	0.1433	55.662
65.0980	1.43173	1.43157	300	0.1738	46.354

Figure 3 shows the "Fourier-transform infrared" (FTIR) spectra of chromium oxide (Cr_2O_3) sample prepared by SP technique. As it can be observed, at a frequency of 3420 cm^{-1}, where the broad band, it compatible with the "stretching modes" of OH groups. Generally, Cr_2O_3 absorption bands appear below 1000 cm^{-1} due to "inter-atomic vibrations". Two sharp peaks present at 630 and 578 cm^{-1} attributed to Cr-O "stretching modes", are clear evidence of the presence of crystalline Cr_2O_3 [28].

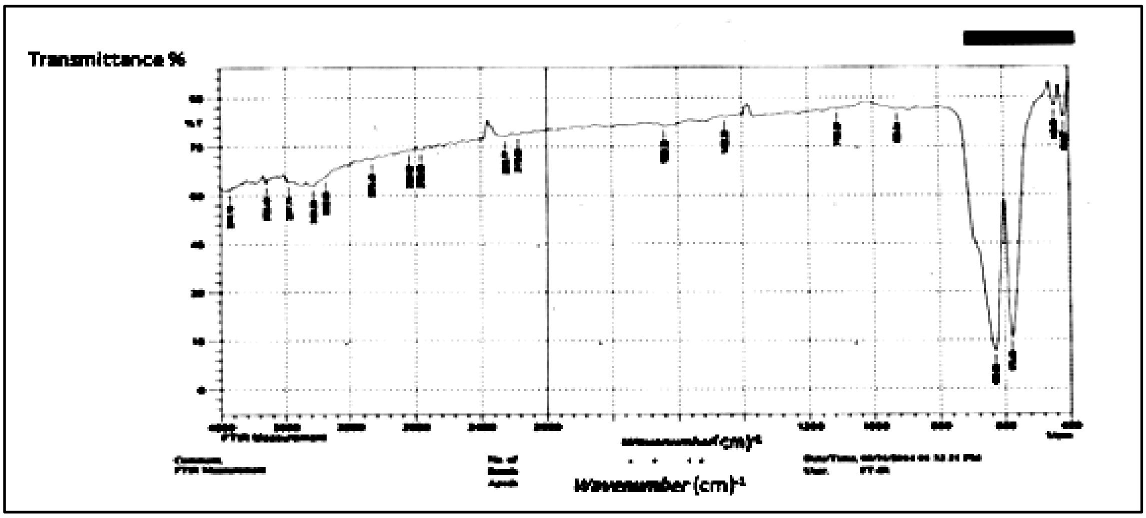

Figure 3: FTIR spectrum of the nano-sizedCr2O3 prepared by SP technique.

3.2. Morphological analysis

The morphology of Cr_2O_3 nanostructure prepared with SEM images was distinguished as shown in Fig. 4. From the images results, it can be noticed that a large amount of nanostructure (NPs) with an average crystallite size of 46.3-55.6 nm, which indicates that our synthesis process is a successful method to prepare Cr_2O_3 nanoparticle.

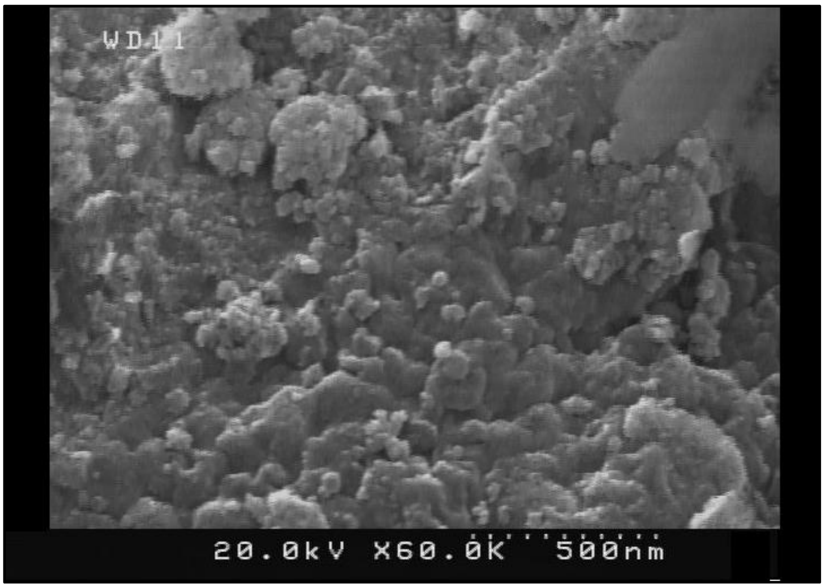

Figure 4: SEM image of Cr2O3 sample obtained at precursor concentration of 0.1 5M by SP.

3.3. Optical analysis

Figure 5 shows the "optical absorption spectrum" of the Synthesized Cr_2O_3 nanostructure. The spectrum shows the generic direction of absorption, i.e., reduced absorption of material with a decrease in the frequency of incident radiation. Figure 6, shows the alteration of $(h\upsilon\alpha)^2$ with photon energy ($h\upsilon$) for the synthesized Cr_2O_3 thin film of thickness 170 nm, the film thickness was measured by a thin film measuring system (Stellar Net Inc. Thin Film measurement systems), which works on the principle of spectroscopy. The plot shows the direct band gap of ~3.4 eV, which is agreed with the report [29].

