Int. J. Nano Dimens. 6 (4): 401-407, Autumn 2015
ISSN: 2008-8868

Contents list available at **IJND**

**International Journal of Nano Dimension**

**Journal homepage: *www.IJND.ir***

# Synthesis and characterization of Nickel (II) Chloride nanoparticles with the study of their thermal behavior

**Z. Rahmani[1,*]**
**Sh. Ghamamy[2]**

*[1]Department of Chemistry, Takestan Branch, Islamic Azad University ,Takestan , Iran.*
*[2]Department of Chemistry, Faculty of Science, Imam Khomeini International University, Qazvin, Iran.*

Received 21 September 2014
Received in revised form
24 December 2014
Accepted 29 December 2014

* Corresponding author:
Zahra Rahmani
Department of Chemistry,
Takestan Branch, Islamic Azad University, Takestan, Iran.
Tel +98 9372491211
Fax +98 28305270145
Email *rahmaniz33@yahoo.com*

**ABSTRACT**

In this research nanoparticles of Nickel (II) Chloride were synthesized and characterized using fourier transform infrared (FT-IR) spectra. Nanoparticles of Nickel (II) Chloride were prepared by using of ball mill device. A ball mill is one kind of grinding machine, and it is a device in which media balls and solid materials (the materials to be ground) are placed in a container. In the research Nickel (II) Chloride compound was milled for 10 h at 250 rpm in a hardened stainless steel medium. The resulting nanoparticles were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The SEM imaging technique was applied for the observation of grain sizes and the morphology of the nanoparticles. The Debye-Scherrer formula was used to confirm the grain sizes determined by the SEM slides. XRD results of nanoparticles showed that the crystallite size of reaching 59 mm after 10 h at 250 RPM. Thermal behavior of nanoparticles was considered by using of DTA /TGA thermal analysis device. TGA analysis reveals that the synthesized Nickel (II) Chloride nanoparticle was thermally stable up to 900°C.

**Keywords:** *Synthesis; Characterization; Nickel (II) Chloride nanoparticles; Ball mill; X-ray Diffraction (XRD); Scanning Electron Microscopy (SEM); Thermal behavior*.

## INTRODUCTION

Nanomaterials and nanoparticles are used in a broad spectrum of applications. Today they are contained in many products and used in various technologies. Most nanoproducts produced on an industrial scale are nanoparticles, although they also arise as byproducts in the manufacture of other materials [1,2].The most important technologies are applied in the formulation development to enhance solubility and dissolution which include particle size reduction, salt formation, and amorphous systems, as well as co-solvents, complexes, surfactants, and lipid formulations. Among these techniques, particle size reduction is highly promising. Therefore, reducing the particle size to the nano range has been investigated through several approaches.

