Clinical Cancer Investigation Journal-ISSN: 2278-0513

# Preparation of chromium oxide nanoparticles by decomposition of $[Cr(urea)_6(NO3)_3]$ complex by microwave irradiation


## Abstract

The present study aimed to prepare chromium oxide nanoparticles from the decomposition of [Cr (urea) 6 (NO3) 3] complex by microwave irradiation. In this study, Cr2O3 nanoparticles were prepared from the decomposition of the **[$Cr(urea)_6(NO3)_3$]** quickly and cleanly in 10 minutes. The product was identified using FT-IR, XRD, SEM and EDX analyses. All data showed that these compounds were formed in pure phase with a particle size of less than 30 nm. Also, nanoparticles of this oxide can be used in a wide range of areas such as electronic devices, heterogeneous catalysts, solar energy and optical materials. Many Metal Oxide Nanoparticles (MONPs) are synthesized by various methods in industry and laboratory. The development of new methods for the synthesis of MONPs is a general and essential aspect of chemistry. Heating by microwaves is one of the most essential applications of these waves and has been well-used in various chemical reactions in the last few decades. Some of the advantages of using microwaves are: (i) cost-effectiveness, (ii) increasing the load-bearing capacity of mineral and ceramic materials, which reduces the size of particles (grains) and increases the density, resulting in strengthening the mechanical properties of the produced minerals, (iii) reducing the possibility of contamination, (iv) the ability to penetrate (diffuse) into glasses and other objects, and (v) microwave can promote the reactions in a closed system well. Examples of chemical reactions by microwaves are various organic and inorganic syntheses, non-selective absorption, oxidation and reduction reactions, polymerization, synthesis of some nanoparticles, and catalytic reactions


**Keywords:** *Chromium oxide nanoparticles,* ***[Cr (urea)$_6$(NO3)$_3$]*** *complex, Microwave irradiation*
Chromium, Urea, Decomposition, Microwave, Nanoparticles, Chromium Oxide

Fatemeh Fatahi Chagani
Master Of Mining Chemistry, Department Of Chemistry,payame Noor University (PNU),P.O.Box 19395-4697,Tehran,Iran

Supervisor: Dr. Mansoureh Maleki,Department of Chemistry, payame Noor Universkity (PNU),P.O.Box 19395-4697, Tehran, Iran

Consultant Professor: Dr. Pouranpour Hakak , Department Of Chemistry, Payame Noor University (PNU), P. O. Box 19395- 4697, Tehran, Iran.

fatemeh.fatahichagani@yahoo.com
mansourehmaleki@pnu.ac.ir
p_pourhakkak@pnu.ac.ir

## Introduction

The effect of microwave heating is due to the ability of some liquids and solids to convert the energy of electromagnetic waves of microwave irradiation into heat. This conversion is followed by chemical reactions. This method of energy conversion has many attractions for chemists. The amount of energy conversion depends on the properties of the molecules. By controlling these properties, selectivity in the reactions is possible to some extent. In the microwave process, heat is generated inside the compound, while in the traditional furnace heating method, heat is absorbed from the outside and may cause heat stress, breakage, and other problems. The use of microwaves is important in the synthesis of materials. Microwave synthesis is usually faster, cleaner, and more cost-effective than conventional methods. Various minerals such as carbides, nitrites, simple and complex oxides, silicides, zeolites, glass, metals in nanoparticles, etc. have been synthesized using microwaves.

Rao et al. (2005) used a microwave solvothermal method for the preparation of nano-oxides (2). CeO2 nanoparticles with a size of 8 nm were prepared under microwave irradiation of 900 watts for 20 minutes from an aqueous solution of ammonium Ce (IV) nitrate and sodium hydroxide (2). Also, CeO2 single-phase crystalline ceramic powder was synthesized from the combustion of cerium nitrate mixture as oxidant and urea as fuel (3). ZnO was prepared by decomposition of Zn nitrate (NO3) 2 with and without microwave (4). In another work, the preparation of bar-shaped and monocrystalline ZnO has been reported (35).

