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Chemically Synthesized of Cobalt Oxide (Co_3O_4) Nanoparticles by Self-Propagating Low Temperature Combustion method And its Characterizations

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Abstract — *Cobalt Oxide (Co_3O_4) nanoparticles have been synthesized by self-propagating low temperature Combustion synthesis (SPLTC) method using Cobalt salt with polyethylene glycol as fuel. As synthesized Co_3O_4 nanoparticles was characterized by employing Fourier-transform infrared spectroscopy (FTIR), Scanning Electron Microscopy (FESEM), and X-ray diffraction (XRD) confirmed particles are in nanosized.*

Keywords- *Cobalt Oxide (Co_3O_4) nanoparticles self-propagating low temperature Combustion synthesis, Fourier-transform infrared spectroscopy, Field Emission Scanning Electron Microscopy.*

I. INTRODUCTION

Nanoscale oxide particles of transition metals are gaining continuous importance for various applications such as catalysts, passive electronic components and metal oxide materials [1-3]. Due to their ultra-small size, nanoparticles exhibit novel material properties that are suggestively different from those of their bulk ones. Cobalt oxide nanoparticles with a uniform size and well dispersion is necessary for numerous applications in designing ceramic, magnetic, electro chromic and heterogeneous catalytic materials [5]. Several researchers have prepared Cobalt oxide nanoparticles by various methods like sol–gel [4], surfactant-mediated synthesis thermal decomposition, polymer-matrix assisted synthesis and spray-pyrolysis [6]. ultrasonic radiation, hydrothermal synthesis, carbonyl method, laser chemical method, pyrolysis by microwave, precipitation-calcination, micro emulsion method, and so on [7-9]. However, to the best of our knowledge, most of the reported experimental techniques for the synthesis of nanopowders are still limited in laboratory scale due to some unresolved problems, such as special conditions, tedious processes, complex apparatus, low yield, and high cost [10-14]. From a useful viewpoint, it is vital to develop a way to manufacture high-quality nanopowders at high throughput with low cost [15-17].

Current investigation is a self-propagating low temperature combustion route using cobalt oxalate precursor for the synthesis of Cobalt Oxide. Polyethylene glycol was used as a fuel for the precursor. The characterization study of as prepared Cobalt oxide was undertaken by employing FTIR, XRD, FESEM.

II. MATERIALS AND METHODS

Cobalt Sulphate and oxalic acid used are AR grade chemicals. Polyethylene glycol of molecular weight 6,000 was obtained commercially (Merck Chemicals). The double distilled water is used is for preparation of solution. The Cobalt oxide is synthesized through self-propagating low temperature combustion route, employing Cobalt oxalate as precursor. This precursor is prepared by dissolving equimolar quantity of Cobalt sulphate heptahydrate and oxalic acid in minimum amount of water. This mixture was well stirred in a three-necked flask. The Light green precipitate of Cobalt oxalate dihydrate obtained was filtered through sintered glass funnel and washed with double distilled water. Finally, it was washed with dry acetone and dried under vacuum.

