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Synthesis and identification of nanoparticles cobalt (II) bromide by ball mill method

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ABSTRACT

Synthesis, identification and thermal behavior studies of nanoparticles cobalt (II) bromide has been studied in this research. Cobalt (II) bromide was synthesized by planetary high-energy ball mill. The general formula of this compound is $CoBr_2.6H_2O$. 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. This compound has many applications in mineral synthesis as a catalyst. The nanoparticles of $CoBr_2.6H_2O$ synthesized with smaller size of 35 nm, and its SEM images show that the morphology of particles surface is as like as layer. Also, the thermal behavior of these nanoparticles is considered by using of DTA /TGA thermal analysis.

Keywords: *Synthesis; Identification; Cobalt (II) bromide; Mill device; X-ray diffraction; Scanning electron microscopy (SEM); Thermal behavior.*

INTRODUCTION

Cobalt (II) bromide ($CoBr_2.6H_2O$), is an inorganic compound. It use frequently as a catalyst in some processes. When anhydrous, cobalt (II) bromide appears as green crystals. The hexahydrate loses four waters of crystallization molecules at 100 °C forming dehydrate. Nanoparticles of cobalt (II) bromide may be prepared through many processes but in this work top to down approach was considered. In the manufacturing of functional inorganic materials, "grinding" can be cited as an important unit operation. Grinding operations do not simply grind materials. They are used for the purpose of mixing, transporting, promoting physical properties and heat transfer, preprocessing for recovery of valuable materials, expression of functions and the like. 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.

The materials are ground by moving the container. Because the structure of ball mills is simple and it is easy to operate, and so they are widely used. However, designing these devices and selecting conditions depend in many ways on empirical knowledge, and they have not been sufficiently systematized. Therefore, to scale-up these devices is not always easy, and collecting data requires a lot of effort and cost. Reaction was performed in a mechanical manner by co-grinding the reactants with agate milling balls using a planetary ball mill as the source for alternative energy input [1-15]. Developed by Benjamin, mechanical alloying (MA) is an alternative technique for the fabrication of powder particles [16]. The powder mixture is mechanically ball milled using a high-energy ball mill by which different alloys, ceramics, composites and amorphous materials can be synthesized [17-18].

In this work, nanoparticles of $CoBr_2.6H_2O$ compound were synthesized in planetary high-energy ball mill and it's characterize and thermal behavior were studied.

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 K Br 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 Kα radiation. Sizes of selected samples were estimated using the Scherer method. For identification a scanning electron microscope samples were gold coated.

Synthesis of nanoparticles cobalt (II) bromide

First: about 5 grams of $CoCl_2.6H_2O$ was weighed and placed in the oven at a temperature 110° C for 30 minutes to completely dry out humidity and therefore was cooled to room temperature.

Second: $CoBr_2.6H_2O$ powder 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.

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 was characterized and compared via FT-IR analysis. FT-IR spectrometer at room temperature is in the range from 400 to 4000$cm^{-1.}$

RESULTS AND DISCUSSION

Analysis of infrared spectroscope (IR)

In this paper, we reported the synthesis nanoparticles cobalt (II) bromide. After preparing nanoparticles, it was characterized by IR. (Figure 1).

$CoBr_2.6H_2O$: IR (KBr): ν (Co-Br): 560.08, ν (O-H): 1618. 85, ν (O-H): 3197.22 cm^{-1}.

Analysis of X-ray diffraction (XRD)

Figure 2 shows the XRD pattern of nanoparticles prepared by the planetary high-energy ball mill process. Estimated from the Debye-scherrer 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, λ is the X-ray wavelength (0.154 nm), and θ and β are the diffraction angle and full width at half maximum of an observed peak, respectively).

- **Calculations**

FWHM = 0.2362° β = 0.00412
cosθ = 0.974 2θ = 26.2072, θ = 13.1036
D = 0.9 × 0.154 / 0.004013 D = 34.53 nm