In one study, microwave irradiation of a mixture of cobalt acetate solution in water with NaOH and citric acid resulted in the production of cobalt oxide nanoparticles (5). In another study, Co3O4 nanocrystals were prepared from microwave irradiation to Co_oleate precursor under solvothermal conditions (6). Co3O4 nano-sheets with a wall thickness of 20 nm were prepared using an aqueous solution of cobalt acetate (C4H6COO4) and Noah as a precursor and using citric acid as a coating agent under microwave irradiation (7).

In another study, the preparation of Co3O4 nanoparticles by microwave method with adding ethylene glycol to cobalt nitrate and trioctylphosphine oxide has been reported as a surfactant (8). In another study, CuO nanosheets were obtained by decomposing precursors resulting from the reaction of CuCl2.2H2O with NaOH without the use of surfactant. The reaction container is placed in the microwave after transfer to an autoclave. The reaction system is heated to a temperature of 103 for 30 min (9).

In another paper, the synthesis of LaCoO3 nanoparticles by microwave irradiation led to the production of La [Co (CN) 6] .5H2O precursor complex. Mixed oxides of SmCoo3, NdCOO3, and GdCoo3 can also be synthesized from Ln [Co (CN) 6] complexes by the same method (10). Also, the

microwave synthesis of barium titanate nanocrystals has been examined (67). Rao et al. (2000) used microwave irradiation to prepare electrode materials for lithium batteries (11). In an article, a raw material including LiCO3 and TiO2 was used to prepare Li4Ti5O12 spinel by microwave method. In this study, charcoal was used as a microwave-sensitive material (12).

In a study conducted by Mangalaraja et al. (2004), stable CoFe2o4 nanoparticles were obtained by microwave irradiation as a heat source (13). Ferrite powder with the formula of Ni8.0Zn2.0Fe2O4 was synthesized by the combustion method under microwave irradiation (14). Fu et al. (2004) synthesized ferrite powders containing Ni-Cu-Zn by microwave combustion method (15). In 2004, the LiMn2O4 nanocomposite was synthesized as a cathode electrode material for lithium batteries under microwave irradiation (16). In another study, small, LiMn2O4 spinal nano-powders with uniform and small particle sizes were synthesized by Fu et al. in 2004 (17). Kanoh et al. synthesized LiMn2 nano-composition using microwave irradiation and examined its thermal behavior (18). LiNiO2 was synthesized in 2003 by Kaliani et al. (2003) under microwave irradiation (19). Liu et al. (2004) synthesized gold nano-rods from HAuCl4, sodium nitrate, acetone, and TOAB (20).

Liu et al. (2004) obtained silver nanoparticles in the presence of gold granules under microwave heating with a power of 1200 watts (21). Jiang et al. (2005) prepared hydrophobic gold nanoparticles with harmless organic solvents by microwave irradiation (22). Following the studies conducted in this area, H2 c [Cu (Sac) 2] complex CuO and ZnO nanoparticles were prepared with the easy, fast, and cost-effective method and were examined for the first time under microwave irradiation. The results of microwave analysis of these classical complexes were collected and analyzed.

## Materials and Methods

To prepare the precursor complex [Cr (urea) 6 (NO3) 3], 8 g of Cr (NO3) 39H2O and 7.25 NH2CONH2 urea were dissolved separately in 25 ml of ethanol. Then, the obtained two solutions were mixed, and then the above mixture was stirred for half an hour. Then, we put it aside to precipitate completely. The resulting dark green precipitate was washed with ethanol and diethyl ether and dried at 60 ° C for 12 hours. To prepare the required amount of this complex, the above method was repeated several times. To prepare Cr2O3 nanoparticles, 1 g of complex powder [Cr (NH2CONH2)6 $(NO3)_2$] was placed in a small porcelain crucible. Then, it was exposed to microwave irradiation. After 10 minutes, the complex was completely decomposed within a small crucible. The resulting black powder was detected by XRD, FT-IR, and SEM techniques.

