Journal of Applied Chemical Research, Special issue, 47-53 (2015)

Journal of Applied Chemical Research
www.jacr.kiau.ac.ir

Synthesis of Chromium Oxide Nano Particles by Thermal Decomposition Method

Bagher-Aziz kalantari[1], **Mohamad Reza Talei Bavil Olyai**[*2]

[1]*Department of Chemistry, Faculty of Science, Islamic Azad University, Karaj Branch, Alborz, Iran*

[2]*Department of Chemistry, Faculty of Science, Islamic Azad University, South Tehran Branch, Tehran, Iran*

(Received 23 Nov. 2015; Final version received 27 Dec. 2015)

Abstract

Disclosed is a method of preparing pigmentary chromium oxide nano particles having 50 percent particle size less than about 100 nm. Preparation of nano chromium (III) oxide is investigated by thermal decomposition method. According to the disclosed method, a substantially dry solid composition of potassium dichromate and carbon active is heated in CO_2 atmosphere to a temperature of about 600ºC for 1hour. Thereafter, the solid Cr_2O_3 product was washed twice with distilled water.

Keywords: *Green Chromium Oxide, Nano Particles, Color Performances, Particle Size.*

Introduction

Among inorganic nanoparticles, chromium oxide (Cr_2O_3) nanoparticles have received great attention due to its numerous application domains, including green pigments [1] heterogeneous catalysts [2-4] coating materials for thermal protection [5-6], hydrogen storage [7-9], digital recording system [10], photonic and electronic devices [11-12]. Various techniques have been developed to synthesize Cr_2O_3 nanoparticles such as precipitation, precipitation gelation, sol gel, mechanochemical reaction, oxidation of chromium in oxygen and sonochemical methods [13]. But since either these processes are complex or their reaction apparatus are expensive, most of them have difficulties in being industrialized. Some new methods of preparation should be explored.

In the present study, a simple and original method for the synthesis of green chromium oxide nano particles is described. The surface properties,

---

***Corresponding author:** *Mohamad Reza Talei Bavil Olyai, Department of Chemistry, Faculty of Science, Islamic Azad University, Karaj Branch, Alborz, Iran. E-mail: mr_talei@azad.ac.ir.*

size, morphology and crystallographic structure of Cr_2O_3 particles are characterized by means of X-ray diffraction (XRD), scanning electron microscope (SEM) which will give much valuable information about these materials. However for the determination of particle distribution, was used from laser particle sizer (LPS) Fritsch Gmbh, the model of Analyssett 22. The use of nanoparticles can improve the pigment performance: such as the coloring performance of ceramic pigments depends on both optical properties, which are expected to improve in nanoparticles, and chemical stability, since the dissolution rate in glazes, is expected to increase with surface area of pigment. The best compromise in conventional ceramic pigments is usually found with particle size distribution in the 1-10µm range [1]. However, ceramic nano-pigments (10-80 nm) behaved satisfactorily in preliminary printing tests on ceramic tiles, developing intense colors in a wide range of firing temperature [1]. In this work is compared nano pigment chromium oxide on color intensity of the ceramic tile decoration than micro pigment chromium oxide.

Experimental

All reagents were purchased from Aldrich or Merck and were used without further purification. Experimental procedure for chromium oxide (Cr_2O_3) nanoparticles is briefly summarized in Fig.1. According to the disclosed method, particulate potassium dichromate, having a particle size of about 10microns, is admixed with particulate bone charcoal, having particle size of about 10 microns, at a mole ratio 0.5 mole of carbon per total moles of potassium dichromate .The resulting mixture is the heated to a temperature of about 600ºC under a carbon dioxide atmosphere at about one atmosphere total pressure with 1ml/sec rate for 1 hours. In the quenching process, the water solubilizes the sodium or potassium compound such as $M_2Cr_2O_7$ and M_2CrO_4 where M is as defined above. The resulting solid, Cr_2O_3, is then separated from the soluble alkali metal compounds under vacuum with twice elution by distilled water. The pigmentary Cr_2O_3 having 50 percent particle size less than about 100 nm is recovered.

