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Preparation of chromium(III) oxide sub-micron powders by solid state method without fuel

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Abstract

A solid state synthesis of Cr_2O_3 Sub-micron powders were reported by using $[Cr^{III}(acac)_3]$ by combustion synthesis without fuel. , The powder x-ray diffraction technique was used To investigate the phase formation. Also, FESEM micrographs were used to investigate the morphology of the obtained materials. It showed that the morphology of the obtained materials was in the form of porous and characterized in terms of particle size materials. The optical properties of the obtained materials were studied by FTIR spectra. According to the PXRD data, it was found that at an annealing temperature of 1000 °C, the main phase of the obtained materials was hexagonal crystal structures with cell parameters a = b = 4.95876Å and c = 13.5942 Å.

Keywords: Cr_2O_3, Solid State, Combustion, Acetylactone, Porous, sub-micron powders

1. Introduction

Nano powders with particles of uniform shape and narrow size distribution lying in the nanometer range have been shown to possess interesting properties. Nanoparticles of Cr_2O_3 are widely applied in fields such as refractories and as a chromium source for $C_{r3}C_2$ synthesis [1] doping in varistor compounds [2], wearing [3,4] gas sensing [5], pigments [6] catalysis[7], electric [8] and magnetic properties [7–10]. Numerous methods to synthesize nanostructured Cr_2O_3 have been developed, e.g. arc discharge and annealing [9], microwave plasma[10], mechanochemical [11], combustionreaction[12], template-based thermal decomposition [13], hydroxide precipitation and decomposition [8,14], hydrothermal[5,15] and sol–gel [4,16]. It is necessary to find an economical process which can be used to prepare them on a large scale. In fact, there have been already a lot of ways to prepare Cr_2O_3 nanoparticles, including precipitation-gelation process [17], gas condensation [18], sonochemical reaction [19], microwave plasma [20], decomposition of chromium nitrate solution [21], laser-induced deposition [22], but most of them have difficulties in being scaled up due to the more complex processes or more expensive reaction apparatus. A better process should be employed to meet the demands of industrialization. The aim of the present article is the investigation of preparation of Cr_2O_3 nanoparticles by a novel solution combustion method using a complex without using fuel.

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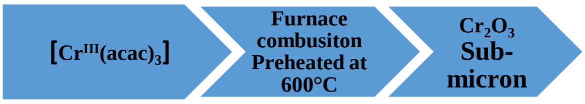

Scheme1. Formation of sub- micron particles

2. Experimental procedure

2. 1. Materials and Methods

All chemicals were of analytical grade, obtained from commercial sources and used without further purification. Phase identifications were performed on a powder X-Ray diffractometer Siemens D5000 using Cu Kαradiation (λ=1.542Å). The FESEM (Hitachi, model S-4160) was used to investigate the morphology of the synthesized materials.

2.2. Synthesis of complexes [Cr^{III}(acac)$_3$]

0.7 g of chromium (III) chloride hexahydrate ($CrCl_3$ $6H_2O$) dissolved in 25 mL distilled water. Then 3 g urea in 3 or 4 steps was added to the green chromium solution. Then 1.75 mL of acetylacetone was dded to the solution in drop-wise. The final solution in a beaker was kept in a boiling water bath and heated while stirring for one hour. The solution should initially be very dark and almost black in appearance, but as the reaction performed, deep maroon plate-like crystals formed as a crust on the surface of the reaction mixture. The solution was cooled and the obtained crystals were vacuum-filtered and a product was dried in air.

2.3. Synthesis procedure of Cr_2O_3 micro-size layer

A certain amount of the synthesized complex was powdered and transferred into a 25 mL crucible. Then the crucible was transferred into a preheated oven at 1000 °C and was annealed for 3h. After the reaction completed, the crucible was cooled to the room temperature normally. The obtained powder was collected for further analyses.

3. Results and discussion

The FTIR spectrum of Cr_2O_3 is shown in figure 3. Metal oxide Cr_2O_3 generally reveal absorption bands below 1000 cm^{-1} due to inter-atomic vibrations. The Two sharp peaks displayed at 640 and 574 cm^{-1} attributed to Cr-O stretching modes, are clear evidence for the presence of the crystalline Cr_2O_3 [26].