## Results

To prepare and identify Cr2O3 nanoparticles using the microwave method from the prepared precursor, 1 g was repeatedly placed in the microwave for 10 minutes with a power of 900 watts. The resulting powder was tested using FT-IR, VSM, SEM, and XRD techniques.

### Analysis and interpretation of the FT-IR spectrum of the precursor complex [Cr (urea) 6 $(NO3)_3$]

To examine the thermal behavior of the precursor [Cr (urea) $_6$ $(NO3)_3$] and to determine its possible decomposition path, the FT-IR spectrum was recorded for this precursor complex, and the samples were obtained from its decomposition at different temperatures. For this purpose, the FT-IR spectrum of crude complex powder [Cr $(urea)_6$ $(NO3)_3$] is shown in Figure (1). In this spectrum, several bands characteristic of the organic ligands urea and NO3 appeared. The O = C band was observed in the range of 1625 $cm^{-1}$, the NO3 band in the range of 1550 $cm^{-1}$, and the NH2 band in the range of 3300 to 3500 $cm^{-1}$.

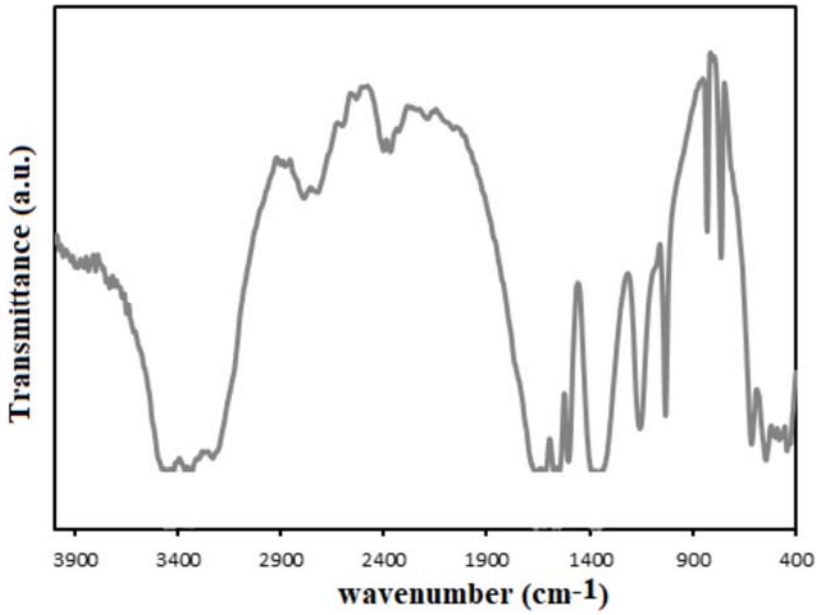


Figure 1: FT-IR spectrum of raw powder of [Cr (urea) $_6$ (NO3) $_3$] complex

**Analysis and interpretation of FT-IR spectra of Cr2O3 nanoparticles prepared by microwave method**

Figure (2) shows the IR-FT spectrum of several synthesized Cr2O3 samples from the decomposition of [Cr (urea)$_6$ (NO3)$_3$] under microwaves for 10 min. As seen, in this spectrum, two strong and sharp bands are seen in the area of about 600 cm$^{-1}$, which indicates the bond between chromium and oxygen. These bands appear at 570 and 630 cm$^{-1}$, which are attributed to the symmetric and asymmetric tensile vibrations of the chromium-oxygen bonds in the Cr2O3.

