

# Preparation and Identification of Nanoparticles Iron(II) Chloride and Thermal Behavior Studies

Mohamad Mirzaie\*, Zahra Rahmani, Ali Taherkhani

Department of Science, Takestan Branch, Islamic Azad University, Takestan, Iran

Received: June 10 2013

Accepted: July 11 2013

## ABSTRACT

Synthesis, characterization, and thermal behavior studies of nanoparticles Iron(II) chloride has been studied in this research. Iron(II) chloride was synthesized by planetary high-energy ball mill. The general formula of this compound is  $\text{FeCl}_2$ . The synthesized nanoparticles were characterized by Fourier transform infrared spectroscopy (FT-IR) and also Size and structure of synthesized nanoparticles were studied by analyzing X-ray diffraction (XRD) and morphology of surface and structure of synthesized nanoparticles were studied by scanning electron microscopy (SEM). This compound has many applications in mineral synthesis as a catalyst. The nanoparticles of  $\text{CoBr}_2$  synthesized with smaller size of 35 nm, and its SEM images show that the morphology of particles surface is as layer and hunk. Also thermal behavior of this nanoparticles considered by using of DTA /TGA thermal analysis.

**KEYWORDS:** Synthesis, characterization, Iron(II) chloride; mill device, X-ray diffraction, scanning electron microscopy, thermal behavior

## 1. INTRODUCTION

Ferrous chloride occurs in nature as the mineral lawrencite. Its applications include its use in metallurgy as an electrolytic agent and as a mordant in dyeing.  $\text{FeCl}_2$  has been used in the synthesis of pent substituted acylferrocenes [1]. In biological research, ferrous chloride is used as a source of  $\text{Fe}^{2+}$  ion. Studies of oxidative stress in biology have utilized  $\text{FeCl}_2$  with hydrogen peroxide to generate hydroxyl radicals via the Fenton reaction [2,3].  $\text{FeCl}_2$  has been shown to block calcium influx through N-methyl-D-aspartate receptor channels in immature cultured rat cortical neurons [4]. It has also been used in a study of the interaction between hypoxia-inducible factor (HIF- $\alpha$ ) and the von Hippel-Lindau ubiquitylation complex [5].

Nanocrystalline metallic compounds are said to have enhanced ductility and yield strength as compared to conventional grain-sized materials [6]. The type of mill employed in the mechanical milling process accounts for different milling mechanisms, that is, the way in which the available energy is transferred from the milling media to the material.

Developed by Benjamin, mechanical alloying (MA) is an alternative technique for the fabrication of powder particles [7]. MA is a solid state powder process in which MA. The powder mixture is mechanically ball milled using a high-energy ball mill by which different alloys, ceramics, composites and amorphous materials [8-12] can be synthesized.

In this work, nanoparticles of  $\text{CoBr}_2$  compound were synthesized in planetary high-energy ball mill and its characterize and thermal behavior were studied.

## 2. EXPERIMENTAL

### 2.1. Materials and Instruments

Starting materials were obtained from Merck (Berlin, Germany) and were used without further purification. Fourier transform infrared (FT-IR) spectra were recorded on a Bruker spectrophotometer in KBr tablets. Surface morphology of product was characterized by using a LEO-1430.VP scanning electronic microscopy (SEM) with an accelerating voltage of 15 kV. X-ray powder diffraction (XRD) measurements were performed using a Philips diffractometer manufactured by X'pert with monochromatized Cu K $\alpha$  radiation. Sizes of selected samples were estimated using the Scherer method. For identification a scanning electron microscope samples were gold coated.

### 2.2. Synthesis of nanoparticles Iron(II) chloride

Initially 5.5 g of cobalt chloride weighed for produce about 5 grams of Iron(II) chloride ( $\text{CoBr}_2 \cdot 6\text{H}_2\text{O}$ ), and poured in a baker, we've added to it 11 ml of acid hydrobromic and we've heated to it, and we've putted in the oven for 30 minutes at a temperature of 50 °C, were completely wiped out and humidity combine dry. Then Cobalt bromide without of water was transported to a lacuna the mill and the was milled in a planetary high-

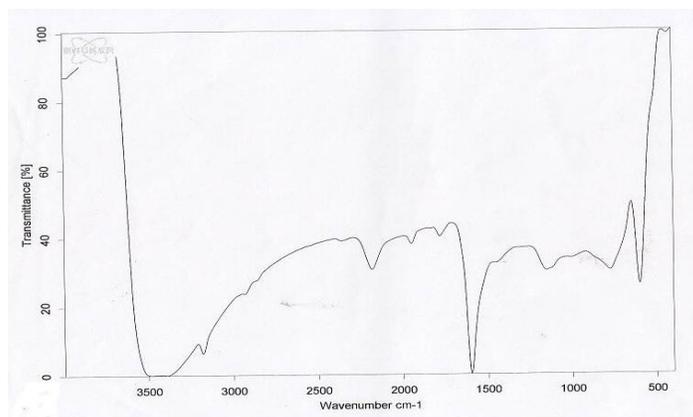
energy ball mill operated at 250 rpm for 10h . Twenty zirconium balls of 10 mm diameter are being used in all milling processes.