Unlike nanoparticles produced from "bottom-up" processes such as self-assembly and template synthesis, nanoparticles from mechanical attrition are produced by a "top-down" process **(**Figure1). Such nanoparticles are formed in a mechanical device, generically referred to as a "mill" in which energy is imparted to a course-grained material to effect a reduction in particle size [3,4]. Under certain conditions, the resulting particulate powders can exhibit nonstructural characteristics on at least two levels. Firstly, the particles themselves, which normally possess a distribution of sizes, can be "nanoparticles" if their average characteristic dimension (diameter of spherical particles) is less than 100 mm [5]. Secondly, many of the materials milled in mechanical attrition devices are highly crystalline, such that the crystalline (grain) size after milling is often between 1 and 10 nm in diameter. Such materials are termed "nanocrystalline". The sizes of the nanocrystals and the nanoparticles may or may not be the same. In some of the nanostructure materials literature, particularly that involving bottom-up processes, the term "nanocrystal" is reserved for crystalline particles with low concentrations of defects, such as are found in single crystals, whereas "nanoparticles" are those nanoscale particles that contain gross internal grain boundaries, fractures, or internal disorder, whether the crystals they contain are nanocrystalline or not.Ball milling is a mechanochemical technique, which is widely applied to the grinding of minerals into fine particles and the preparation and modification of inorganic solids [6]. Its use in synthetic organic chemistry is relatively scarce, but attains more importance during the past decade. Kaupp *et al.* have discovered the usefulness of Ball-milling in synthetic organic chemistry, which have been the subject of some papers and reviews [7-13].Some recent examples include solvent-free Knoevenagel condensation [14], protection of diols and diamines [15], functionalization of fullerenes [16]**,** reductive benzylation of malononitrile **[**17] **,** preparation of phosphorus yields **[**18]**,** and Heck-type cross-coupling reactions [19,20]. The major two processes are ball milling and high-pressure homogenization, either in water or in non-aqueous water-reduced media. Disadvantages of the ball milling process are the relatively long milling times in the case of hard drug material, the limited scaling up ability (the weight of the ball mill and the milling material limit maximum size), and potential contamination of the milling material by erosion from the milling pearls [21]. In this work, nanoparticles of Nickel (II) chloride compound were synthesized in planetary high-energy ball mill. The synthesized nanoparticles were characterized by Fourier transform infrared spectroscopy and also size and structure of synthesized nanoparticles were studied by analyzing X-ray diffraction and morphology of surface and structure of synthesized nanoparticles were studied by scanning electron microscopy.

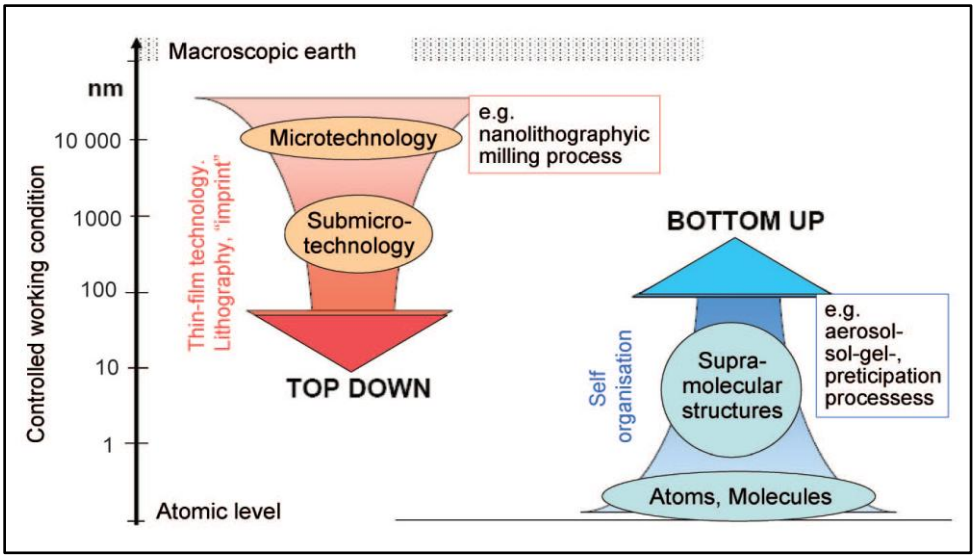


**Fig. 1.** Methods of nanoparticle production: top-down and bottom-up.

## EXPERIMENTAL

### *Materials and Instruments*

Starting materials were obtained from Merck (Berlin, Germany) and were used without further purification. Ball milling was conducted using the planetary ball mill "Pulverisette 7 classic line" (Fritsch GmbH, Germany). For balancing, two grinding beakers ($V$ = 45 ml) of nearly the same weight were placed inside the ball mill. Fourier transform infrared (FT-IR) spectra were recorded on a Bruker spectrophotometer in KBr tablets. The surface morphology of product was characterized by using a LEO-1430.VP scanning electronic microscope (SEM) with an accelerating voltage of 15 kV. X-ray diffraction (XRD) measurements were performed using a commercial diffractometer with Cu Kα radiation in the 2u range from 10 to 808. Also the thermal behavior of nanoparticles was considered by using of DTA /TGA thermal analysis device.