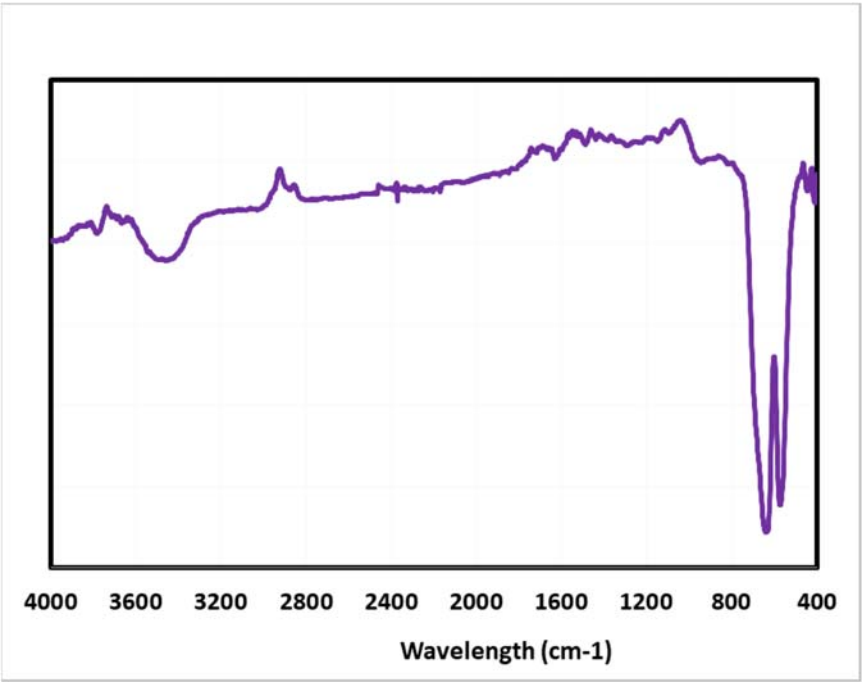


Figure 2: FT-IR spectrum of Cr2O3 nanoparticles prepared by microwave method

**Analysis and interpretation of XRD spectra of Cr2O3 nanoparticles prepared by microwave method**

Fuzzy and structural changes in powders of precursor decomposition of [Cr (urea) $_6$ (NO3) $_3$] under microwave irradiation by XRD were examined. Figure (3) illustrates the diffraction pattern of XRD spectroscopy of Cr2O3 nanoparticles resulting from the decomposition of [Cr (urea) $_6$ (NO3) $_3$] under microwave irradiation for 10 minutes. By comparing the spectrum observed with the standard diffraction pattern related to Cr2O3, the single-phase XRD spectrum

shown in Figure (3) related to the Cr2O3 phase is confirmed. The mean particle size (D) and Cr2O3 nanoparticles can be estimated by line width in the XRD pattern using Debye-Scherrer equation $D = 0.9\ \lambda\ /\ (\beta Cos\theta)$. Using the above formula, based on the line width of 0.35 at half the height of the major peak (0.100) appearing in Θ2-36, the particle size of about 35 nm was calculated.