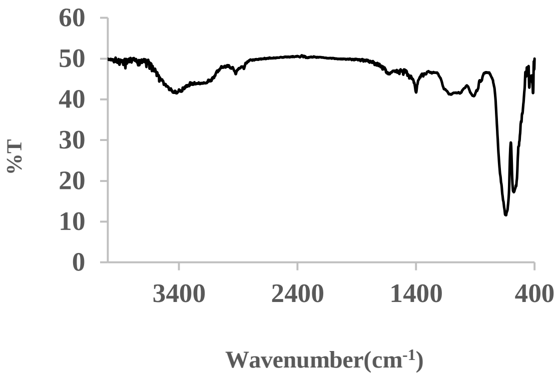

Figure 8. FTIR spectrum of the synthesized Cr_2O_3 Sub-micron via a combustion method without fuel at 600°C.

3.1 PXRD analysis

Figure 1a, b and c show the XRD patterns of the Cr_2O_3 sub-micron prepared by complex [Cr(acac)$_3$] after annealing at 1000 °C. Inspection of the results revealed that annealing of Cr_2O_3 at 1000 °C resulted in the formation of a main phase of Hexagonal (JCPDS no. 38-1479 with a = b 4.95876 Å, b = 13.5942 Å and space group R3^{-}c) and minor phases of simple-cubic (JCPDS no. 06-0532 with a = b = 8.36 Å and space group Face-centred) and rhombohedral (JCPDS no. 06-0139). During the preparation of the nanoparticles, it is very difficult to avoid impurity phases. For example, CuO nanoparticles could be contaminated by Cu_2O [32]. In the main phase the major peaks were indexed as (012), (110), (104), (113), (024), (116), (214), and (300) [17]. The average crystallite size was estimated by Scherrer equation using the most intense peak (3 1 1):

$$d = K\lambda/(\beta\cos\theta) \qquad \text{(Equation 1)}$$

where d [nm] is the mean size of the crystalline domain (crystalline size) of the synthesized materials, K is the shape factor (dimensionless) which has the typical value of 0.9 (but depends on the shape of particles), λ [nm] is the X-ray wavelength, β [rad] is the peak width at halfof the maximum intensity (FWHM) and θ [rad] is the peak position (Bragg angle). K=0.9, and λ=0.15418 nm were used to calculate the mean crystalline size (d) of the Cr_2O_3 sub-micron. Based on this equation, the average crystallite size was calculated as ~400 nm.

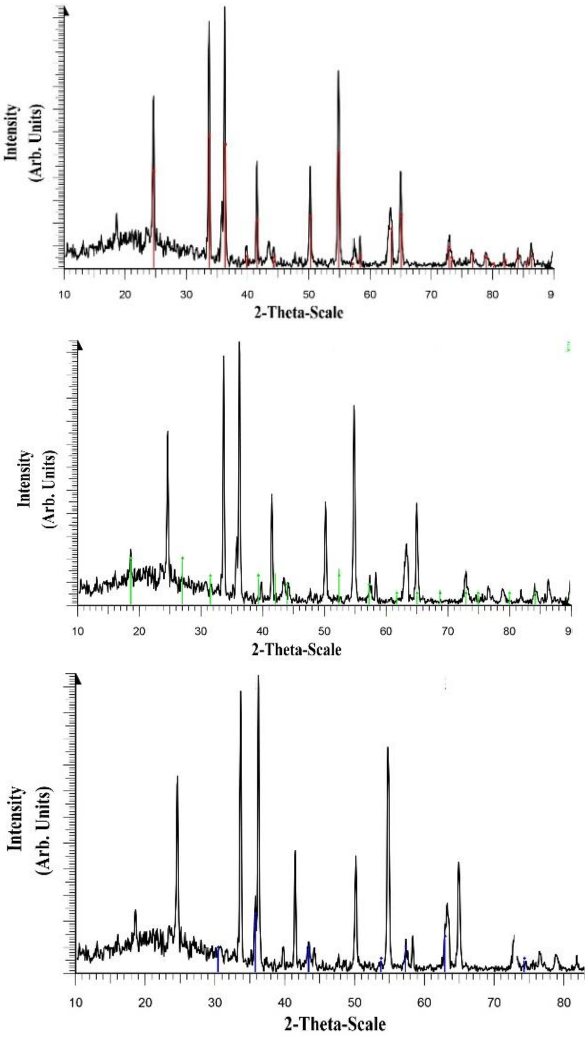

Fig. 1. PXRD pattern of the synthesized Cr_2O_3 Sub-micron via a combustion method without fuel at 1000 °C.