### *Synthesis of Nickel (II) chloride nanoparticles*

Ball-milling was performed at 20-25 Hz frequency usually at room temperature (without circulating liquid the temperature did not rise above 30°C) .The mechanical alloying was executed in a one station planetary ball with the following parameters:

Ball-to-powder weight ratio: 10:1; ball diameter: 16 mm; ball and vial material: hardened Stainless steel; speed: 250 RPM. The Nickel (II) chloride powder was milled up to 10 h to reach steady state condition. The milling was performed under Toluene as the process control agent (PCA) to avoid the formation of inter-metallic compounds during milling.

## RESULTS AND DISCUSSION

The FTIR spectroscopy is an analysis method which provides structural studies to explore the fundamental and functional groups in crystalline and non-crystalline matrices. The transmission spectra of the Nickel (II) Chloride nanoparticles are recorded in Figure 2. The FTIR spectra of the Nickel (II) Chloride nanoparticles were recorded in the range of 400 – 2000 $cm^{-1}$.

Spectroscopic data of synthesized compound are reported below.

$NiCl_2{\cdot}6H_2O$: IR (KBr): ν (Ni-Cl): 723.05, ν (O-H): 1610.98, ν (O-H):3396.67 $cm^{-1}$.

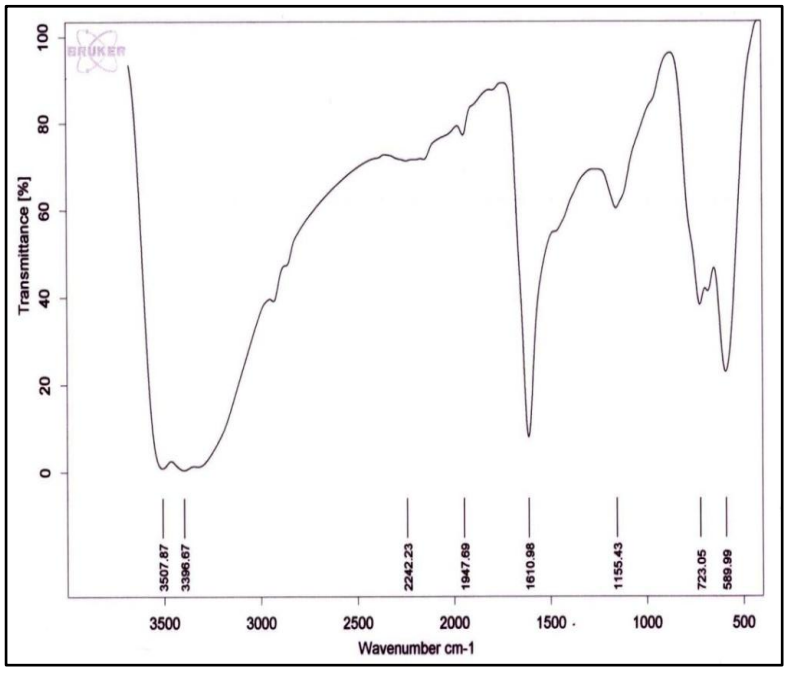


**Fig. 2.** FT-IR spectra of $NiCl_2{\cdot}6H_2O$ nanoparticles.

X-ray diffraction (XRD) technique was used to determine the ingredients of the milled powder. Figure 3 shows the XRD patterns of Nickel (II) chloride nanoparticles prepared by ball mill process. The morphology of nanoparticles was observed using a scanning electronic microscopy (SEM). The mean size of nanoparticles was calculated to be about 59 nm using Scherrer's equation: $D = K\lambda/(\beta \cos \theta)$, where D is the mean crystalline size (nm), λ is the wavelength of Cu Kα (0.154 nm), β is the full width at half maximum intensity in radian, and θ is the Bragg angle (°) [22]. Figures 4-6 showing SEM micrograph confirms almost spherical shaped particles which is the characteristics of the steady state milling condition in the sample milled for 10h at 250 rpm.