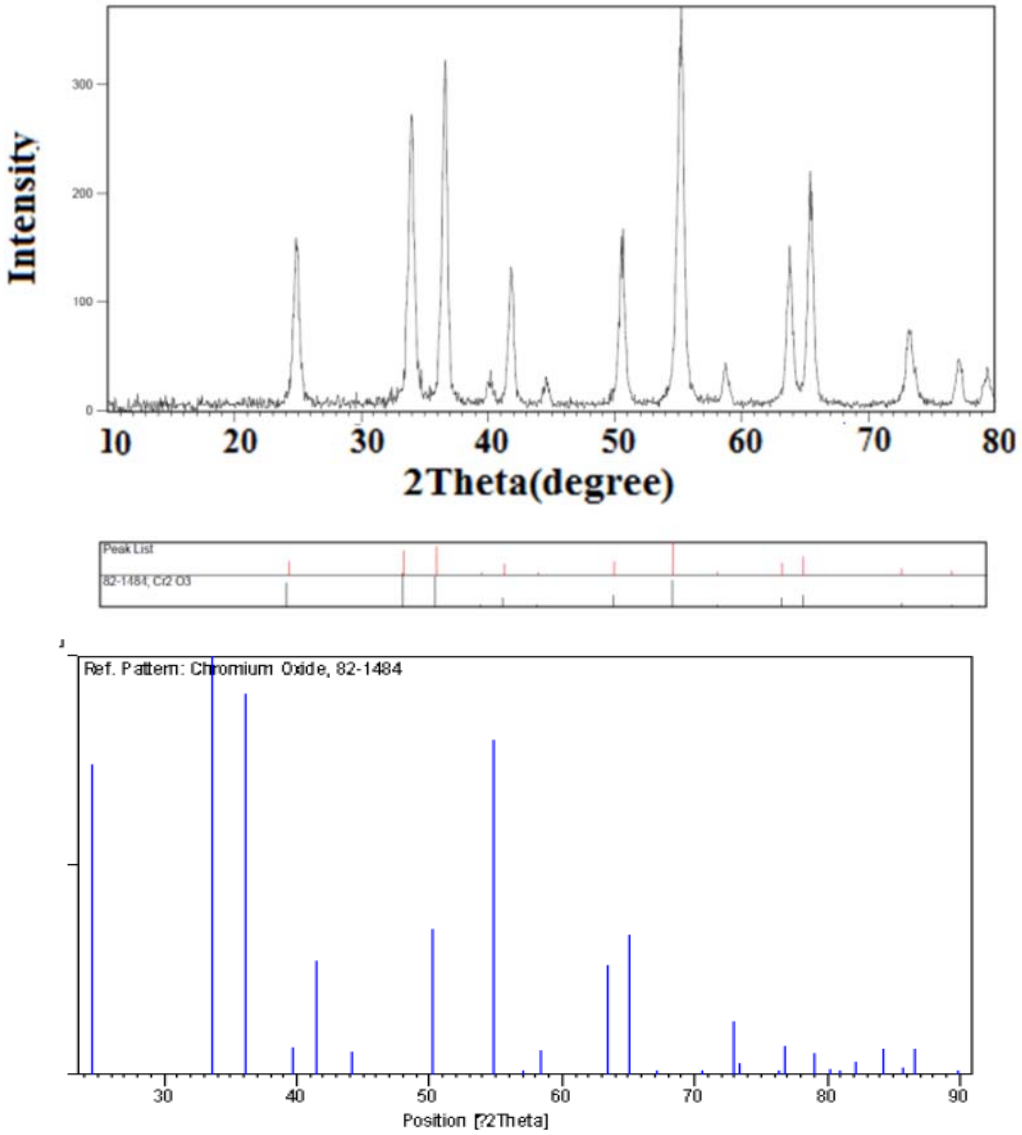


Figure 3: XRD spectrum of Cr2O3 nanoparticles prepared by microwave method

**Analysis and interpretation of SEM images of Cr2O3 nanoparticles prepared by microwave method**

IR-FT and XRD data confirm the preparation of Cr2O3 nanoparticles from the decomposition of precursor [Cr (urea) $_6$ (NO3) $^3$] synthesized under microwave waves. Figure 4 illustrates the SEM images for morphology and topography examination of these nanoparticles at 300 and 100 nm. Based on its shape and scale, it can be concluded that the powder is composed of almost spherical nanoparticles. Nanoparticles are about 30 nm and appear as large spheres due to their small size and tendency to stick to each other.

