

COMBUSTION SYNTHESIS OF NANOSIZED 5- Fe_2O_3 : STRUCTURE MORPHOLOGY AND BONDING

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ABSTRACT

A new combustion route for the synthesis of nanosized $\alpha\text{-Fe}_2\text{O}_3$ through a self-propagating combustion synthesis-using Poly (ethylene glycol) as a fuel is reported. The precursor, ferrous citrate is employed in the synthesis of Nanosized $\gamma\text{-Fe}_2\text{O}_3$. The structure, morphology and bonding of as synthesized $\alpha\text{-Fe}_2\text{O}_3$ sample are characterized by X-ray diffraction (XRD), Scanning electron micrograph (SEM) and infrared (IR) techniques respectively.

Keywords: Nanosized $\alpha\text{-Fe}_2\text{O}_3$ Poly (ethylene glycol), Combustion reaction and precursor.

INTRODUCTION

Interest has increased following the observation that the properties are strongly dependent on the size of particles with dramatic changes when ultrafine/nanometric sizes are acquired¹. The new synthetic routes for the preparation of Nanosized $\gamma\text{-Fe}_2\text{O}_3$ are under constant investigation and some of them include the ion-exchange reaction in $\alpha\text{-Fe}_2\text{O}_3$ with benzoic acid², co-precipitation³, thermal decomposition of metal hydrazine complexes⁴, through a wet chemical synthesis of successive hydrolysis, oxidation and dehydration of ferrous chloride to obtain as small as 5 nm particles⁵, selective oxidation of Fe^{2+} and Fe^{3+} ⁶, thermal decomposition of lipodocracite from solid solutions⁷, and thermal decomposition of metal carboxylate under the controlled conditions⁸, and through ore beneficiation⁹. Many of these methods involved in the synthesis of $\gamma\text{-Fe}_2\text{O}_3$ are tedious, some of them are time consuming with many reaction steps and often in many cases a small quantity of the material could be synthesized.

It is reported in literature that polyvinyl alcohol can be employed as an oxidizing agent along

with urea for the synthesis of other spinel ferrites through the combustion of citrate precursor¹⁰. However, this technique has some limitations viz., use of urea as a fuel, and also that polyvinyl alcohol has a wide variation of molecular weight, both of these parameters would influence the combustion to undergo as a highly exothermic reaction. Hence a new fuel which can also work as a controllable oxidant in a combustible reaction generating self-propagation is essential.

Present investigation is a new synthetic routes using new precursors for the synthesis of nanosized monophasic $\gamma\text{-Fe}_2\text{O}_3$. In the present study we have employed ferrous citrate along with polyethylene glycol as an oxidant in a fixed ratio of 1:5 through a self-propagation combustion reaction¹¹.

EXPERIMENTAL

MATERIAL AND METHODS

Polyethylene glycol with molecular weight 4,000 was obtained commercially. All the other chemicals employed were of AR grade.

Preparation

The precursor ferrous citrate was prepared by mixing equimolar solutions of ferrous ammonium sulphate and citric acid. The precipitate of ferrous citrate obtained was filtered through sintered glass crucible and was washed with cold oxygen free distilled water and finally with dry acetone and dried under vacuum. The above precursor was mixed with polyethylene glycol in the weight ratio 1:5 and ground together in a mortar. The resultant solids were placed in a crucible and heated in the air. It was observed that polyethylene glycol first melts, forms froths and ignites to form α -Fe₂O₃. On cooling to room temperature no traces of carbon impurities were observed in the final residue of α -Fe₂O₃. This reaction which occurs with the evolution of lot of gases and ignites autocatalytically is called a strong self-propagating combustion reaction, as it resembles the strong combustion reaction for the preparation of SnO₂.

Characterization

The powder X-ray diffraction patterns were obtained from GEOL JDX-8P or SEIMEN (Japan) X-ray diffractometer using CoK α radiation. The morphology of the maghemite (γ -Fe₂O₃) samples prepared by mixing equimolar solutions of ferrous ammonium sulphate and citric acid. The precipitate of ferrous citrate obtained was filtered through sintered glass crucible and was washed with cold oxygen free distilled water and finally with dry acetone and dried under vacuum. The above precursor was mixed with polyethylene glycol in the weight ratio 1:5 and ground together in a mortar. The resultant solids were placed in a crucible and heated in the air. It was observed that polyethylene glycol first melts, forms froths and ignites to form α -Fe₂O₃. On cooling to room temperature no traces of carbon impurities were observed in the final residue of α -Fe₂O₃. This reaction which occurs with the evolution of lot

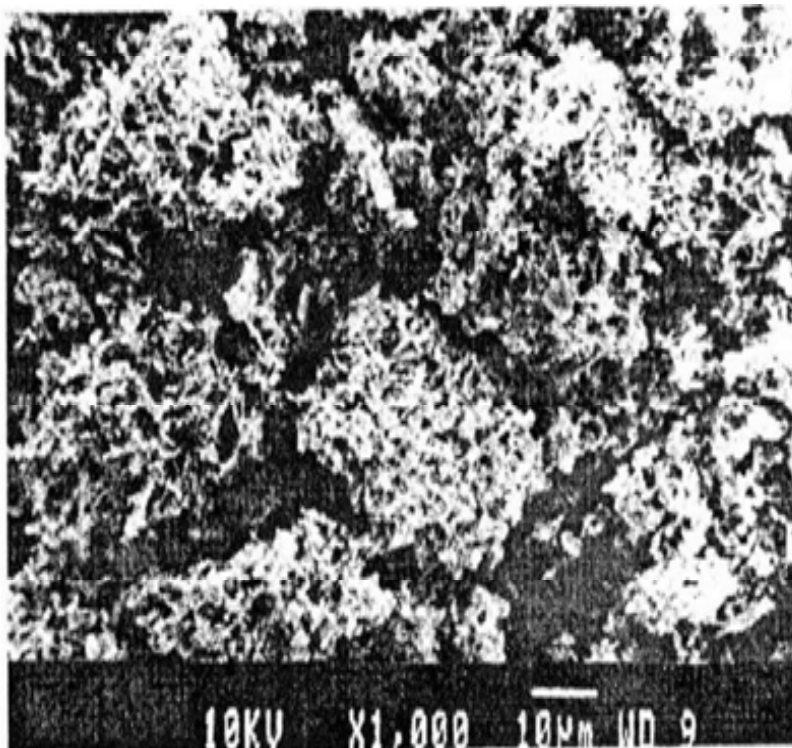


Fig. 1. SEM image of as synthesized γ -Fe₂O₃

Table. 1: Indexed of XRD data of γ - Fe_2O_3 sample

Hkl	d_{obs}	D_{lit}	I_{obs}
2 2 0	2.960	2.959	27
3 1 1	2.526	2.524	100
4 0 0	2.094	2.093	1 6
4 2 2	1.708	1.708	21
5 1 1	1.610	1.708	1 7

Table. 2: Vibrational Frequencies of γ - Fe_2O_3 sample

Sl. No.	Vibrational Frequencies
1	3100
2	1085
3	505
4	455

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