TGA has been applied to the investigation of the thermal behavior and structure of this compound. Thermal decomposition of Nickel (II) Chloride shows multistage processes. The TGA curve of Nickel (II) chloride nanoparticles is shown in Figure 7. The degradation has occurred in three steps. The first degradation from 60°C to 100°C is attributed to the removal of water from the surface (mass loss of about 5%). The second degradation is from 100°C to 190 °C. 15% mass reduction has been observed in this region. This is due to the dehydration and removal of organic residues. The third degradation from 510°C to 780°C results in an additional mass loss of about 45%. Above 900°C, an essentially constant mass (35% sample) has been found indicating the thermal stability of the sample.

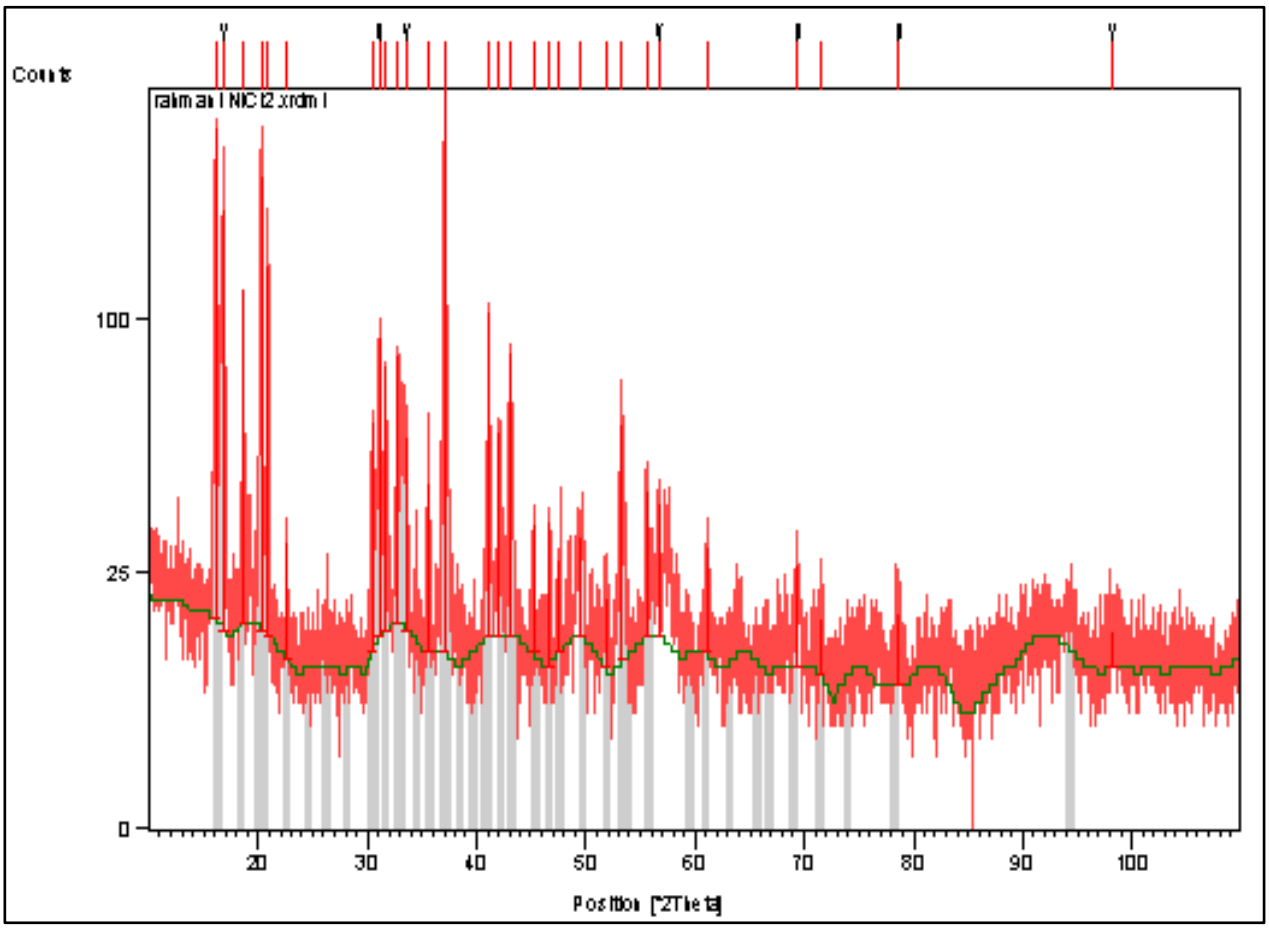


**Fig. 3.** The XRD pattern of $NiCl_2 \cdot 6H_2O$ nanoparticles.

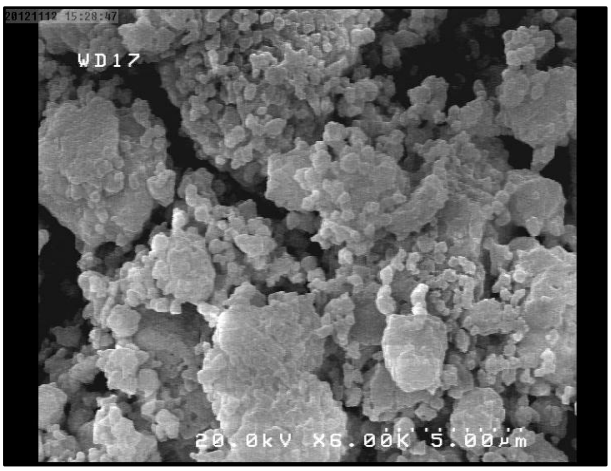


**Fig. 4.** SEM images of the $NiCl_2 \cdot 6H_2O$ nanoparticles.

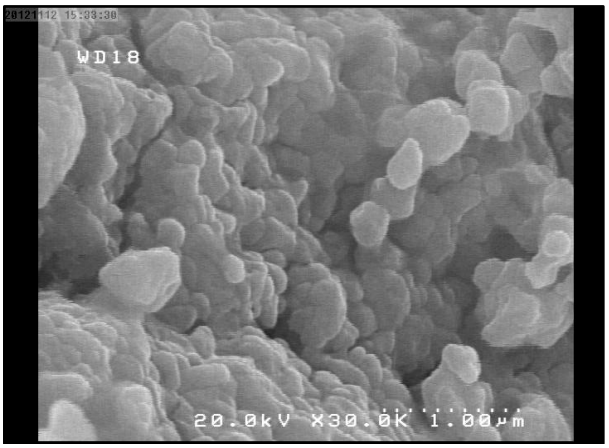


**Fig. 5.** SEM images of the $NiCl_2 \cdot 6H_2O$ nanoparticles.

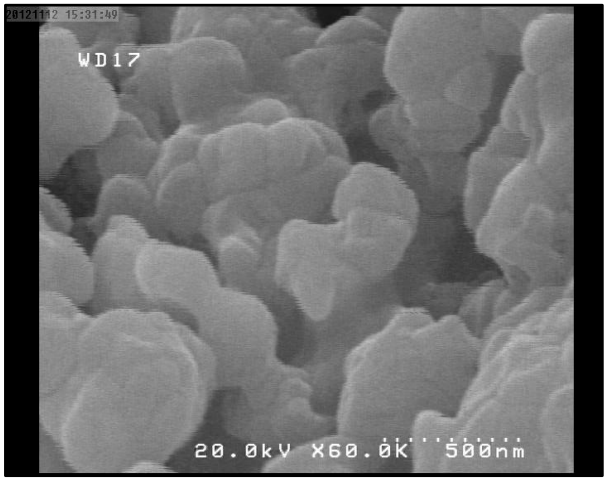