3.2. Morphology analysis

Figure 3 shows the FESEM images of the synthesized Cr_2O_3 sub-micron via a combustion method without using fuel annealed at 1000 °C for 4 h. Figure 4a shows the low magnification image of the synthesized material. It is clear that the sample is in a porous like structure. It shows that the porosity distribution is nearly homogeneous. Figure 4b shows that the porosity is in micro-porosity range structure. It shows that the porosity diameter is in a range of about 60-80 nm. Figure 4 c and d shows the high magnification image of the synthesized submicron-materials. It is clear that there are small particles as uncus on the synthesized materials. It shows that the particles sizes are in a range of about 40-200 nm in diameter.

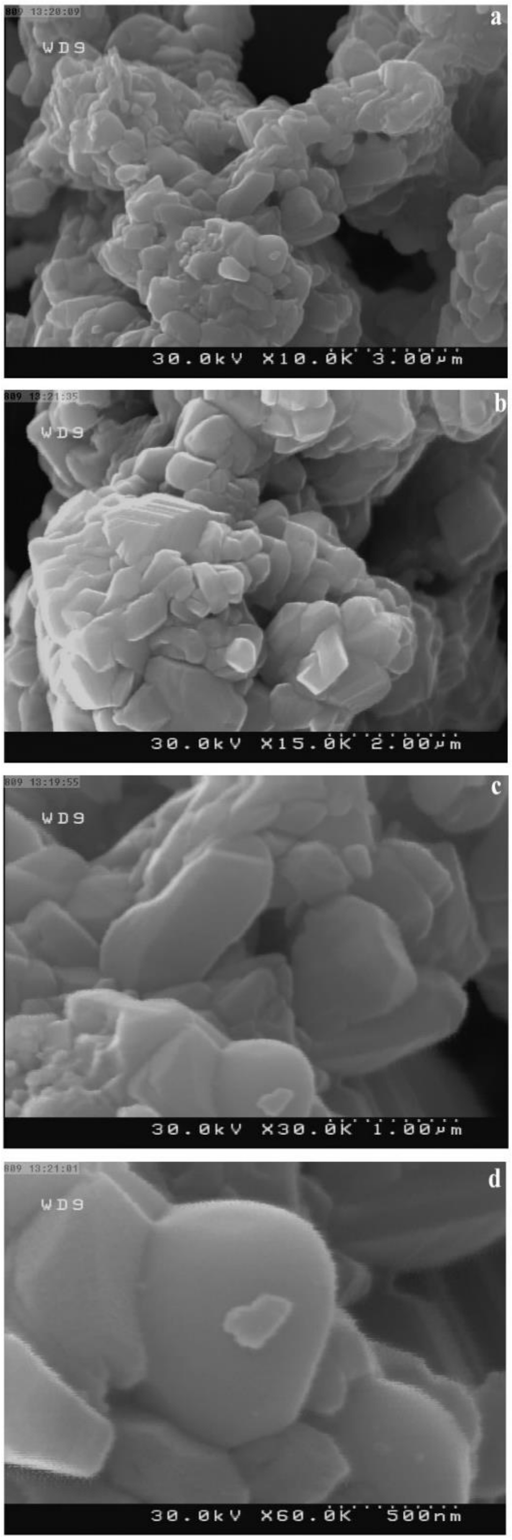

Figure 4. SEM images of the obtained Cr_2O_3 from the combustion method without using fuel annealed at 1000 °C for 4 h.

4. Conclusion

Synthesis of Cr_2O_3 submicron-materials was performed. PXRD data showed that the main phase for the synthesized submicron-materials was Hexagonal. FESEM images showed that the synthesized materials were in particle and porous morphologies. FTIR spectrum of the synthesized material was investigated.

Acknowledgments

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