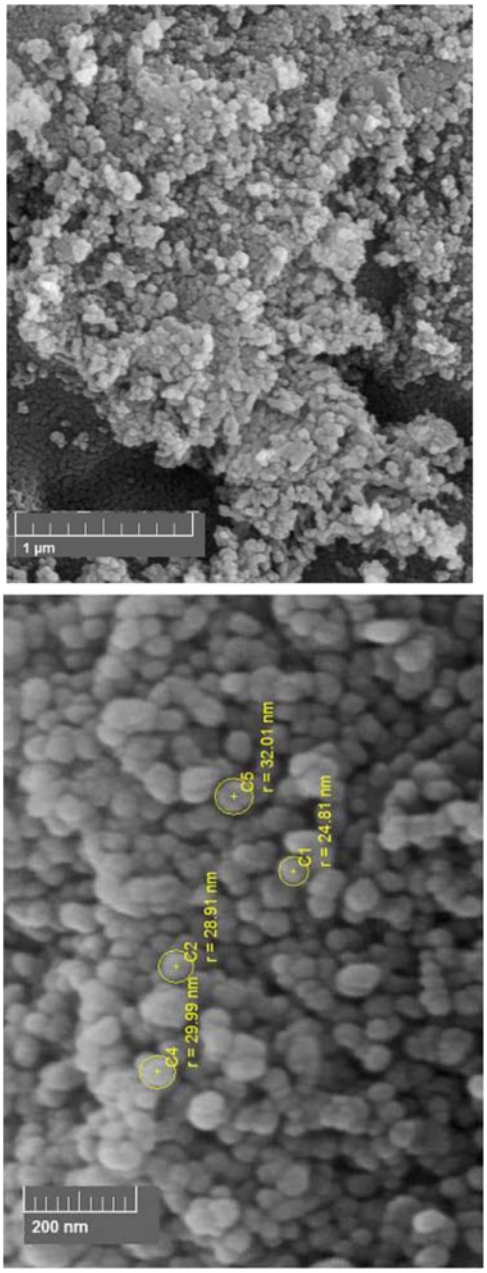


Figure 4: SEM images of Cr2O3 nanoparticles prepared by microwave method

Figure (5) illustrates shows the roles of Cr2O3 nanoparticles and shows the uniform dispersion of Cr and O elements in the nanoparticle composition.

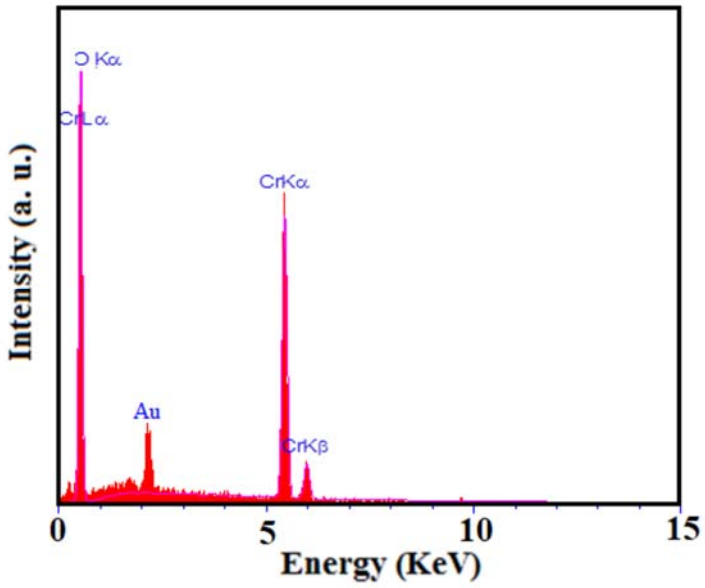


Figure 5: Analysis of Cr2O3 nanoparticles by decomposition of [Cr (urea) $_6$ (NO3) $_3$] complex using EDS in microwave method

**Conclusion**

In general, in this study, a new, simple and fast method was presented for preparing pure nanoparticles from Cr2O3 nanoparticles from the decomposition of [Cr (urea) $_6$ (NO3) $_3$] precursor under irradiation in a microwave oven. Preparation of metal monoxide based on the decomposition of molecular precursor decomposition under microwave irradiation has many benefits such as no need for solvents, surfactants, and complex devices. The results of FT-IR, XRD, and SEM-EDS confirmed that the nanoparticles of this oxide were formed in pure form with fine and homogeneous granules. XRD analysis showed that particle size is at the nanoscale in all cases.