**Fig. 6.** SEM images of the $NiCl_2 \cdot 6H_2O$ nanoparticles.

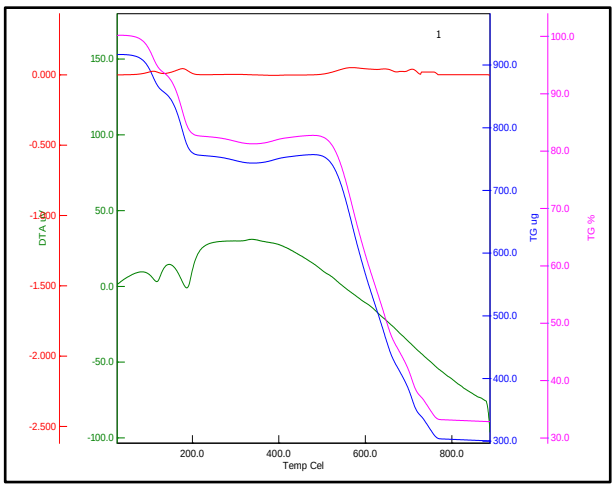


**Fig. 7.** TGA graph of $NiCl_2 \cdot 6H_2O$ nanoparticles.

## CONCLUSIONS

Mechanical alloying is a simple, elegant, and useful processing technique that continues to attract the serious attention of researchers. In this research the detail studied the synthesis, characterization**,** and thermal behavior of Nickel (II) Chloride nanoparticles. In summary, the molecular structure of nanoparticles is confirmed by the presence of functional groups in FTIR spectra. XRD shows the formation of high purity Nickel (II) chloride nanoparticles. SEM analysis reveals the average particle size of the synthesized Nickel (II) Chlorideto be 59 nm. TGA analysis confirms the thermal stability of nanoparticles (nearly 35% remained after 900°C).

## ACKNOWLEDGEMENTS

We gratefully acknowledge the financial support from the Research Council of Imam Khomeini International University and Abhar Payam Noor University. This work was supported by Takestan Branch, Islamic Azad University.

