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Microwave combustion method: Effect of starch, urea and glycine as processing fuels in the Co_3O_4 nanostructures

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ABSTRACT

A novel synthesis method is presented for the preparation of nanosized-semiconductor cobalt oxide formed successively from a single-source solid various fuel (starch, urea and glycine). The as obtained samples were characterized by the X-ray diffraction, scanning electron microscopy, transmission electron microscopy; diffuse reflectance spectroscopy (DRS) and photoluminescence (PL) spectroscopy and vibrating sample magnetometer (VSM). XRD pattern confirmed the formation of cubic phase Co_3O_4. The materialization of Co_3O_4 nanoparticles is confirmed by HR-SEM and HR-TEM. The optical absorption and photoluminescence emissions were determined by DRS and PL spectra, respectively. The relatively high saturation magnetization (starch) of Co_3O_4 shows that it is ferromagnetic and low saturation magnetization (urea and glycine) confirms the super paramagnetic behavior.

1. Introduction

Nanoscience and nanotechnology is attracting the attention of the researchers due to their multipurpose uses that the covers approximately all field of life. Nanometer scale semiconductor crystallites, also referred to as nanoparticles or quantum dots, have been extensively studied to explore their unique properties and the potential applications. The significant features of the catalytic system are the desired band gap, suitable morphology, high surface area, stability and reusability [1,2]. Developing new routes to synthesize nanocrystalline metal oxides are challenging tasks for solid-state chemists and the materials scientists. During the past decade, synthesis of nanocrystalline particles is the basic condition for construction of see-through ceramics, because nanopowders exhibit high surface area and the offer low temperature for achieving nano and microstructures. Extremely pure phase, sufficient mono discrete particles, with nearby to spherical shape and size are desirable to get dense ceramics [3].

Cobalt oxides is a ceramic material, known for its excellent catalysis, magnetic, mechanical, electrical, thermal, and optical properties [4]. Cobalt oxides are produced by the techniques, such as, hydrothermal method, co precipitation, microwave combustion, sol–gel, solvothermal and the electrodeposition [5,6]. The use of microwave in the combustion synthesis has several advantages. The foremost of which are the shortened periods of synthesis, enhanced reaction kinetics, and the reactant selectivity, during the energy transfer from the microwave field, which assists the stabilization of the metastable

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phase of the material. Therefore, it is possible to control both kinetic and thermodynamic factors of the chemical reactions using microwave in the combustion process.

Combustion synthesis is a one step process: formation of originator and then auto explosion. This auto-ignition is frequently termed as self-propagating microwave synthesis. The processing parameters that the effect the properties of final product are fuel to oxidizer ratio, type of fuel, amount of fuel percentage, ignition temperature, water content in precursor, pH of the solution. A good fuel should possess the properties like non-violent reactions and without evolution of toxic gases [7,8]. A number of fuels like starch, urea and glycine are being used to prepare technologically important metal oxides. These fuels achieve two purposes: throughout combustion forms CO_2, H_2O and release heat in exothermic procedure and forms developments with metal ions smoothing homogeneous mixing of the cations in solution [9,10].

The properties of nanoparticles are strongly dependent on its morphology. Therefore, specific attention must be given to the excellent of its preparation. The fuel also forms complexes with the metal ions, facilitating the homogeneous mixing of the cations in the solution. In recent years, different types of fuels can also be used in the combustion synthesis. Pure and homogenous cobalt oxide powders have been synthesized through new single fuels mixture of low temperature flame fuels, represents a suitable alternative to control the flame temperature, and, implicit the size distribution, surface area and agglomeration degree of the particles of the final product [11].

In the past years, changed physical and chemical methods were used to prepare Co_3O_4 which include possible to control the size and shape of nanoparticles in solution by variable synthesis parameters, such as, reaction temperature, time, solution composition and additives, such as, soft or hard fuels. Currently, single-source solid various fuel (starch, urea and glycine) combustion reaction is one of the most accessible, fast and low-energy soft methods for the synthesis of simple and mixed oxide materials. Pure and homogenous Co_3O_4 powders have been synthesized through new single combustion methods, in which the oxidants are the corresponding nitrate salts of the metals, while starch, urea and glycine are used as reducing organic fuels. As a consequence, that the particles morphology, crystalline phase, surface area and other particular properties of the final oxide products could be directed formed. A fuels mixture solution of low temperature flame fuels, represents a suitable alternative to control the flame temperature and implicit the size distribution, surface area and agglomeration degree of the particles of the (Co_3O_4) final product. Consequently, from both environmental and economic points of view, preparation of the modified Co_3O_4 magnetic nanoparticles via a simple method and without calcination temperature [12].

We have microwave method used for the preparation of various fuels into synthesis of nano sized cobalt oxide, which has many applications in several cutting edge areas of science. The yield of the concluding products was effectively quantitative and the analytically pure. Fast reaction, less expensive, high yield of nano cobalt oxide and the fewer requirements of apparatus/instruments are the added advantage of our developed process.

Compare to microwave combustion method route, the conventional combustion method have some disadvantageous such as required long time and high temperature for calcinations to complete final products and low production rates. In microwave combustion approach, due to the direct interaction of microwave combustion method to the material interior with homogeneously, this can help to produce such active nanomaterials within few minutes of time [13].

In the present research, here we demonstrate the synthesis of bulk quantity of inherent cobalt oxide of many combinations through a simple microwave method via different fuel starch, urea and glycine. The structure, morphology, optical and magnetic of the as-prepared cobalt oxide nanoparticles were investigated by XRD, HR-SEM, TEM,VSM, DRS and PL spectra.

2. Experimental section

2.1. Materials

Cobalt nitrate and different fuel starch, urea and glycine were used as the starting material, (Merck chemicals, India) and were used as received without further purification.

2.2. Synthesis of cobalt oxide

Cobalt nitrate was used as precursors and the different fuels starch, urea and glycine as a fuel for this reaction. The synthetic procedure is based on a redox reaction. The stoichiometries of metal nitrate salts and fuels (used single or in mixture) were chosen considering the total reducing (VR) and the oxidizing (VO) valences of the raw materials and were quantified in equivalence ratio equal with VR/VO ratio. In which the cobalt nitrate acts as an oxidizing reactant and fuel starch, urea and glycine as a reducing reactant. Stoichiometric amounts of cobalt nitrate and starch, urea and glycine were dissolved separately in 10 ml of de-ionized water and poured into a silica crucible and stirred for 15 min to obtain a clear solution. For the preparation of pure cobalt oxides by using the microwave combustion technique, the precursor mixture in starch, urea and glycine was placed into a domestic microwave oven and exposed to the microwave energy in a 2.45 GHz multimode cavity at 850 W for 10 min. After the completion of the reaction, the solid powder was obtained and then it was washed with ethanol and dried at 70 °C for 1 h and was labeled as cobalt oxides samples starch, urea and glycine for (C1,C2 and C3).

In synthesis part domestic microwave oven, the magnetron repeated use for synthesis for 1–15 times. In present study, Co_3O_4 prepared 1–3 times (all fuels) repeatability a similar level to that of the run cycles and yield is high. Furthermore,