Mechanical dry method for synthesis of cobalt metal nano particles

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Abstract:

Cobalt-chloride coordination used as simple solid state method to synthesized in the solid phase, product of magnetic cobalt nano hybride was produce, the complexes undergoes a reversible phase transition, changing color at room temperature. The structure and the morphology of the produced materials was studied using FTIR, SEM and XRD spectroscopy, magnetic susceptibility measurements was done, the need of new magnetic materials is distinct interest of researchers on the study of magnetic materials, complexes transform to nanoparticles electromagnetic Cobalt metals were successfully synthesized by dry mechano method, decomposition synthesis was successfully occur. Structure morphology and magnetic properties were studied and reported. properties are characterized by X-ray diffract metric and scanning electronic microscope, SEM

Introduction:

The most important of these are phase transition temperature, irregular Color change, Leads to a change in the structural phase.

As in this phase Change is controlled by the thermodynamics of the system resulted from the mechanical process this is a kind of second order reversible or irreversible (1). So far, a series of a phase change Co(II) complexes containing chloride or amine ligand have been synthesized and characterized by different technics such as XRD, FT, SEM and susceptibility magnetic (2).

As a phase transfer has been widely investigated by different of researchers
Groups, it was generally accepted that there must be a change of Nanoscale structure creates a rise of phase transition and a modify of color Thermo process(3). observed in Co(II)complexes is due to a change in geometrical and coordination numbers.

Oxide synthesis method is really a good approach when considering a synthesis tool. Installation of chemical synthesis, which has been widely used Synthesize different solid phase metal ions to eliminate unnecessarily complex problems(4). Need for new magnetic materials seems to be of particular interest to researcher in this study(5), nano magnetic cobalt metals synthesis.

The far more important of these are the irregular, phase change temperature Color changes, leads to significant liquid state(6). As Change is driven by the system thermodynamics occurring from the mechanical process throughout this step(7), it is a kind of reversible or irreversible second order (8). A sequence of phase change Co(II) complexes that contain chloride ligand. (9). As a phase transfer has been widely investigated by different of researchers Groups(10), this has been commonly agreed that maybe a change in nanoscale structural system creates an increase in the transition phase and a change in color Changing noted in complexes of the Co(II) is due to a change in coordinating.

numbers and positions. Selecting dry synthesis Process of metal reduction synthesis method is a successful approach(11). of chemical synthesis, that has been extensively used Synthesize various solid phase metal ions to prevent unnecessary complex issues.

Specially all occasions in electromagnetic of compounds.

Heating by the mechanical method detriment of a successful inorganic conversion (12). In addition to the benefits of superior solid state production over dry process, chloride and amines complexes of cobalt(II) are believed to be paramagnetic in mechano heating system.

By many preparation methods like chemical co-precipitation [13], hydrothermal [14], sol-gel (13) combustion (14) etc. have been reported. In the literatures, mechano cobalt magnetic study in this work.

Materials and method:

1- Take 2 g of CoCl$_2$.6H$_2$O and crush it slightly using the mortar manually, then 0.31 g of NaBH$_4$ sodium Borhydride was added and crushed with it for 5 minutes, then 0.33 g of NaOH was added to the previous powder and crushed for 20 minutes. Then
the powder and grinning together, it distilled water several times to washing from the impurities. Finally wash with acetone and leave to dry.

2- Take 2 grams of [Co (NH₃)₆] Cl₃ and crush a little using the mortar manually, after that, 0.567 grams of NaBH₄ sodium boro hydride was added and grinding for 5 minutes, then 0.12 grams of NaOH was added to the previous powder and crushed for 20 minutes. The powder was washed it with distilled water several times to remove the impurities. Washed with acetone leave to dry.

3- 2 g of [Co (NH₃)₅NO₂] Cl₂ and crush using the mortar manually, then add 0.57 g from grams of NaBH₄ sodium boro hydride crush for 5 minutes, 0.2 g of NaOH was added and crushed with it for 20 minutes. Then final powder was washed with distilled water several times to get rid of the impurities. Then wash with acetone and leave to dry.