## REFERENCES


[1] Rössler A., Georgios Skillas Sotiris E., Pratsinis S., (2001), Nanopartikel – Materialien der Zukunft: Maßgeschneiderte Werkstoffe. *Chemie in unserer Zeit*. 35: 32-41.

[2] Stefan E., (2004), Chemische Technik. Prozesseund Produkte, 5.Aufl. Herausgegeben vonRoland Dittmeyer, Wilhelm Keim, GerhardKreysa und Alfred Oberholz. *Angewandt Chemie*. 116: 5687-5788.

[3] Merisko-Liversidge E., Liversidge G. G., Cooper E. R., (2003), Nanosizing: A formulation approach for poorly-water-soluble compounds. *Eur. J. Pharm. Sci*. 18: 113–120.

[4] Chen X., Young T. J., Sarkari M., Williams 3rd., R. O, Johnston K. P., (2002), Enhanceddrug dissolution using evaporation precipitation into aqueous solution. *Int.J. Pharm*. 242: 3–14.

[5] Datta M. K., Pabi S. K., Murty B. S., (2000), Thermal stability of nanocrystalline Ni Silicides synthesized by mechanical alloying. *Sci. Eng. A*. 284: 219–225.

[6] Kaupp G., Naimi-Jamal M. R., Ren H., Zoz H., (2003), In Advanced Technologies Based on Self-Propogating and Mechanochemical Reactions for Environmental Protection, Cao G., Delogu F., Orru R., Ed.; *Research Signpost: Kerala. India*. 4: 83-100.