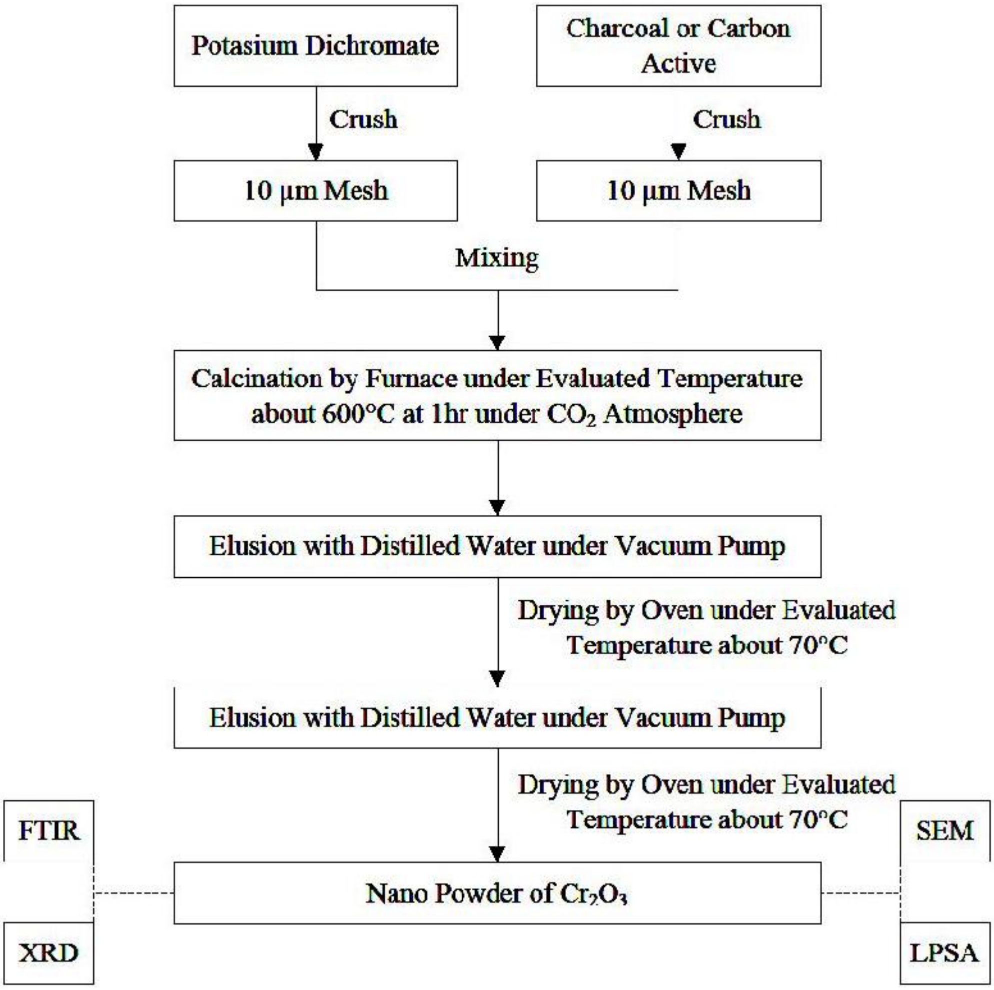


Figure 1. Experimental procedure for the formation of chromium oxide (Cr_2O_3) nanoparticles.

Results and discussion

In this work, nano chromium oxide (Cr_2O_3) prepared with thermal method. The overall reaction scheme is shown below:

$$K_2Cr_2O_7 + 2C \rightarrow K_2CO_3 + CO + Cr_2O_3$$

where charcoal or carbon active is used as carbon source. For preparation of nano chromium oxide, it is necessary the grain size of potassium dichromate and carbon are less than 10µm. However reaction is carried out under a CO_2 gas atmosphere.

In order to procedures, purity of chromium oxide nanoparticlesis shown with Fig.2 that indicate high purity of the components.

