



Magnetite Nanoparticle Green Synthesis from Canola Oil

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ABSTRACT

Nanomagnetite can be synthesized in lab by decomposition method using FeOOH and oleic acid in 1-ODE but the cost of production is very high. Here we are trying to synthesize cost effective nanomagnetites by using common materials present in our daily life which are freely available in market. Magnetite nanocrystals are highly useful in arsenic remediation as arsenic contamination in groundwater is a severe global problem. In India, millions suffer from acute and chronic arsenic poisoning. It is possible to create functional and high-quality nanocrystals using this greener and cost effective method. Our research revealed that the costs of the starting materials can be reduced by using this greener synthetic route

Key words: Nanomagnetite, contamination, Arsenic remediation, Drinking water, green synthetic, Magnetite nanocrystals.

INTRODUCTION

Aims and Background

Physical substances with at least one characteristic dimension between 1-150 nm can be defined as nanomaterials. Nanomaterials properties can differ from those of the same materials with micron- or mm-scale dimensions. Nanomaterials are the building blocks of practical nanotechnology and can be physically and chemically manipulated for specific application. Nanotechnology has been proved efficient in improving air, water, and soil quality in the environment. It can progress detection and sensing of pollutants and help in the development of other

new technologies for control. The synthesis of fine sizes of magnetite usually require highly pure chemicals and laboratory tools, such as temperature-controlled heaters, stirrers, and inert atmosphere. Non-aqueous mediums are also preferred for high monodispersity and uniformity^[1] In a typical synthesis, an iron salt or oxide is mixed with a surfactant and boiled in a high boiling point solvent under air-free reflux conditions. The most notable examples of this type of synthesis are refluxing iron acetylacetonate, oleic acid, oleylamine and 1,2-hexadecanediol in diphenyl ether^{2,3}, heating iron oxohydrate (FeOOH) with oleic acid in 1-octadecene (ODE)⁴, precipitating FeCl₃ with oleic acid and further heating to a boiling in 1

–octadecene (ODE)⁵, and mixing FeCl₃ and sodium oleate before refluxing the precipitate in a mixture of oleic acid and ODE⁶.

All of these methods, however, require high purity grade chemicals, thereby limiting their use in very low cost operations. So our aim is to synthesize magnetite nanoparticles for environment friendly uses through chemicals readily available in open market. Earlier soya bean, corn and olive oil were used to synthesize nanomaterials by kitchen synthesis¹².

EXPERIMENTAL

Materials

Edible oil: Canadian pride Canola oil, Pidilite klog remover, Top's vinegar was purchased from market. Water was used from laboratory tap water without further purification. Rust was obtained from different sources of rusted iron, such as the iron sheet that was found inside university campus, iron angles and rods found in the playground of university (Fig.1-5)

Soap making process

Pidilite klog remover (15 g) is dissolved in 30 ml of tap water and, while warm, poured into a glass bowl containing 100 g of canola oil. The mixture is stirred with a wooden spoon and kept in open area to dry and cure, until a thick, hard soap is obtained. (Fig.6)

Production of fatty acid mixture

The cured soap (50 g) is finely grated and mixed with 500 ml of tops vinegar. The resulting solution is boiled for 15–30 min, with stirring, until all soap gets dissolved. Upon cooling, two layers

are formed. The top organic layer is removed and placed in a pan. This cloudy yellow mixture is heated to a boil until the remaining water and acetic acid evaporates.

Magnetite nanocrystal synthesis

Rust is obtained from rusted steel sources. Rust powder (2 g) is mixed with 30 g fatty acid mixture (FAM) and placed into the pan. Covered by a lid, the solution is boiled for 2 hr, producing a thick smoke. This thick smoke diminishes in time, and the end product is a black gelatinous magnetic material (Fig.7). The soap (5 g) is dissolved into 50 ml of hot water and boiled until dissolved. 5g slurry from the pan is mixed with the soapy water and boiled for 30 min. The unreacted solid is removed by filtering. Water-soluble magnetite nanocrystals are then magnetically collected. The deposits on the magnet are then washed with water and redispersed in water or ethanol.

RESULTS AND DISCUSSION

Limited greener and cost-effective synthetic methods are reported in the field of nanotechnology. The most common olive oil⁹ and Therminol 66¹⁰ have been shown to be directly usable as solvents.