[7] Kaupp G., (2003), Solid-state molecular syntheses: complete reactions without auxiliaries based on the new solid-state mechanism. *Cryst. Eng. Comm*. 5: 117-133.

[8] Naimi-Jamal M. R., Kaupp G., (2005), Quantitative evaluation of nanoindents: Do we need more reliable mechanical parameters for the characterization of materials. 96: 1226-1236.

[9] Kaupp G., Schmeyers J., Naimi-Jamal M. R.,Zoz H., Ren H., (2002), Reactive milling with the Simoloyer R: Environmentally benign quantitative reactions without solvents and wastes. *Chem. Engin. Sci*. 57: 763-765.

[10] Kaupp G., Naimi-Jamal M. R., Ren H., Zoz H., (2002), Environmentally Protecting Reactive Milling. *Chemie Technik*. 31: 206-208.

[11] Krupp G. U., Mey K., Giertler A., Kaupp G., Benz H. U., Zoz H., (2012), Development of ultrafine-grained Zinc-ferrite-strengthened aluminum alloys by high-energy milling. *Rev. Adv. Mater. Sci*. 31: 19-24.

[12] Kaupp G., (2011), Reactive milling with metals for environmentally benign sustainable production, Highlight. *Cryst. Eng. Comm*. 13: 3108-3121.

[13] Kaupp G., (2009), Mechanochemistry: the varied applications of mechanical bond-breaking. *Cryst. Eng. Comm*. 11: 388-403.

[14] Kaupp G., Naimi-Jamal M. R., Schmeyers J., (2003), Solvent-free Knoevenagel condensations and Michael additions in the solid state and in the melt with quantitative yield. *Tetrahedron*. 59: 3753-3760.

[15] Kaupp G., Naimi-Jamal M. R., Stepanenko V., (2003), Waste-free Solid-state Protection of Diols, Diamines, Amino Acids and Polyols width Phenylboronic. *Acid Chem. Eur. J*. 9: 4156-4161.

[16] Komatsu K., (2005), The Mechanochemical Solid-State Reaction of Fullerenes. *Curr. Chem*. 254: 185 – 206.

[17] Zhang J., Gao J., Xia J., Wang G. W., (2005), Solvent-free mechanochemical and one-pot reductive benzylizations of malononitrile and 4-methylaniline using Hantzsch 1,4 dihydropyridine as the reductant. *Org. Biomol. Chem*. 3: 1617 – 1619.

[18] Balema V. P., Wiench J. W., Pruski M., Pecharsky V. K., (2002), Mechanically Induced Solid-State Generation of Phosphorus Ylides and the Solvent-Free Wittig Reaction. *J. Am. Chem. Soc*. 124: 6244 – 6245.

[19] Tullberg E., Schacher F., Peters D., Frejd T., (2006), Solvent-Free Heck-Jeffery Reactions under Ball-Milling Conditions Applied to the Synthesis of Unnatural Amino Acids Precursors and Indoles. *Synthesis*. 7: 1183-1189.

[20] Tullberg E., Peters D., Frejd T., (2004), The Heck reaction under ball-milling conditions. *J. Organomet. Chem*. 689: 3778-3781.

[21] Bang J. H., Suslick K. S., (2010), Applications of ultrasound to the synthesis of nanostructured. *Mat. Adv. Mater*. 22: 1039–1059.

[22] Darezereshki E., (2010), Synthesis of maghemite ($\alpha$-$Fe_2O_3$) nanoparticles by wet chemical method at room temperature. *Mater. Lett*. 64: 1471–1472.

**Cite this article as:** Z. Rahmani & Sh. Ghamamy: Synthesis and characterization of Nickel (II) Chloride nanoparticles with the study of their thermal behavior.
***Int. J. Nano Dimens.*** 6 (4): 401-407, Autumn 2015.