https://doi.org/10.30799/jnst.271.19050420 J. Nanosci. Tech. - Volume 5 Issue 4 (2019) 820–821

ISSN: 2455-0191

Share Your Innovations through JACS Directory

Journal of Nanoscience and Technology

Visit Journal at http://www.jacsdirectory.com/jnst

Synthesis and Characterization of Chromium Nanoparticles by Thermal Method

Jyoti Chaudhary[1], Giriraj Tailor[1], Deepak Kumar[2], Deepshikha Verma[3], Ajit Joshi[3,*]

[1]*Department of Polymer Science, M.L.S. University, Udaipur – 313 001, Rajasthan, India.*
[2]*Department of Chemistry, IIT, Jodhpur – 342 001, Rajasthan, India.*
[3]*Department of Chemistry, Mewar University, Chittorgarh – 312 901, Rajasthan, India.*

ARTICLEDETAILS

Article history:
Received 23 May 2019
Accepted 11 June 2019
Available online 13 June 2019

Keywords:
Monoclinic
Chromium Nanoparticles
Morphology Study

ABSTRACT

Monoclinic chromium nanoparticles were synthesized by thermal decomposition method using $CrCl_3$ as precursor. This is simple but fast and effective. The synthesized nanoparticles were analyzed by infrared spectroscopy (IR) and nuclear magnetic resonance spectroscopy (NMR), scanning electron microscopy (SEM), and X- ray diffraction (XRD) techniques. The SEM analysis confirms the spherical shape morphology and presence of stress on the surface of the nanoparticles. The XRD analysis proves that the crystal structure is monoclinic and the size of particles are 45.20 nm.

1. Introduction

Nanoparticles of chromium are important technological material which show novel electronic, thermal, optical, mechanical and chemical properties. These properties are significantly different from those of bulk materials because of large surface to volume area and extremely small size. Transition metals play an important role in many areas of chemistry, physics, material science and electronic engineering. Chromium nanoparticles have wide industrial applications as pigment [1], resistance [2-3], electric [4] materials, gas sensors [5], catalysts [6] and advance colorants [7]. There are so many synthesis techniques available for chromium nanoparticles such as precipitation, hydrothermal [8-10] mechano-chemical process [11], gas condensation, combustion [12] and urea assisted homogeneous precipitation [13,14]. A better process should be implored to meet the demands of industry. In this research nanostructure chromium (III) nanoparticles were synthesized easy, and fast technique involving the water-based complexation of polymer and chromium metal salt followed by thermal decomposition. The idea behind preparation of nanocomposite is to use there on building blocks with dimensions in nanometer range to design and create new materials with unpresented flexibility and improvement in their physical properties.