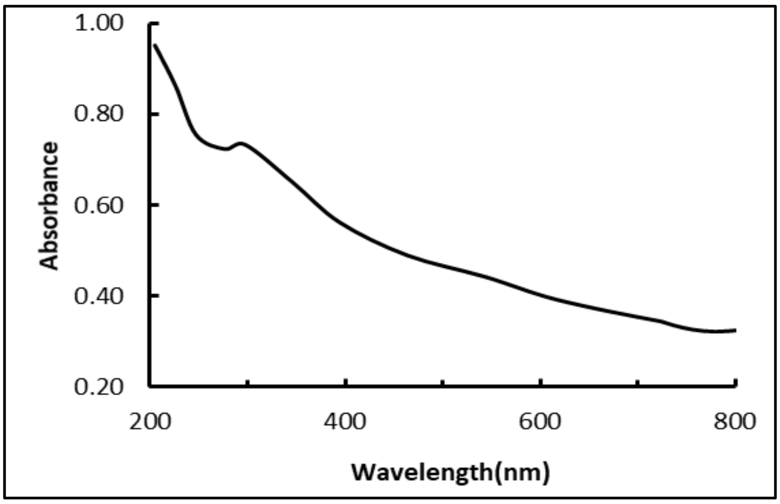

Figure 5: UV-VIS-NIR spectrum typical for Cr2O3 thin film prepared by SP technique.

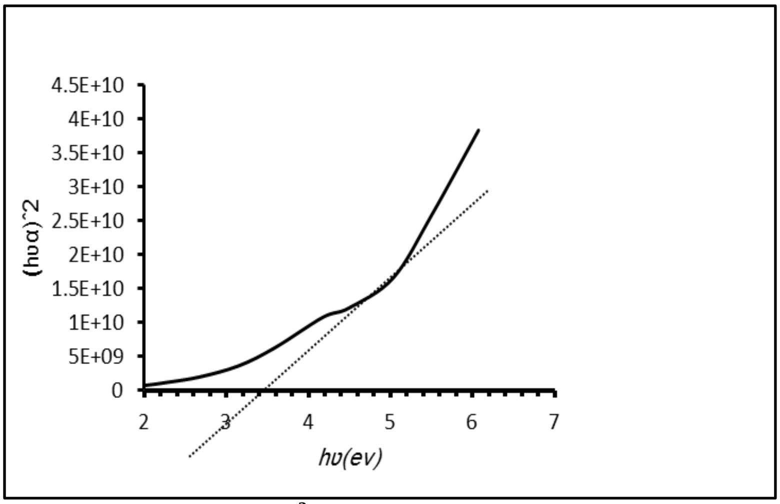

Figure 6: Variations of $(\alpha h\nu)^2$ with photon energy (hν) for Cr_2O_3 thin film of thickness 170 nm.

4. Conclusions

Using spray pyrolysis technology, a thin film of chromium oxide nanostructure was fabricated using simple chromium chloride materials as primers. The results were obtained by XRD, FTIR, SEM, and UV-VIS. spectrometers. Identical and specific crystal phase (Cr_2O_3), bonding (Cr-O), purity (Cr, O) and energy bandgap (3.4 eV) of Cr_2O_3 nanostructures. The average crystallizes size (46.3-55.6) nm calculated from diffraction peaks indicating the formation of a nanostructured layer.

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Conflict of interest

We certify that we have NO affiliations with or involvement in any organization or entity with any financial interest.

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الخصائص البصرية والمورفولوجية والهيكلية للبنية النانوية لاوكسيد الكروم المصنعة باستخدام تقنية الانحلال الحراري

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الخلاصة

تم تحضير االهيكل النانوي لأكسيد الكروم (Cr_2O_3-NPs) ذات البنية المعينية بواسطة تقنية الانحلال الحراري بالرش باستخدام محلول مائي من كلوريد الكروم ($CrCl_3$), تم ترسيب الأفلام على ركائز مسخنة إلى 450 درجة مئوية ,تم تشخيصها باستخدام حيود اشعة اكس (XRD) الذي يظهر ان النماذج االمحضرة ذات طبيعة متعددة التبلور قيمة ثابت الشبكة المحسوبة للبنى النانوية Cr_2O_3 المزروعة هي $a = b = 4.959$ Å & $c = 13.594$ Å ومتوسط حجم التبلور كان بحدود(46.3-55.6)نانومتر والمحسوب من قمم الحيود .كما ان التحليل الطيفي لمقياس فورييه لتحويل الاشعة تحت الحمراء(FTIR)كشف الاهتزازات المميزة لذروة Cr-O الممتدة . وتوجد قمتان حادتان عند 630 و 578 سم$^{-1}$ تُعزى إلى "أوضاع التمدد" Cr-O ، وهي دليل واضح على وجود Cr_2O_3 البلوري. تم قياس فجوة نطاق الطاقة (3.4 الكترون فولت) للبنية النانوية لأكسيد الكروم باستخدام مقياس الطيف الضوئي UV-VIS-NIR. لقد وجد أنه عن طريق مسح المجهر الإلكتروني (SEM) ونتائج الصور ، هناك كمية كبيرة من البنية النانوية بمتوسط حجم بلوري يبلغ 46.3-55.6 نانومتر ، مما يشير إلى أن عملية التوليف لدينا هي طريقة ناجحة لإعداد الجسيمات النانوية Cr_2O_3.