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Solventless synthesis, morphology, structure and magnetic properties of iron oxide nanoparticles

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Abstract

In this study we report the solventless synthesis of iron oxide through thermal decomposition of acetyl ferrocene as well as its mixtures with maliec anhydride and characterization of the synthesized product by various comprehensive physical techniques. Morphology, size and structure of the reaction products were investigated by scanning electron microscopy, transmission electron microscopy and X-ray powder diffraction technique, respectively. Physical characterization techniques like FT-IR spectroscopy, dc magnetization study as well as ⁵⁷Fe Mössbauer spectroscopy were employed to characterize the magnetic property of the product. The results observed from these studies unequivocally established that the synthesized materials are hematite. Thermal decomposition has been studied with the help of thermogravimetry. Reaction pathway for synthesis of hematite has been proposed. It is noted that maliec anhydride in the solid reaction environment as well as the gaseous reaction atmosphere strongly affect the reaction yield as well as the particle size. In general, a method of preparing hematite nanoparticles through solventless thermal decomposition technique using organometallic compounds and the possible use of reaction promoter have been discussed in detail.

Keywords: Solid state synthesis; acetyl ferrocene; hematite; SEM/TEM; XRD; magnetization; Mössbauer study; reaction pathway

1. Introduction

Metal oxide magnetic materials have been the subject of intense scientific research owing to their unique physical characteristics. Iron oxides are the most useful among the ferromagnetic and ferrimagnetic metal oxides from the application point of view. Among different synthetic routes for preparing metal oxides, the technique of thermal decomposition of metal complexes becomes increasingly important mainly due to easy control of process conditions, purity, phase, composition, microstructure, etc. of the resultant products. Thus, among different reported synthetic methods for preparing metal oxides, thermal decomposition of organometallic compounds at relatively low temperatures becomes increasingly important [1-5]. So it is quite promising and facile to be applied into material industry. In case of thermal decomposition process for preparing metal oxides, preference is generally given to precursor compounds that decompose at low temperatures to minimize sintering of the resulting oxide and have minimum interfering side reactions or by-products (i.e., volatile compounds) [6]. Recently, usage of metal-organic compounds as precursors becomes more and more regular. Especially, metallocenes [7], metal complexes of acetyl acetonates [8], alkoxides [9], oxalates [2, 4, 5] or citrates [10] are used as typical organic precursors for this purpose. Thermal decomposition of iron-bearing organic precursors is a very popular way to produce iron oxides of different phases [11, 12] and has been demonstrated to be very successful in the preparation of iron oxides with controllable

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