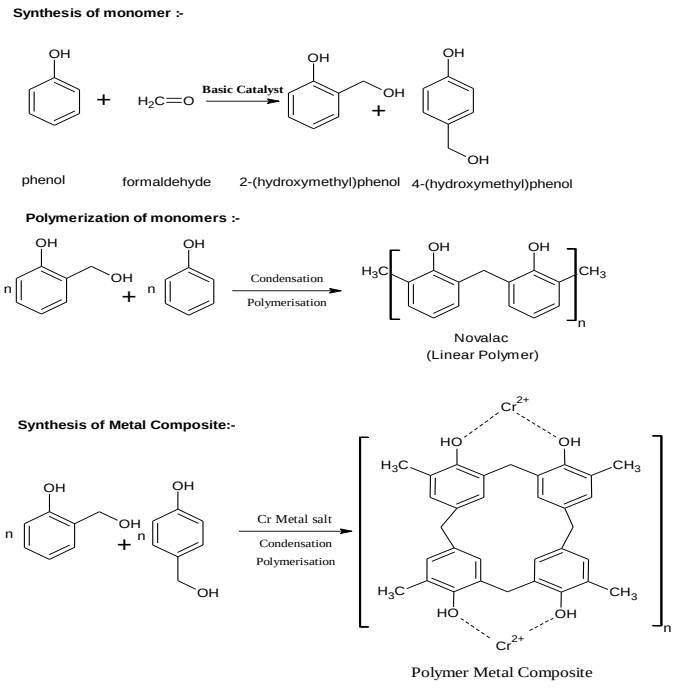
2. Experimental Methods

2.1 Materials

Important chemical like chromium (III) chloride hexahydrate $CrCl_3.H_2O$ (Central Drug House (P) Ltd), formaldehyde HCHO (Central Drug House (P) Ltd.), phenol C_6H_5OH (Central Drug House (P) Ltd.), glacial acetic acid CH_3COOH (Fisher Scientific), hydrochloride acid HCl (Fisher scientific), distilled water used to prepare composites were of chemically pure grade. Metal solution was prepared by dissolving appropriate amount of its chloride salt in distilled water.

2.2 Synthesis of Polymer Composite (Reaction Scheme)

Three-necked round bottom flask equipped with stirrer and reflux condenser is charged with (1.38 moles) of phenols and (1.13 moles) of formaldehyde solution and 15 mL of glacial acetic acid. The mixture is agitated thoroughly and is allowed to cool off. Then drawn off and the remaining water is removed by slowly raising the temperature to 50 °C and applying a vacuum by means of water pump. This temperature is maintained until sample from the melt, which solidifies on cooling and forms a rigid, pink color solid. Further 15 mL of 1 N chromium salt solution added and the mixture is shaken for 5 minutes. The reaction is exothermic in nature. The dried solid sample has been purified by the washing with distilled water solution. The excess metal ion and impurities on the sample were removed by washing. The procedure was followed as per Scheme 1.



Scheme 1 Synthesis polymer metal composite

2.3 Synthesis of Chromium Nanoparticles

The synthesis of nanoparticles is done by calcination method. The above polymer metal composite is allowed to decompose at 900 °C for 45 minutes. Black colored particles are obtained.

*Corresponding Author:ajitjoshi2k2@gmail.com(Ajit Joshi)

https://doi.org/10.30799/jnst.271.19050420

Cite this Article as: Jyoti Chaudhary, Giriraj Tailor, Deepak Kumar, Deepshikha Verma, Ajit Joshi, Synthesis and characterization of chromium nanoparticles by thermal method, J. Nanosci. Tech. 5(4) (2019) 820–821.

2.4 Purification

Step-1: Removal of Volatile Impurity: At the time of decomposition many volatile impurities got separated and nanocomposite is free from such impurities.

Step-2: Removal of Metallic Impurities: Metallic ions were removal from the nanocomposite by keeping it in 12 N hydrochloric acid solution for 24 hours. The mixture was centrifuged and washed with distilled water till hydrochloric acid was completely removed [15].

3. Results and Discussion

The analysis of polymer composites by NMR spectra reveals existence of three type of protons in the sample (Fig. 1). Peak at 3.524 ppm shows phenolic proton in compounds, peak at 2.471 ppm represents that the benzylic proton presence of polymer composite and the one at 6.744 ppm is indicative of aromatic proton present in polymer composite. Bunch of peaks at 6.744 ppm, 3.695 ppm, 2.471 ppm indicate that the complex formed was a polymerized substance. Fig. 2 is the IR spectra of the substance and Table 1 represent the peaks and the corresponding functional group in the composite.

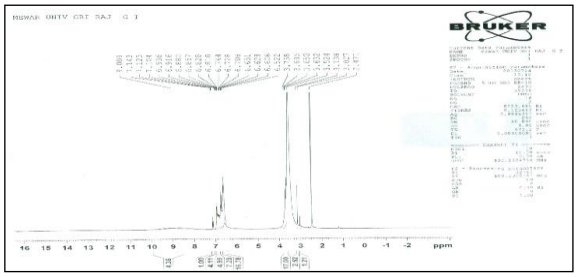


Fig. 1 NMR spectra of chromium polymer composite

Table 1 IR spectra of polymer composite

Prominent absorption band cm^{-1}	Functional Group
3323 cm^{-1}	O-H stretching (broad peak)
3010-2899 cm^{-1}	C-H stretching
1705-1645 cm^{-1}	C-O stretching

