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Hydrothermal synthesis of α–Fe_2O_3 nanorods prepared by a new and a fast route

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ABSTRACT

We have described a very fast approach for the formation of α–Fe_2O_3 nanorods by a simple reaction of iron with water at very low temperature of 190°C. It is shown that the nanorods have diameters ranging from 70-120nm and their typical lengths are in micrometers. The initial formation and subsequent growth of α–Fe_2O_3 nanostructures may be explained by the iron metal corrosion mechanism. There are other different existing approaches which were applied for the synthesis of iron oxide nanostructures, but most of the methods involve environmentally malignant chemicals and organic solvents which were toxic and not easily degraded in the environment. Compared with others, the reported method is fast, economical, environmentally benign and free of pollution, which will make it suitable for large scale production.

Keywords: Iron powder; simple synthesis; nanorods; FESEM.

INTRODUCTION

Iron oxide especially hematite (α-Fe_2O_3), is one of the most important magnetic materials and exhibits numerous potential applications. It is one of the most stable oxides under ambient conditions and is widely used in catalysts, solar energy conversion, environmental protection, sensors and biomedical applications[1]. Its non toxicity, low cost and corrosion resistant property are definitely very attractive features for potential applications. As an example of potential applications, the as obtained iron oxide nanomaterials were used as adsorbent in waste water treatment and showed an excellent ability to remove various water pollutants[2]. It is also used in ultrahigh density magnetic storage devices, in drug delivery and tissue repairing engineering[3]. Because of its excellent properties, much attention has been directed to the controlled synthesis of nano and micro structures with various morphologies. Shuttle like Fe_2O_3 have been synthesized via Fe^{3+} as a source[4]. Plate shaped Fe_2O_3 have been prepared at room temperature through reduction oxidation[5]. Their rod and belt forms have been achieved by direct deposition[6,7]. By controlled exposure, Fe particles have been converted to Fe-Fe_2O_3 core shell structures[8]. But all the above methods involve environmentally malignant chemicals and organic solvents which were toxic and not easily degraded in the environment. To the best of my knowledge, approaches that are simple, direct, controllable and suitable for large quantity production remains as a challenge for the synthesis of well crystallized nanostrucrtures of α-Fe_2O_3.

Hydrothermal techniques have found their place in several branches of modern science and technology. Owing to their special physical properties, particularly high salvation power, high compressibility and mass transport of these solvents, one can also expect the occurrence of many novel reactions[9]. In this letter, we describe a facile method for the preparation of α-Fe_2O_3 nanorods by a simple reaction of iron powder with water using much lower temperature of 190°C. The aim of the study is to provide the feasibility of the simple route for the preparation of nanorods and nanostructures of iron oxide.

EXPERIMENTAL

In a typical synthetic producer, 5 mg of iron powder was added to 20 ml of distilled water. The reaction mixture was well sonicated for 25 minutes, transferred into a stainless steel autoclave and then sealed. Samples prepared have been kept at room temperature (Rt) and 190°C for 24h respectively. The mixture of the samples was centrifuged to reclaim the precipitated sample. The final product thus obtained was vacuum dried for 6h at room temperature.

The morphology, crystalline size and structures of α-Fe_2O_3 samples were studied by high resolution FESEM (FEI NOVA NANOSEM) coupled with energy dispersive spectroscopy (EDS). X-ray diffraction patterns of the samples were recorded with Siemens D 5005 diffractometer using Cu Ka (λ =0.15141 nm) radiation.

RESULTS AND DISCUSSIONS

Fig. 1 shows Field Emission Scanning Electron Microscopy (FESEM) images of the as

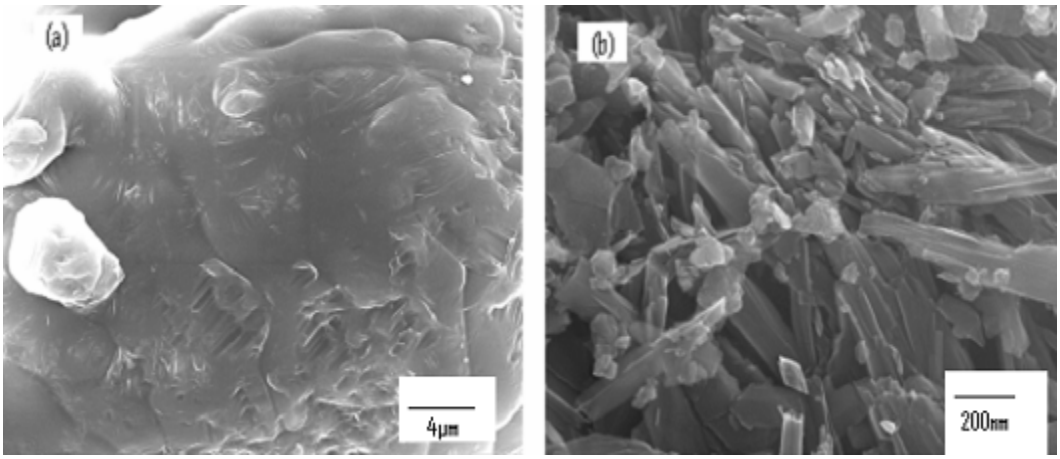

Fig. 1: FESEM images of various as prepared samples. Experimental Parameters: a) Rt, 24 h; d) 190oC, 24h