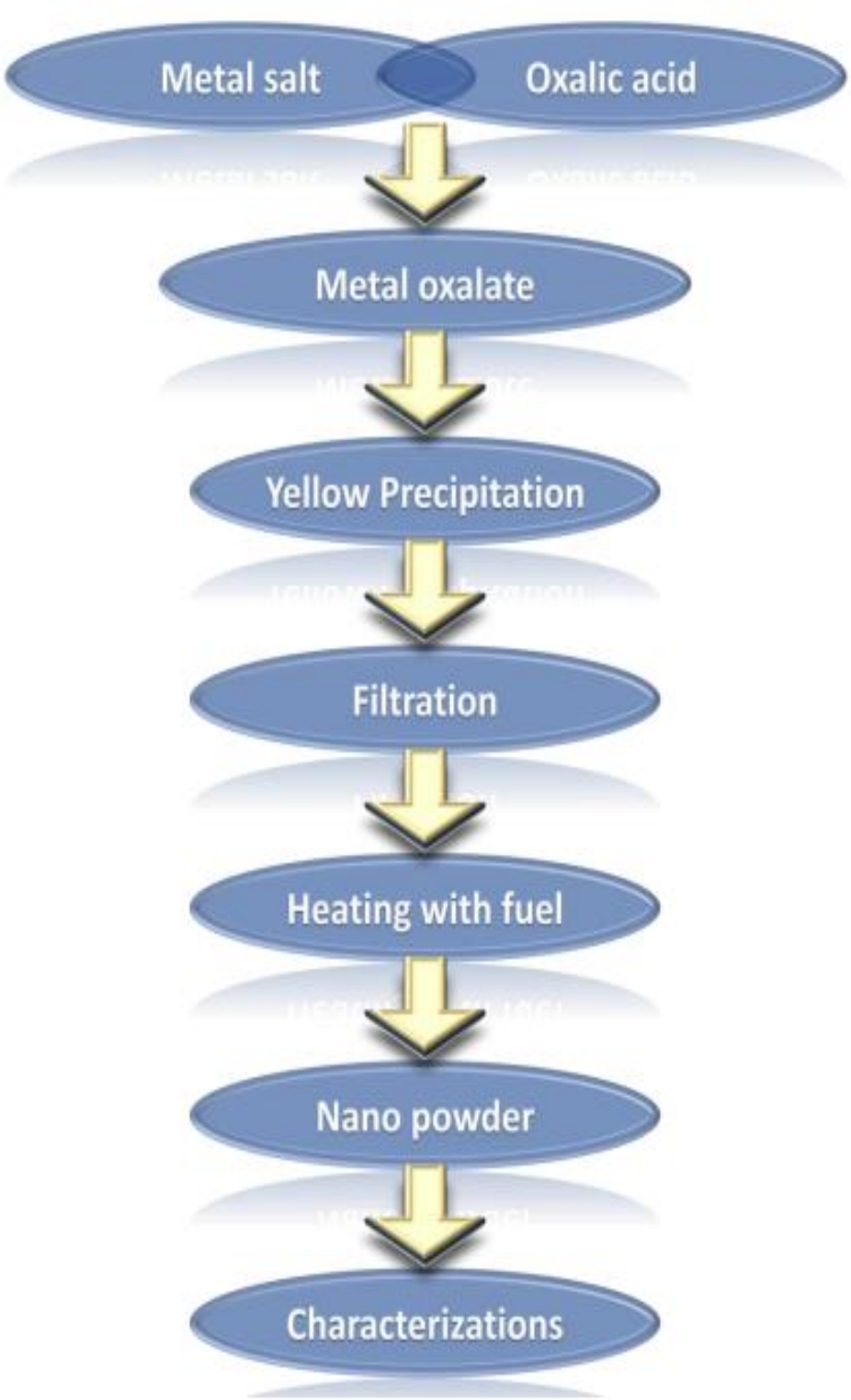

Fig 1. Flow diagram of self-propagating low temperature Combustion synthesis to characterizations of cobalt oxide

Thermal decomposition of Cobalt Oxalate precursor with a fuel leads to the formation of high surface area Cobalt oxide. The above prepared Cobalt Oxalate was mixed with Polyethylene glycol (PEG) in the weight ratio 1:5 (Vijay et al 2000; Mallikarjuna et al 2003) and ground well in a pestle and mortar. The resultant solid was placed in a crucible and heated in air. It was observed that initially PEG melted, then frothed and finally ignited to give Cobalt oxide as a residue. On cooling to room temperature, no traces of carbon impurities were observed in the final residue of Cobalt oxide. As the reaction is fast, i.e. going to completion within 10 min, and ignites auto-catalytically, the exact temperature of the reaction could not be measured. However, using a thermocouple, the highest temperature of the reaction was found to be around 500°C. Fig 1. Shows the flow diagram of self-propagating low temperature Combustion synthesis to characterizations of cobalt oxide

III. CHARACTERIZATION TECHNIQUES

Fourier transformed infrared spectroscopy (FTIR) measurements carried out on a Perkin-Elmer spectrum one, instrument at a spectral resolution of 4 cm−1 in KBr pellets. The X-ray diffraction patterns were obtained employing a JEOL JDX-8p spectrometer using CuK radiation. The X-rays generator was operated at 30 kV. High purity silicon powder was used as an internal standard. The FESEM was operated at 20 kV. The samples were made conducting by the sputtering of gold using a Poloron DC "sputtering unit" operated at 1.4 kV and 18-20 mA.

IV. RESULTS AND DISCUSSION

Figure 2 shows the FTIR spectra of Cobalt oxide nanoparticles, which showed substantial absorption peaks. The broad absorption band in the region of 650–710 cm−1 is assigned to Cobalt and Oxygen stretching vibration mode the broadness of the absorption band indicates that the Cobalt oxide powders are nanocrystals. The size of samples used in this study was much less than the bulks form Cobalt oxide, so that Cobalt oxide nanoparticles had its FTIR peak of Cobalt and Oxygen stretching vibration and shifted to blue direction. Due to their quantum size effect and spherical nanostructures, the FTIR absorption of Cobalt oxide nanoparticles is blue-shifted compared to that of the bulk form. Besides the cobalt single bond oxygen vibration, it could be seen from Figure 2 that the broad absorption band centered at 3440 cm^{-1} is attributable to the band oxygen single bond hydrogen stretching vibrations and the weak band near 1635 cm^{-1} is assigned to hydrogen single bond oxygen single bond hydrogen bending vibrations mode were also presented due to the adsorption of water in air when FTIR sample disks were prepared in an open air. These observations provided the evidence to the effect of hydration in the structure. Meanwhile, it implied the presence of hydroxyl in the precursor, and the broad absorption around 767 cm^{-1} is assigned to the band carbon double bond oxygen stretching vibrations. The serrated absorption bands in the region of 1000– 1500 cm^{-1} are assigned to the oxygen single bond carbon double bond oxygen symmetric and asymmetric stretching vibrations and the carbon single bond oxygen stretching vibration, but the intensity of the band has weakened, which indicated that the ultra-fine powers tend to strong physically absorption to water molecule and carbon dioxide.