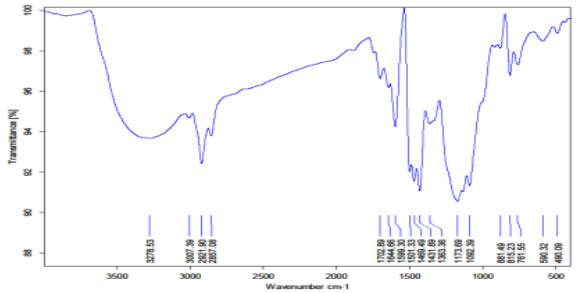


Fig. 2 IR spectra of chromium polymer composite

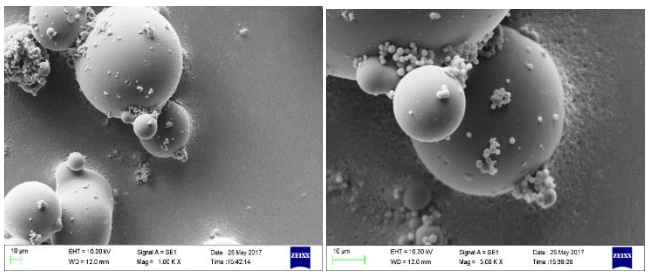


Fig. 3 SEM image of chromium nanoparticles

The morphology of the resulting powder was examined by scanning electron microscope. Fig. 3 shows that the morphology same is as the unrecompensed sample. The small spherical like structure present on the surface is due to the presence of metal ion on the surface of nanomaterial. The analysis of mechanical properties shows that toughness is much more than 1.5 TPa which is about 20 times of steel. The spherical metal ions present on the surface of composite material plays an important role in the one-dimensional electron transportation and confirmation of the electrical properties of compound.

X-ray diffraction pattern stipulate an effective method for determining the phase and crystalline size of chromium nanoparticles. The monoclinic chromium nanoparticles have been determined using XRD technique. The particle size evaluates by Debye Scherrer formula, $D = K\lambda/\beta\cos\theta$, where D is the mean size of crystallites, K is crystallite shape factor a good approximation is 0.9, λ is X-ray wave length, β is full width at half maximum (FWHM) in radians of X-ray diffraction peak and θ is the Bragg angle. By applying Debye- Scherrer formula obtained XRD pattern (Fig. 4) of the chromium nanoparticles, the average nanoparticles size was found to be 45.20 nm at $2\theta = 30.12°$ and 39.45°.

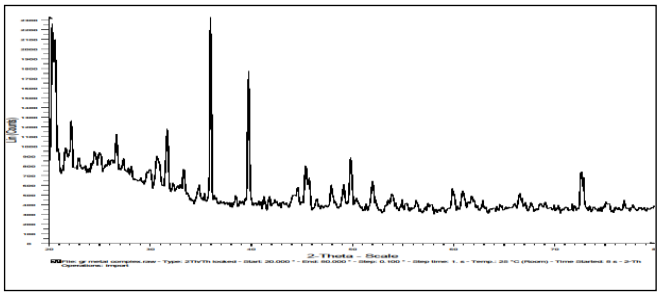


Fig. 4 XRD pattern of chromium polymer composite in aqueous solution at 900 °C

4. Conclusion

A thermal decomposition simple but effective method using polymer and chromium salt as precursor to synthesize chromium nanoparticles has been described. The reports from NMR, FTIR, SEM and XRD studies reveals that the synthesized chromium particles are in nano range. The XRD analysis establishes that the crystal pattern in the composite is monoclinic and crystal size is 45.20 nm. The synthesized nanoparticles may find applications in industry because of its characteristic mechanical and electrical properties.

Acknowledgement

The authors are grateful to The Head, Department of Polymer Science, M.L.S. University, Rajasthan for provide lab facilities. Authors thanks to IIT Jodhpur, Rajasthan for the result analysis.