### 3. Characterization of nanoparticles

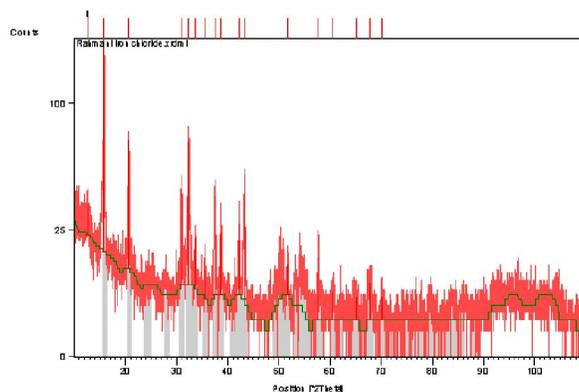
X-ray diffraction (XRD) technique was used to determine the ingredients of the milled powder. The morphology of nanoparticles was observed using a scanning electronic microscopy (SEM). The obtained samples were characterized and compared via FT-IR analysis with bulk (non-nano) forms. FT-IR spectrometer at room temperature in the range from 400 to 4000 $\text{cm}^{-1}$ .

## 4. RESULTS AND DISCUSSION

In this paper, we report the synthesis nanoparticles Iron(II) chloride. After preparing nanoparticles, it was characterized by IR.(Figure 1)



**Fig. 1.** FT-IR spectra of  $\text{FeCl}_2$  nanoparticles



**Fig. 2.** The XRD pattern of  $\text{FeCl}_2$  nanoparticles

Figure 2 shows the XRD pattern of nanoparticles prepared by the planetary high-energy ball mill process. Estimated from the Sherrer formula for the calculation of particle sizes from the broadening of the XRD peaks ( $D = 0.9\lambda/\beta\cos\theta$ , where  $D$  is the average grain size,  $\lambda$  is the X-ray wavelength (0.154 nm), and  $\theta$  and  $\beta$  are the diffraction angle and full width at half maximum of an observed peak, respectively). The average size of the particles was found to be around 35 nm, which is in agreement with the value obtained from the SEM images (Figure 3 &4).

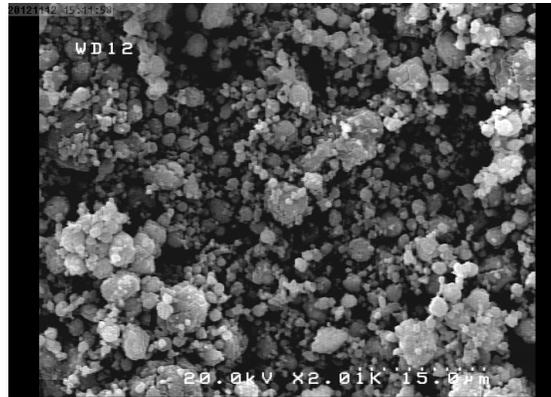


Fig. 3.SEM images of the FeCl<sub>2</sub> nanoparticles



Fig. 4.SEM images of the FeCl<sub>2</sub> nanoparticles

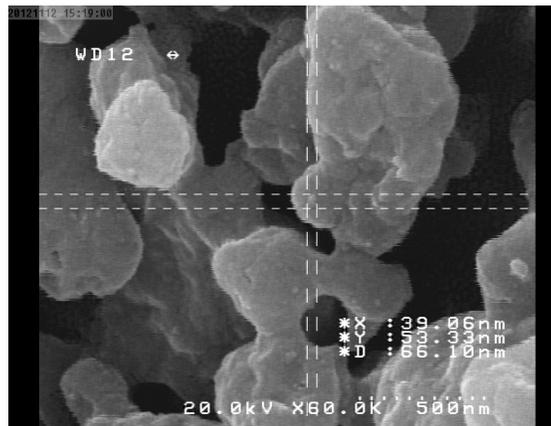


Fig. 5.SEM images of the FeCl<sub>2</sub> nanoparticles

#### 4.1. Study the thermal behavior

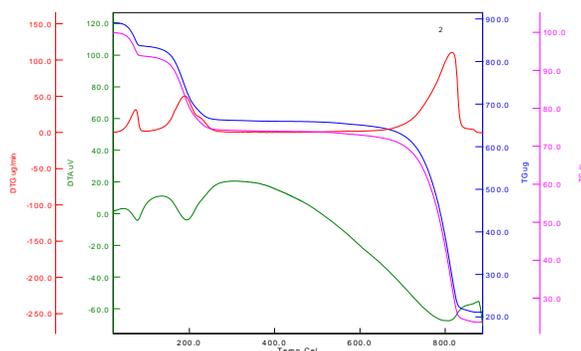
To study the thermal stability of the ferric chloride by weight calorimetry (TG) and differential thermal analysis (DTA) of the temperature range of zero to used. Figure (2-3) shows the combination of thermal analysis chart.