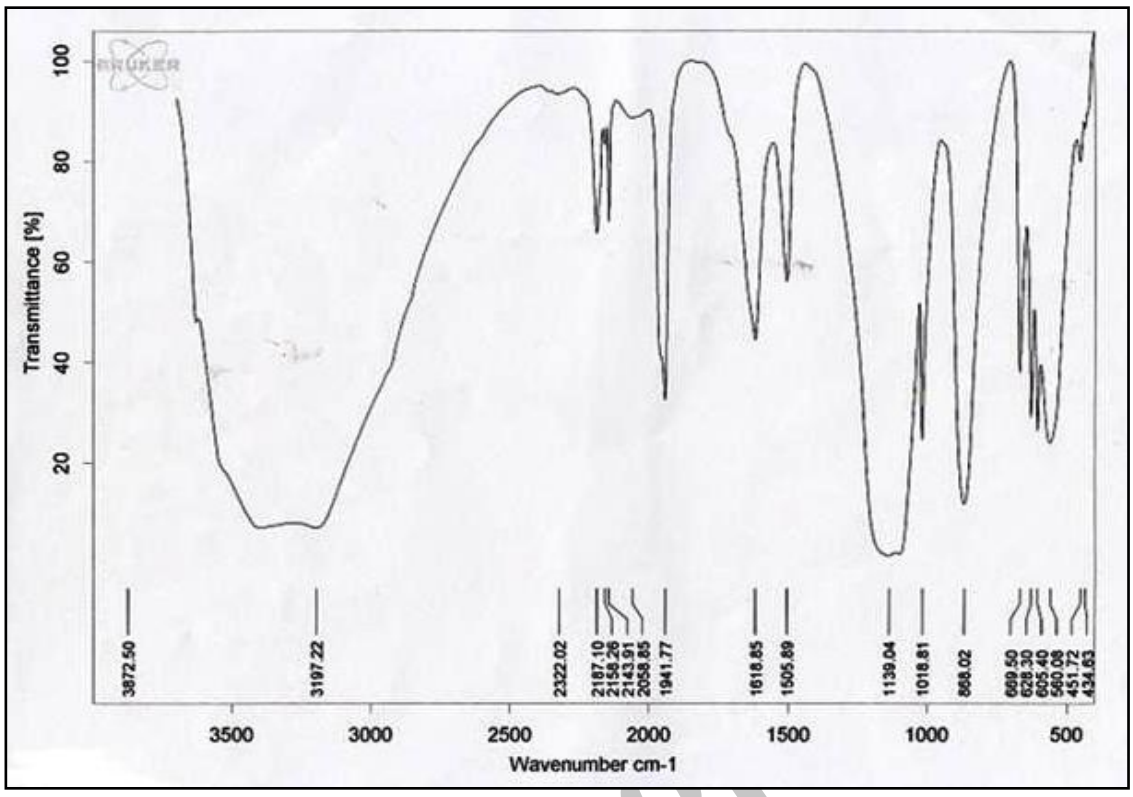

Fig. 1. FT-IR spectra of $CoBr_2.6H_2O$ nanoparticles

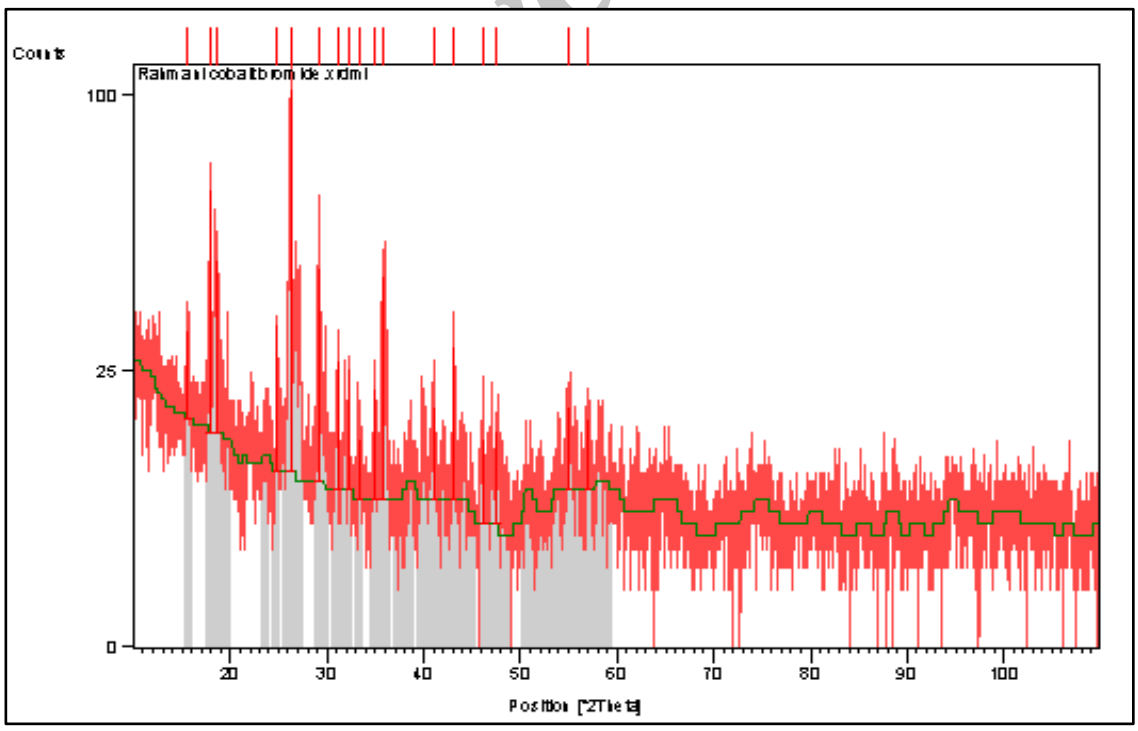

Fig. 2. The XRD pattern of $CoBr_2.6H_2O$ nanoparticles

Analysis of scanning electron microscope (SEM)

In the present study, the particle size of the cobalt (II) bromide prepared by ball mill technique was found to be 35 nm. Scanning electron microscopy (SEM) of the sample was carried out to estimate the surface morphology of the sample. XRD and SEM together provide exact knowledge regarding the of the synthesized cobalt (II) bromide sample. Figures 3 & 4, shows the SEM images of the synthesized cobalt (II) bromide sample.