References

[1] A. Stanoiu, C.E. Simion, L. Diamandescu, D. Tarabasanu-Mihaila, M. Feder, NO_2 sensing properties of Cr_2O_3 highlighted by work function investigations, Thin Solid Films 522 (2012)395-400.

[2] X. Pang, H. Yang, X. Liu, K. Gao, Y. Wang, A.A. Volinsky, A.A. Levin, Annealing effects on microstructure and mechanical properties of sputtered multilayer $Cr_{(1-x)}Al_xN$ films, Thin Solid Films 519 (2009) 5831-5837.

[3] X. Hou, K.L. Choy, Synthesis of Cr_2O_3-based nanocomposite coatings with incorporation of inorganic fullerene-like nanoparticles, Thin Solid Films 516 (2008) 8620-8624.

[4] A.S. Makhlouf, Z.H. Bakr, H. Al-Attar, M.S. Moustafa, Structural, morphological and electrical properties of Cr_2O_3 nanoparticles, Mater. Sci. Eng. B 178 (2013) 337-343.

[5] U. Balachandran, R.W. Siegel, Y.X. Liao, T.R. Askew, Synthesis, sintering, and magnetic properties of nanophase Cr_2O_3, Nanostruct. Mater. 5 (1995) 505-512.

[6] A.C. Santulli, M. Feygenson, F.E. Camino, M.C. Aronson, S.S. Wong, Synthesis and characterization of one-dimensional Cr_2O_3 nanostructures, Chem. Mater. 23 (2011) 1000-1008.

[7] S. Lei, X. Peng, Z. Liang, X. Li, C. Wang, et al., Self-template formation and properties study of Cr_2O_3 nanoparticle tubes, J. Mater. Chem. 22(4) (2004) 1643-1651.

[8] Z. Pei, H. Xu, Y. Zhang, Preparation of Cr_2O_3 nanoparticles via C_2H_5OH hydrothermal reduction, J. Alloys Compd. 468 (2009) L5-L8.

[9] Z. Pei, Y. Zhang, A novel method to prepare Cr_2O_3 nanoparticles, Mater. Lett. 62 (2008) 504-506.

[10] L. Sheng, L. Feng-Li, Z. Shao-Min, W. Peng, C. Ke, D. Zu-Liang, Highly sensitive room-temperature gas sensors based on hydrothermal synthesis of Cr_2O_3 hollow nanospheres, Chin. Phys B 18 (2009) 3985-3989.

[11] T. Tsuzuki, P.G. Mccormick, Synthesis of Cr_2O_3 nanoparticles by mechanochemical processing, Acta Mater. 48 (2000) 2795-2801.

[12] M.D. Lima, R. Bonadimann, M.J. Andrade, J.C. Toniolo, C.P. Bergmann, Preparation and catalytic application of chromium oxide thin films, J. Eur. Ceram. Soc. 26 (2006) 1213-1219.

[13] M. Abecassis-Wolfovich, H. Rotter, M.V. Landau, E. Korin, A.I. Erenburg, et al., Texture and nanostructure of chromia aerogels prepared by urea-assisted homogeneous precipitation and low-temperature supercritical drying, J. Non-Cryst. Solids 318 (2003) 95-111.

[14] M.J. Ocana, Electron spin resonance study of – Cr_2O_3 and $Cr_2O_3.nH_2O$ quasi-spherical nanoparticles, J. Eur. Ceram. Soc. 21 (2001) 931-939.

[15] J. Chaudhary, G. Tailor, D. Kumar, A. Joshi, Synthesis and thermal properties of copper nanoparticles, Asian J. Chem. 29 (2017) 1492-1494.

https://doi.org/10.30799/jnst.271.19050420

Cite this Article as: Jyoti Chaudhary, Giriraj Tailor, Deepak Kumar, Deepshikha Verma, Ajit Joshi, Synthesis and characterization of chromium nanoparticles by thermal method, J. Nanosci. Tech. 5(4) (2019) 820–821.