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Synthesis of non-aggregated nicotinic acid coated magnetite nanorods via hydrothermal technique

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ABSTRACT

Non-aggregated magnetite nanorods with average diameters of 20–30 nm and lengths of up to 350 nm were synthesized via *in situ*, template free hydrothermal technique. These nanorods capped with different concentrations (1, 1.5, 2 and 2.5 g) of nicotinic acid (vitamin B3); possessed good magnetic properties and easy dispersion in aqueous solutions. Our new synthesis technique maintained the uniform shape of the nanorods even with increasing the coating material concentration. The effect of nicotinic acid on the shape, particle size, chemical structure and magnetic properties of the prepared nanorods was evaluated using different characterization methods. The length of nanorods increased from 270 nm to 350 nm in nicotinic acid coated nanorods. Goethite and magnetite phases with different ratios were the dominant phases in the coated samples while a pure magnetite phase was observed in the uncoated one. Nicotinic acid coated magnetic nanorods showed a significant decrease in saturation magnetization than uncoated samples (55 emu/g) reaching 4 emu/g in 2.5 g nicotinic acid coated sample.

The novel synthesis technique proved its potentiality to prepare coated metal oxides with one dimensional nanostructure which can function effectively in different biological applications.

1. Introduction

Magnetite nanoparticles (NPs) have attracted considerable interest in the recent years because of their fundamental importance and potential applications in various fields [1–3]. These magnetic particles can be synthesized so small that each particle becomes a single domain, showing unusual magnetic properties, known as superparamagnetism [4,5]. Nowadays, superparamagnetic particles are among the most studied nano-materials in the biomedical field. Applications such as targeted drug delivery, magnetic fluid hyperthermia, magnetic resonance imaging and tissue engineering are being developed [6–9]. Nevertheless, such applications require the particles to be water soluble and biocompatible [10].

Efficient coverage of the nanoparticle surface is important when considering biomedical applications [11]. The high chemical reactivity of the nanoparticle surface could pose health risk to patients, since the NPs could influence reaction pathways on the cellular level [11]. Aggregates in nano-sized magnetite suspensions can also cause blood vessel blockage after administration, which may result in localized hypoxia, necrosis and hypersensitive reaction [12].

Surface coating plays an important role in shaping the adsorption, distribution, metabolism and excretion (ADME) process of the magnetite NPs after administration [12]. An important example of surface coating materials used in industry is “vitamins”. Vitamins are essential organic nutrients and most of them are not synthesized in the body and have to be obtained through the food we eat. Generally, vitamins are classified into two types. The first type is fat-soluble vitamins; A, D, E and K and the second type is the water-soluble vitamins; B-complex group and vitamin C [13].

Using vitamins as coating material for magnetite (Fe_3O_4) can have several benefits. It is known that most of the vitamins have active functional groups and thus allow their interaction with Fe (II), Fe(III) of magnetite. Water-soluble vitamins can increase the solubility of magnetite NPs and help their uniform dispersion in suspensions [14]. Vitamins are also considered biocompatible, natural materials and thus may help in decreasing the toxicity of magnetite when used for *in vivo* applications.

In the recent years, some studies have adopted the idea of using vitamins as coating materials. For instance, Shouhu Xuan et al.

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reported the preparation of water-soluble Fe_3O_4 nanocrystals through hydrothermal approach [14]. These nanocrystals were capped with $C_6H_6O_6$ (the oxidation state of ascorbic acid) and could be readily dispersed in hydrated aqueous systems [14]. Chandrasekharan et al. succeeded in synthesizing vitamin E TPGS (D-a-Tocopheryl-co-poly(ethylene glycol) 1000 succinate) micelles for superparamagnetic iron oxides formulation to be used in nanothermotherapy and magnetic resonance imaging (MRI) [12]. Their product showed better *in vitro* cellular uptake as well as *in vivo* therapeutic and imaging effects in comparison with commercial products [12]. Janaky et al., incorporated vitamin B12 coated magnetite nanoparticles into a conducting polypyrrole and succeeded to develop an advanced electrode activity [15].

Niacin (nicotinic acid), also known as vitamin B3, is a water-soluble vitamin that occurs in a chemical form of pyridine-3-carboxylic acid [16]. Niacin has shown encouraging results in humans during clinical trials for effectively reducing and controlling Low Density Lipoproteins (LDL) levels, elevation of High Density Lipoprotein (HDL) and reduction in tri-glyceride and cholesterol levels [16]. Currently, Niacin therapy; as a replacement or in combination with existing cholesterol reducing prescription drugs, is under evaluation by various drug regulatory authorities around the world [16]. Trials for grafting niacin on the surface of magnetite NPs as a form of pharmaceutical drug carriers, have been done by Belikov et al. [17]. They studied the types of interaction between niacin and the Fe(II) and Fe(III) of magnetite nanoparticles and found that the percentage of initial drug bound to the surface was 39% [17]. Thus, it can be used successfully as pharmaceutically guided drug.