The TG composition diagram above, there are three stages of weight losses. First weight loss in the temperature range of departure is C 70 -20 (8%) of the initial mass is mixed. These weight loss, water evaporation Surface composition is assigned.

The second weight loss in the temperature range C 220-170 can be seen that the removal (20%) of the mass of iron chloride, the weight loss is attributed to the evaporation of water inside the compound. Temperature Range C 740-220 combined weight is constant and not weight loss.

In the third stage weight loss of about 54% of the weight (gr 375/107) in the temperature range C 830-740 struck down Vhds be combined in this temperature range have been destroyed, what can the rest of the environment. was probably related to the presence of chlorine, as well as metallic iron, molecular weight (gr 45/91) with computational results (gr5/91) is a very good match.

The DTA curve combination, the C 900 at the temperature range from zero to three, °combination of analysis ° C70-0 and °with three exothermic process temperature, respectively Drbaz-h C 800-210. °C210-70 and is associated



**Fig. 6.** thermal behavior of the FeCl<sub>2</sub> nanoparticles

## 5. CONCLUSION

In this research the detail studied the preparation, identification, and thermal behavior of nano particles Iron(II) chloride. In summary, the molecular structure of nano particles is confirmed by the presence of functional groups in FTIR spectra. Also theoretical data show good agreement with the experimental result. In addition, the values of crystallite size in nano scale are demonstrated by X-ray diffraction method for nano Iron(II) chloride powders.

## 6. ACKNOWLEDGEMENTS

We gratefully acknowledge the financial support from the Research Council of Imam Khomeini International University and Abhar payamnour University and many technical supports provided by Takestan Islamic Azad University.

## REFERENCES

- [1]. Norinder, J., et al., An efficient route to pentasubstituted acylferrocenes. *J. Org. Chem.*,67(25), 9096-9098 (2002).
- [2]. Shoker, A. S., et al., Inhibition of human lymphocyte function by organic solvents. *Mol.Cell. Biochem.*, 171(1-2), 49-58 (1997).
- [3]. Yu, T. W., and Anderson, D., Reactive oxygen species-induced DNA damage and its modification: a chemical investigation. *Mutat. Res.*, 379(2), 201-210 (1997).
- [4]. Nakamichi, N., et al., Blockade by ferrous iron of Ca<sup>2+</sup> influx through N-methyl-D-aspartate receptor channels in immature cultured rat cortical neurons. *J. Neurochem.*, 83(1), 1-11 (2002).
- [5]. Jaakkola, P., et al., Targeting of HIF- $\alpha$  to the von Hippel-Lindau ubiquitylation complex by O<sub>2</sub>-regulated prolyl hydroxylation. *Science*, 292(5516), 468-472 (2001).
- [6] R. Bohn, T. Haubold, R. Birringer, H. Gleiter. (1991). Nanocrystalline intermetallic Compounds – an approach to ductility, *Scripta Metall Mater.* 25, 811–816.
- [7] E.T. Kubaski , O.M. Cintho , J.L. Antoniassi , H. Kahn , J.D.T. Capocchi, (2012)Obtaining NiAl intermetallic compound using different milling devices, *Advanced Powder Technology*,23, 667-672
- [8] J.S. Benjamin. (1992). Powder metallurgy, in: *Proceedings of the 1992 Powder Metallurgy World Congress, San Francisco, CA, Publ. Metal Powder Industries*, 7, 155.
- [9] R.B. Schwarz, W.L. Johnson. (1983). *Phys. Rev. Lett.* 41, 415.
- [10] E. Hellstern, L. Schultz. (1987). *Mater. Sci. Eng.* 93, 213.
- [11] J.A. Hunt, I. Soletta, S. Enzo, L. Meiya, R.L. Havill, L. Battezzati, G. Cocco, N. Cowlam. (1995). *Mater. Sci. Forum* 179–181, 255.
- [12] T. Nasu, K. Nagaoka, M. Sakurai, K. Suzuki. (1995). *Mater. Sci. Forum* 179–181, 97.