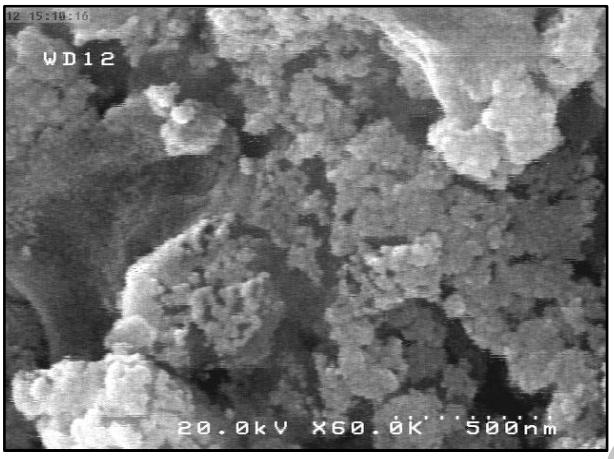

Fig. 3. SEM images of the $CoBr_2.6H_2O$ nanoparticles

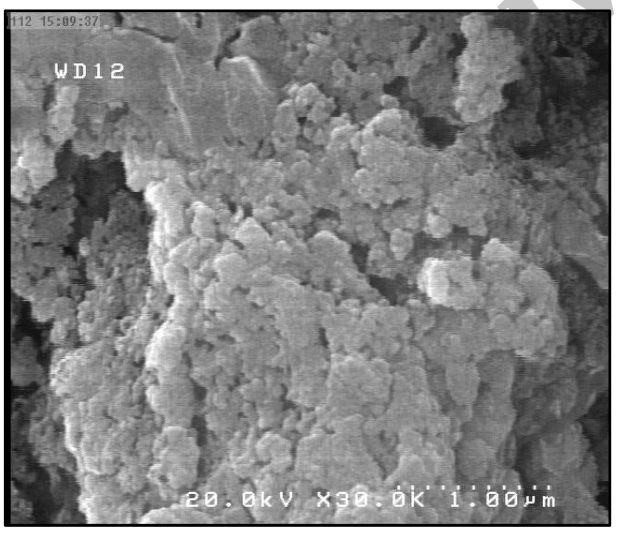

Fig. 4. SEM images of the $CoBr_2.6H_2O$ nanoparticles

Study the thermal behavior

Thermal behavior of nanoparticles cobalt (II) bromide was investigated using thermo gravimetric analysis (TGA). Thermo gravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. The TGA profile reveals four weight changes around 50, 220, 510 and 720 °C. The first degradation is attributed to the removal of water from the surface (mass loss of about 10%). The second degradation is attributed to 9% mass reduction in this region. The third degradation results in an additional mass loss of about 8%. The fourth degradation from 720 °C to 850 °C results in an additional mass loss of about 30%. Figure 5 shows the thermo gravimetric analysis curve of cobalt (II) bromide.

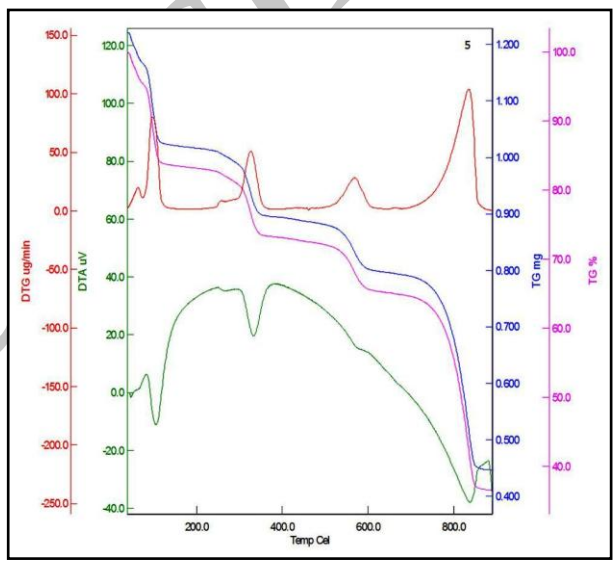

Fig. 5. Thermal behavior of the $CoBr_2.6H_2O$ nanoparticles

CONCLUSIONS

In this research the detail studied the synthesis, characterization**,** and thermal behavior of nanoparticles cobalt (II) bromide. In summary, the molecular structure of nanoparticles 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 cobalt (II) bromide powders. TGA analysis reveals that the synthesized cobalt (II) bromide nanoparticles were thermally

stable up to 900 °C. SEM image shows that the particles of the synthesized sample are in nanometer range.

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