According the following reactions equations:

\[
\text{NaBH}_4 + \text{CoCl}_2 \cdot 6\text{H}_2\text{O} + \text{NaOH} \rightarrow \text{Co} + \text{NaCl} + 2\text{H}_2 + \text{B(OH)}_3 + 2\text{H}_2\text{O}
\]

\[
\text{NaBH}_4 + 2[\text{Co(NH}_3)_6]\text{Cl}_3 + 5\text{NaOH} \rightarrow 2\text{Co} + 12\text{NH}_3 + 6\text{NaCl} + \text{H}_2 + \text{B(OH)}_3 + 2\text{H}_2\text{O}
\]

\[
\text{NaBH}_4 + 2[\text{Co(NH}_3)_5\text{NO}_2]\text{Cl}_2 + 3\text{NaOH} \rightarrow 2\text{Co} + 10\text{NH}_3 + 4\text{NaCl} + 2\text{NO}_2 + 2\text{H}_2 + \text{B(OH)}_3
\]

Results and discussion:

This a study was done using : PANalytical X’pert Pro diffractometer with Cu-Kα radiation (\(\lambda = 1.5405\text{Å}\)) performed structural characterization of the samples in the 2nd range of 10-80, in angular steps of 0.02°. The average particle size D was calculated using the Scherrer formula given by the line extension of (14) reflection.

\[
D = \frac{K \pi}{\beta \cos \theta}
\]

Where \(\beta\) is the width of the angular line at half the maximum intensity and \(\theta\) is the angle for that peak. The lattice constant values ‘a’ and ‘c’ and the unit cell volume ‘V’ were calculated using the following equations. Particular morphology and size were studied using SEM (Fig. 2). The particle size of each sample was analyzed from SEM (Fig. 2). With the help of Image software, the particle size of each sample was analyzed from the SEM images and the values were found to be comparable to calculated values.

Magnetic investigates: The properties were tested using a vibrating sample magnetometer at room temperature. Analyses were taken by applying field inside -20
K→20 K radius. Measured as well as the effects are magnetization, saturation and magnetic permeability.

Results and Discussions:

Fig. 1 xrd of the cobalt nano particles prepared from the CoCl$_2$.6H$_2$O, Co(NH$_3$)$_6$Cl$_3$ and Co(NH$_3$)$_5$NO$_2$Cl$_2$

Shows the x-ray diffractions from the plans (hkl), (311), the sharp diffractions for the plane (400), the planes (101), (111) and (110) in all of the synthesized nano materials from the different starting materials the spectrums were identical(15).

Surface morphology analysis: Sample SEM images are seen in Figures 2.
This is obvious from particles sizes between 17-33nm, exhibits strong peaks for CoO in comparison with weak Co$_3$O$_4$ peaks represented by the pattern, these diffractograms show that the initially synthesized metallic cobalt nanoparticles were oxidized to CoO and Co3O4 nanoparticles Using the Scherrer formula.

Fig.2: SEM image and EDX shows the distributions of particles sizes of the nano cobalt synthesized from the complexes [Co(NH$_3$)$_5$NO$_2$]Cl$_2$

Fig.3  SEM image and EDX shows the distributions of particles sizes of the nano cobalt synthesized from the complexes CoCl$_2$.6H$_2$O .
Figure 4: SEM image and EDX shows the distributions of particles sizes of the nano cobalt synthesized from the complexes synthesized from Co(NH$_3$)$_6$Cl$_3$

**The susceptibility magnetic:**

Hysteresis decreases with the resulted nano particles from the starting materials used in the synthesis of nano cobalt. That may be part of the platform habits with the CoCl$_2$.6H$_2$O and minimum with complexes of cobalt amines, Co(NH$_3$)$_6$Cl$_3$ and[Co(NH$_3$)$_5$NO$_2$]Cl$_2$ the evolution of the susceptibilities according to the coordination number of starting materials(16)
Fig 5: shows the spectrum of FTIR of the synthesized products starting from different complexes

The spectra shows the valence vibrations of starting materials like vibrations of C-N, N-H, between 3700-2900 cm\(^{-1}\) and all the symmetric and asymmetric vibrations of the starting material like amines, and also the disappearance of the above vibrations in the case of forming cobalt nano particles

The vibrations situated between 500-350 cm\(^{-1}\) are attributed to the metals of cobalt and its oxides

CONCLUSIONS

Using mechanical technique, a series of Cobalt substituted complexes samples were prepared using the dry method for the synthesis, the resulted products were characterized by different techniques to proves the magnetic properties and the morphology of the nano cobalt. The X-ray diffraction analysis indicate a hexagonal structure formation with space group P63/mmc, without any trace of secondary phases.

Substitute is attributed to variations in oxidation state of Co\(^{3+}\) ion and Fe\(^{3+}\) ion. Greater volume of Cobalt has influenced the particle morphology. With increase of the Cobalt element, agglomeration is decreased. Increasing Co\(^{3+}\) is confirmed from SEM
That perhaps the particles are Cobalt of Nano particle. It was found that replacing Fe with paramagnetic Co contributes to higher saturation magnetization, magnetic permeability. It's also clear from the average concentration ratio values shows good . .hysteresis and hard magnetic properties out of three samples.

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