Through literature survey, it can be noticed that a large number of studies are focusing on obtaining coated magnetite NPs with adequate solubility and biocompatibility, yet, very few studies targeted the development of uniform shaped NPs after coating [14,18,19]. Generally, coating of previously prepared iron oxide nanoparticles has some drawbacks that include alterations in the shape of NPs after coating and the formation of non-uniform, aggregated particles [20].

In this paper we report a new *in situ* coating technique for nicotinic acid (Vitamin B3) on the surface of magnetite nanorods while studying the alterations in shape, size and chemical structure of particles. Such surface modification may widen the potentiality of the magnetic nanorods to be used in different applications.

2. Materials and methods

2.1. Materials

Ferric chloride ($FeCl_3 \cdot 6H_2O$) was obtained from ALPHA chemika (India), Ferrous sulphate ($FeSO_4 \cdot 7\,H_2O$) was purchased from Oxford Laboratory (Mumbai, India), Urea ($(NH_2)_2CO$) was obtained from FISCHER (Chennal, India), and Nicotinic acid was brought from SDFCL (S d fine- chem. limited) (Mumbai, India). All materials were of analytical grade.

2.2. Synthesis of coated magnetite nanorods

A series of experiments were carried out using different concentrations of nicotinic acid to study the effect of coating on the properties of magnetite nanorods. In the preparation of nicotinic acid coated magnetite nanorods we followed a modified procedure to that in reference [21]; $FeCl_3 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$ and $(NH_2)_2CO$ were all dissolved in 30 ml purified, deoxygenated water at a molar ratio of (Fe^{+3}: Fe^{+2}: urea, 1:1:4 (based on previous studies in our labs [22])). The solution was then magnetically stirred for 30 min during which portion wise addition of different amounts (0 g, 1 g, 1.5 g, 2 g and 2.5 g) of nicotinic acid was done till complete dissolution was achieved and a dark red solution was obtained. Then all the reactants were injected to a Teflon-lined stainless autoclave, which was maintained at 85–90 °C for 18 h, and cooled to room temperature. The precipitate was isolated in a magnetic field and washed three times with distilled water, then dried in a vacuum at 50 °C for 1 h. At last, coated Fe_3O_4 nanorods samples were obtained with a yield ranging from 0.4 to 0.5 g.

2.3. Characterization of the prepared nanorods

The structure and phase purity of the nano- powders were investigated by X-ray diffractometry (XRD). Morphological and particle size analysis of samples was performed by Transmission Electron Microscopy (TEM). The chemical characteristics of the nicotinic acid modified magnetite nanorods were studied using FTIR and magnetic properties of the powder samples were measured using a Vibration Sample Magnetometer (VSM).

3. Results and discussion

3.1. TEM

The TEM microstructures of uncoated magnetite nanorods as well as nanorods coated with various concentrations of nicotinic acid are shown in Fig. 1, 2 and 3. Fig. 1(a) illustrates clearly the

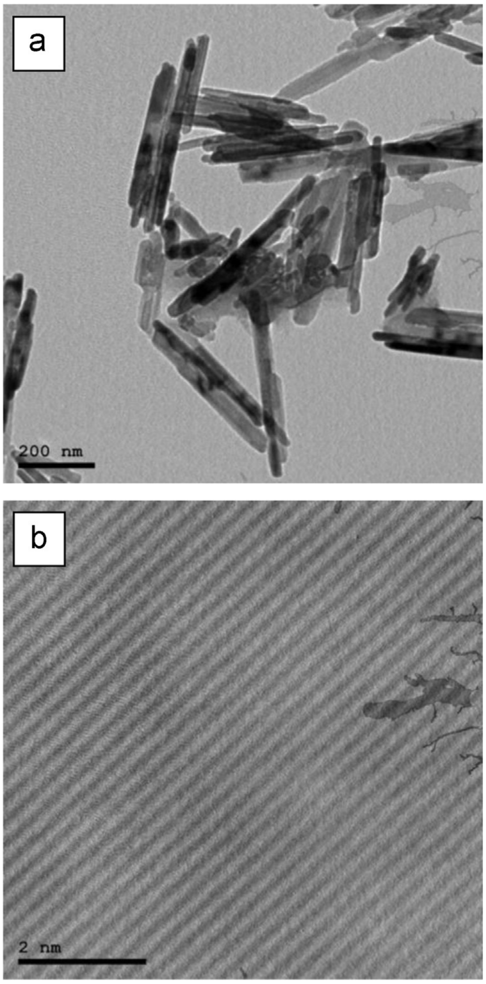

Fig. 1. TEM images of uncoated magnetite nanorods.