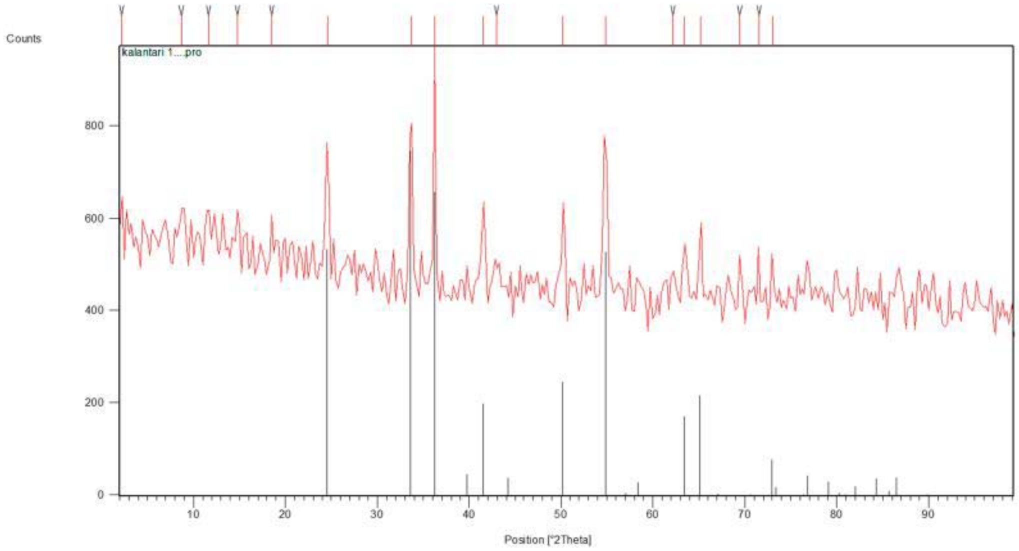


Figure 2. XRD pattern of the Cr_2O_3 nano particles.

The FTIR spectrum of chromium oxide nanoparticles is shown in Figure 3. Two sharp peaks displayed at 566 and 626 cm^{-1} attributed to Cr-O stretching modes, are clear evidence for the presence of the crystalline Cr_2O_3.

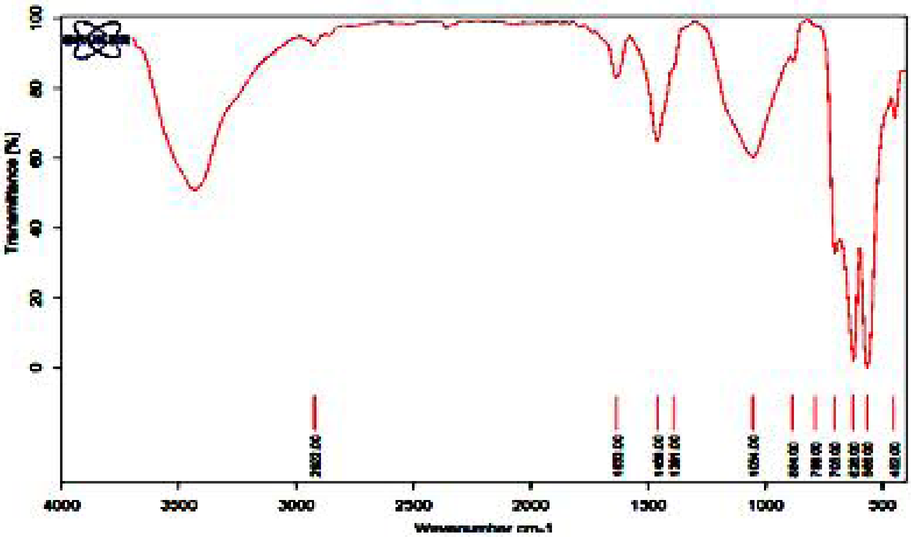


Figure 3. FTIR spectrum of the nano-sized Cr_2O_3.

In order to characterize the nature of the Cr_2O_3 nanoparticles SEM, XRD and Laser Particle sizer measurements were carried out. Fig.4 shows SEM images of the Cr_2O_3 powder. The Cr_2O_3 powder shows agglomeration of particles, with diameters ranging from 60-200nm.