The purity and crystallinity of the as-synthesized Cobalt oxide nanoparticles were examined by using powder X-ray diffraction (XRD) as shown in Figure 3. It can be seen from Figure 1 that the diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The peaks positions appearing at 2θ is 37.21◦, 43.22◦, 63.10◦, and 79.39◦ can be readily indexed as (101), (012), (110), and (006) crystal planes of the bulk Cobalt oxide, respectively. All these diffraction peaks can be perfectly indexed to the face-centered cubic (FCC) crystalline structure of Cobalt oxide, which is in accordance with that of the standard spectrum. The XRD pattern shows that the samples are single phase and no any other impurities distinct diffraction peak except the characteristic peaks of FCC phase Cobalt oxide was detected. This result shows that the physical phases of the Cobalt oxide nanoparticles have higher purity prepared in this work. The Cobalt oxide lattice constant calculated from the XRD data is 4.1729 A°, which is in good agreement with the reported data.

The particles are mostly irregular leaf like shape with a nanosized range 55-80 nm. Morphology of cobalt oxide nanoparticles was studied by scanning Some particles are found as agglomerated and formed like leaf like structure surface are observed. Field Emission Scanning Electron Microscopy (FESEM). Figure 4 shows the nano particles

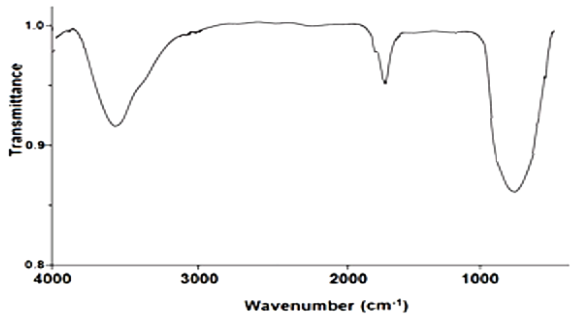

Fig 2. Fourier-transform infrared spectroscopy of Cobalt oxide nanoparticle

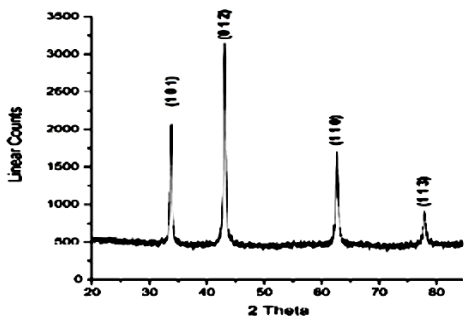

Fig 3. X-ray diffraction pattern of Cobalt oxide nanoparticle

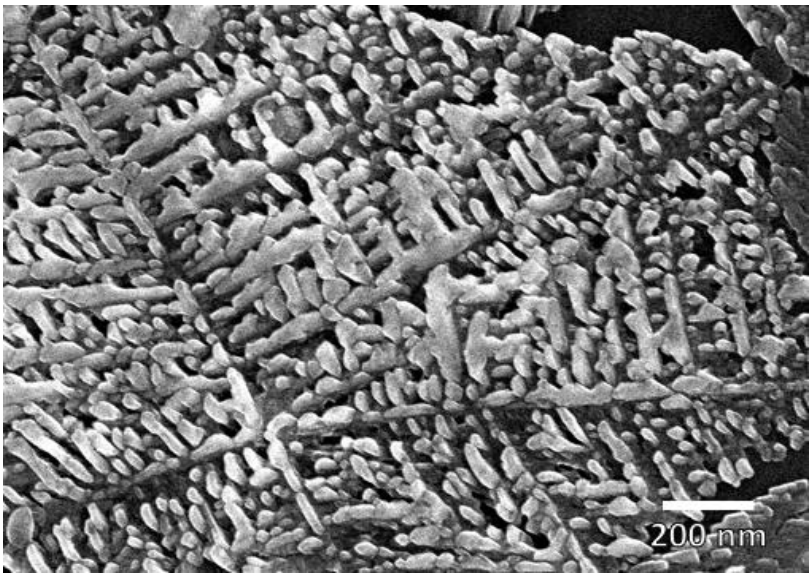

Fig 4. Field Emission Scanning Electron microscopy image of Cobalt oxide nanoparticle

CONCLUSION

Nanosized cobalt oxide particles were synthesized using self-propagating low-temperature combustion route because of its simplicity and easy scale up. The results obtained from crystallite size from FTIR, XRD and FESEM images confirm the nanocrystalline nature of the synthesized materials. The results recommend that the self-propagating low-temperature combustion method is an effective pathway for producing high-quality cobalt oxide nanosized powder.

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