……

## References


1. Rao, K.J.; Mahesh, K.; Kumar, S. …….. *Bull. Mater. Sci.* 2005, *28,* 19.
2. Yong, H.; Huang, C.; Tang, A.; Zhang, X.; Yang, W. *Mater. Res. Bull.* 2005, *40,* 1690.
3. Fu, Y.P.; Lin, C.H.; Hsu, C.S. *J. Alloy Compd.* 2005, *391,* 110.
4. Komarneni, S.; Bruno, M.; Mariani, E. *Mater. Res. Bull.* 2010, *35,* 1843.
5. Al-Tuwirqi, R.; Al-Ghamdia, A.A.; Abdel Aal, N.; Umarc, A.; Mahmouda, W.E. *Superlattice. Microst.* 2011, *49,* 416.
6. Sun, C.; Su, X.; Xiao, F.; Niu, C.; Wang, J. *Sensor. Actuat. B-Chm.* 2011, *157,* 681.
7. Al-Tuwirqi, R.; Al-Ghamdi, A.; Aal, N.; Umar, A.; E.Mahmoud, W. *Superlatt. Microstruc.* 2011, *49,* 416.
8. Bhatt, A.; Bhat, D.; Tai, C.; Santosh, M. *Mater. Lett.* 2011, *125,* 34.
9. Moura, A.P.; Cavalcante, L.S.; Sczancoski, J.C.; Stroppa, D.G.; Paris, E.C. Advance. Powder. 2010 *21*, 197.
10. Farhadi, S.; Sepahvand, S. *J.Alloys Compd*. 2010, *489,* 586.
11. Bhat, M.H.; Chakravarthy, B.P.; Ramakrishnan, P.A.; Levasseur, A.; Rao, K.J. *Bull. Mater. Sci*. 2000, *23*, 461.
12. Amini, M.M.; Torkian, L. *Mater. Lett.* 2012, *57,* 639. Li, J.; Jin, Y.L.; Zhang, X.G.; Yang, H. *Solid State Ionics. 2016, 1590,* 1594.
13. Bensebaa, F.; Zavalich, F.; Lecuyer, P.; Cochrane, R. W.; Veres, T. *J. Colloid Interface Sci*. 2014, *277*, 104.
14. Mangalaraja, R.V.; Ananthakumar, S.; Manohar, P.; Gnanam, F.D. Awana, M. *Mater. Lett.* 2014, *58*, 1593.
15. Fu, Y.P.; Lin, Ch.H.; Liu, Ch.W. *J. Magn. Magn. Mater*. 2014, *283*, 59.
16. Lio, H.; Hu, C.; Zhu, X.; Hao, H.; Luo, J.; Zhou, J.; Ouyang, S.H.; *J. Mater. Chem. Phys.* 2004, *88,* 290.
17. Fu, Y.P.; Lin, C.H.; Su, Y.H.; Su, J.H.; Jean, J.H.; Wu, S.H. *Ceram. Int*. 2014, *30*, 1953.
18. Kanoh, H.; Chitrakar, R.; Yai, Y.M.; Ooi, K. *J. Solid State Chem.* 2012, *163,* 1.
19. Kalyani, P.; Kalaiselvi, N.; Renganathan, N.G. *J. Power Sources.* 2013, *123,* 53.
20. Chen, W-X.; Lee, J.Y.; Liu, Z. *Mater. Lett.* 2014, *58,* 3166.
21. Zhu, Y.J.; Hu, X.L. *Mater. Lett.* 2014, *58,* 1234.
22. Kim, K.H.; Park, H.C.; Lee, S.D.; Hwa, W.J.; Hong, S.S.; Lee, G.D.; Park, S.S. *Mater. Chem. Phys.,* 2015, 62, 178.