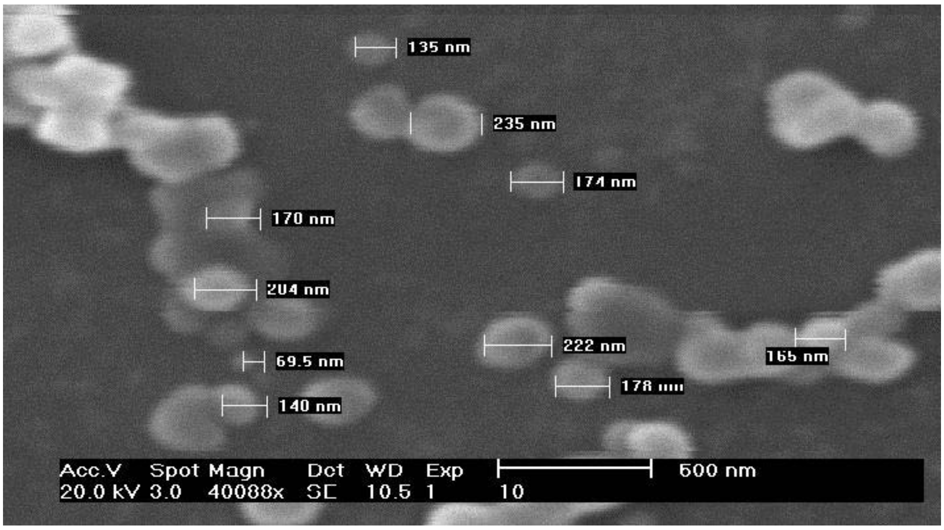
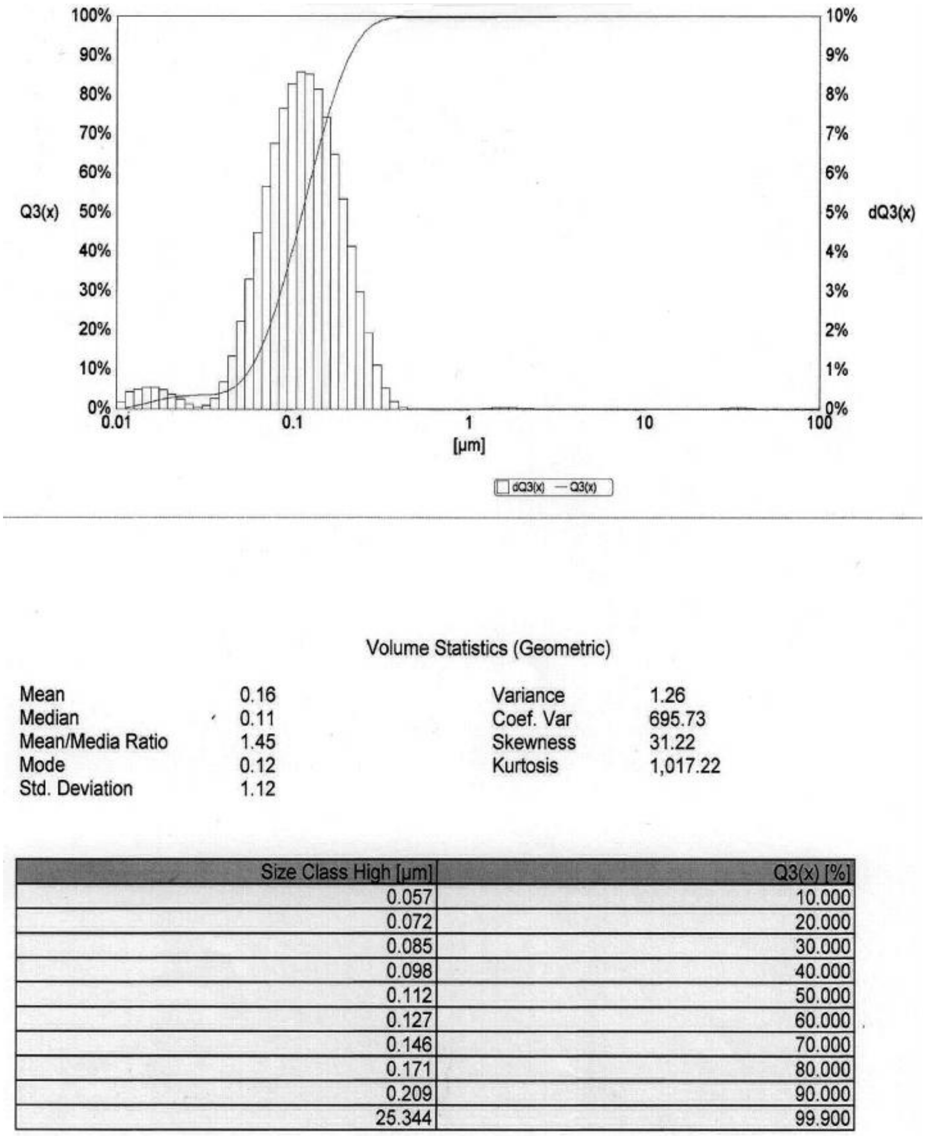


Figure 4. SEM image of the Cr_2O_3 nano particles.