prepared samples obtained by reacting micrometer sized large iron particles with water at room temperature and at 190°C for 24h respectively. Nanorods were not observed for a sample reacted for 24h at room temperature (Fig. 1 a) whilst rods like products radiating from and normal to the original large iron particle surfaces were produced for sample heated at 190oC for 24h (Fig. 1 b). The nanorods have diameters ranging from 70-120nm and their typical lengths are in micrometers. Earlier belt/plate like structures has been observed at 450°C by a solovothermal method[10]. An intermixing of ethylenediamine either with ethanol or water in different volume ratios was used to generate such structural forms of α-Fe_2O_3. The study showed that

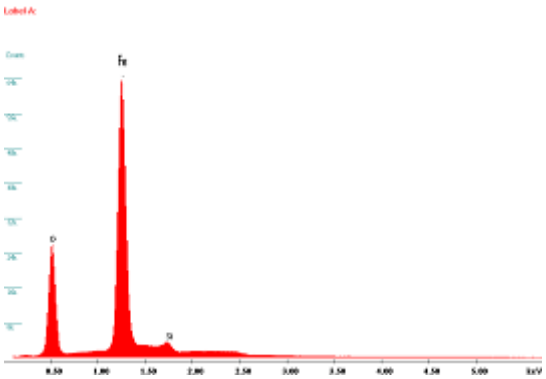

Fig. 2 : EDX spectrum revealing that the nanorods are composed of Fe and O elements.

during synthesis ethylenediamine functioned as a legend and facilitated the growth of nanostructured samples but no such solvent has been used in this study. This work has ruled out the role played by the solvents in the structural evaluation of Fe_2O_3. The EDX analysis is carried out to determine the chemical composition of iron and oxygen with an atomic ratio of approximately 1:1. The measurement on the nanorods indicates that the product is composed of Fe and O as shown in Fig. 2. The XRD spectra (Fig. 3) of all the samples prepared at 190°C reveal a well crystalline hexagonal phase of α-Fe_2O_3 with cell parameters around a = 5.035Å, c = 13.74 Å, space group R3c, (JCPDS file No. 17-0536).

The formation of nanorods by the reaction of iron with water can be explained as follows. Iron gives hydrogen on reaction with water

$$2Fe(s) + 3H_2O\ (l) \rightarrow Fe_2O_3\ (s) + 3H_2\ (g)$$

Here (s), (l) and (g) represents solid, liquid and gas respectively. The similar study has been reported earlier, where evolution of hydrogen has been documented[11].

Based on the corrosion theory, we know that at high temperature in the absence of oxygen, the corrosion of iron by water involves two key component movements: the transport of oxygen-bearing species to the metal/oxide interface and the diffusion of iron ions become saturated at some points on the surface, an iron oxide layer then nucleates and grows. The most widely cited classical model for shape control of crystals is given by Gibbs-Curie-Willff Theorem. This theory suggests that the shape of a crystal is determined by the surface energy of individual crystallographic faces. The final crystal shape is determined in such a way that the total free energy of the system is minimized. Moreover, water at elevated temperatures plays an essential role in the precursor material transformation because the vapour pressure is much higher and the state of water at elevated temperatures is different from that at room temperature. The solubility and the reactivity of the reactants also change at high pressures and high temperatures and high pressure is favorable for crystallizations.

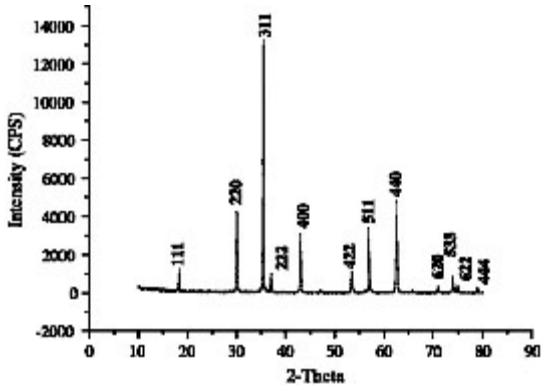

Fig. 3: The XRD pattern of samples at 190°C for 24h.

CONCLUSION

In conclusion, we have found new and simple methods for the preparation of Fe_2O_3 nanorods by a simple reaction of Fe-H_2O at 190°C. Detailed and systematic studies would be necessary to optimize the conditions for obtaining nanorods of desired dimensions. It is an economical low temperature process and does not require any template. This direct and efficient route, extendable to other metal or alloy oxide nanostructures has the potential to be further scaled up. This synthetic technique has the following advantages: Firstly, it is one step synthesis approach, making it easy to control the growth kinetics. Secondly, the synthesis needs no sophisticated equipments since it is conducted at low temperature. Thirdly, the approach is non toxic without producing hazardous waste as water is being used as solvent as well as source of oxygen.

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