From image results, we can observe a large quantity of uniform nanoparticles (NPs) with average particle size (60-200nm), indicates that our synthesis process is an easy method for the preparation Cr_2O_3 nanoparticles. The diffraction patterns are shown in the insets. The peaks of XRD patterns displayed are evidence for the presence of the crystalline Cr_2O_3 with high purity for the powder because of it is cantabile with the peaks of instance sample. For the determination of particle distribution, was used from laser particle sizer (LPS) that the results is shown Figure 5.



Volume Statistics (Geometric)

Mean	0.16	Variance	1.26
Median	0.11	Coef. Var	695.73
Mean/Media Ratio	1.45	Skewness	31.22
Mode	0.12	Kurtosis	1,017.22
Std. Deviation	1.12		

Size Class High [µm]	Q3(x) [%]
0.057	10.000
0.072	20.000
0.085	30.000
0.098	40.000
0.112	50.000
0.127	60.000
0.146	70.000
0.171	80.000
0.209	90.000
25.344	99.900

Figure 5. Particle distribution of Cr_2O_3 nano particles by LPS.

Results is observed that the particle distribution of chromium oxide nanoparticles are 50 percent less than 100 nm that is compatible with results of SEM images.

Conclusion

We have described the use of a thermal decomposition method for preparation chromium oxide nano particles that its main advantage is easily controllable conditions with using low cost chromium source is merit to be considered for scaling up by industrial researchers. These nanoparticles can use easily as pigment in the color of the ceramics.

Acknowledgments

We would like to acknowledge to the Javad Rafiei and Karvaneh for helping us in the measuring of samples using XRD and we are grateful to Islamic Azad University, Karaj Branch for financial support of this work.

References

[1] P.M.T. Cavalcante, M. Dondi, G. Guarini, M. Raimondo, G. Baldi, *Dyes Pigments*, 80, 226 (2009).

[2] (a) T.V. Malleswara Rao, E.M. Zahidil, A.Sayari, *J. Mol. Catal. A*, 301, 159 (2009). (b)Sh. Deng, H. Li, S. Li, Y. Zhang, *Journal of Molecular Catalysis. A, Chemical*, 268, 169 (2007).

[3] (a) G. Dabrilaite-Kudzmiene, S. Kitrys, *Material Sciences*, 19, 89 (2013). (b) T.V. Rao, Y. Yang, A. Sayari, *J. Mol. Catal. A*, 301, 152 (2009).

[4] G. Wanga, L. Zhang, J. Deng, H. Dai, H.He, C. Tong, *Appl. Catal. A,* 355, 192 (2009).

[5] X. Pang, K. Gao, F. Luo, Y. Emirov, A.A.Levin, A.A. Volinsky, *Thin Solid Films*, 517, 1922 (2009).

[6] X. Hou, K.L. Choy, *Thin Solid Films*, 516, 8620 (2008).

[7] R. Vijay, R. Sundaresan, M.P. Maiya, S.Srinivasa Murthy, *J. Alloys Compd.*, 424, 289 (2006).

[8] A. Patah, A. Takasaki, J.S. Szmyd, *Int. J. Hydrogen Energy*, 34, 3032 (2009).

[9] J.L. Bobet, S. Desmoulins-Krawiec, E.Grigorova, F. Cansell, B. Chevalier, *J. Alloys Compd.*, 351, 217 (2003).

[10] M.D. Bijker, J.J.J. Bastiaens, E.A. Draaisma,L.A.M. Jong, E. Sourty, S.O. Saied, J.L. Sullivan, *Tribol. Int.*, 36 227 (2003).

[11] X. He, D. Antonelli, *Angew. Chem. Int. Ed.*, 114, 222 (2002).

[12] X. He, D. Antonelli, *Angew. Chem. Int. Ed.Engl.,* 41, 214 (2002).

.