In this greener route, the main starting materials of the synthesis, iron precursor and the oleic acid, can be replaced with readily available chemicals. Edible oil such as canola oil can replace oleic acid with a mixture of fatty acids that are formed via saponification that is followed by acidification. Rust is the common replacement of FeOOH. The new chemical reaction¹² then becomes:



Fig. 1: Canola oil



Fig. 2: Tops vinegar

Rust (hydrated iron oxides) + Fatty Acid Mixture (FAM) Magnetite (Fe_3O_4) \rightleftharpoons Nanocrystals

Soap (mixture of fatty acid salts) is produced by saponification of canola oil, The FAM, which is a mixture of long-chain organic acids, forms once this soap is acidified with vinegar. There are different fatty acids that constitute FAMs, but four of these are almost always dominant¹³: oleic acid [(9Z)-octadec-9-enoic acid], linoleic acid [(9Z, 12Z)-octadeca-9, 12-dienoic acid], stearic acid (octadecanoic acid), and palmitic acid (hexadecanoic acid)

IR Spectrum

The peak at 570.95 cm^{-1} is assigned to the Fe-O bond vibration of Fe_3O_4 nanoparticles



Fig. 3: Pedillite Clog remover



Fig. 4: View of Rusted iron sheet at 3 feet distance

confirming the formation of nanomagnetites.

TEM Analysis

In order to determine the characteristics of magnetite nanoparticles, high resolution transmission electron microscopy (HRTEM) images were obtained at the main zone axes. Fig.9- HRTEM images

The Fe_3O_4 nanoparticles dispersed in ethanol were dropped on carbon film coated copper grids. The self-assembly formed as the hexane evaporated and its morphology and structure were investigated by means of transmission electron microscopy (TEM). The Fe_3O_4 nanoparticle solution was poured in a capsule and put in a Quantum Design SQUID (MPMS-5s) for magnetic characterization. STEM analysis of nanomagnetites prepared from canola oil show that general size is from 20-100 nm.

Applications of Nanomagnetites

1. Arsenic (As) in drinking water is an epidemic affecting millions of people living in Southeast Asia including India and Bangladesh^[11]. The toxicity of arsenic to human health ranges from skin lesions to cancer of the brain, liver, kidney, and stomach. Nanomagnetites were used as a most efficient agent for removal of heavy metals such as As and Cr for cleaning polluted water. Arsenic is naturally present in groundwater in the forms of arsenite (AsO_3^{3-}) and arsenate (AsO_4^{3-}). These anions resemble phosphite (HPO_3^{2-}) and phosphate (PO_4^{3-}) ions, and it is this similarity that is the dominant source of their toxicity: arsenite and arsenate block ATP—ADP conversions by



Fig. 5: Close view of Rusted iron sheet

permanently replacing phosphate groups¹⁴. Although arsenic-contaminated ground water belongs to local scale pollution, this problem prevails all over the world. Traditional treatment technologies are not effective and magnetite nanoparticle seems to be a good alternative due to its

highly selective adsorption toward arsenic and feasibility on regeneration. This nanomaterial exhibited high As(V) adsorption capacity, up to 206.9 mg g⁻¹, which is the highest reported¹².

2. Nanomagnetites can be used as a green catalyst .



Fig. 6: Canola oil soap

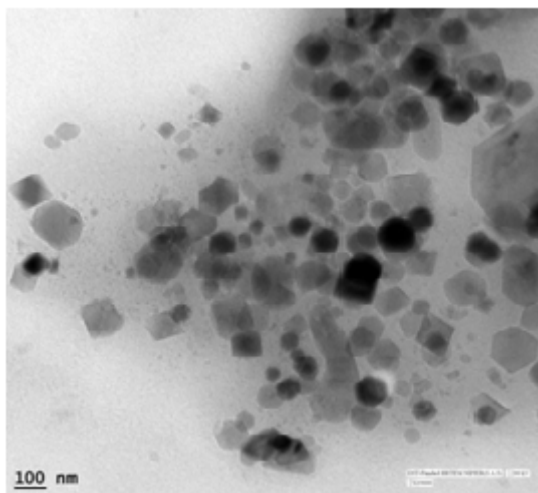
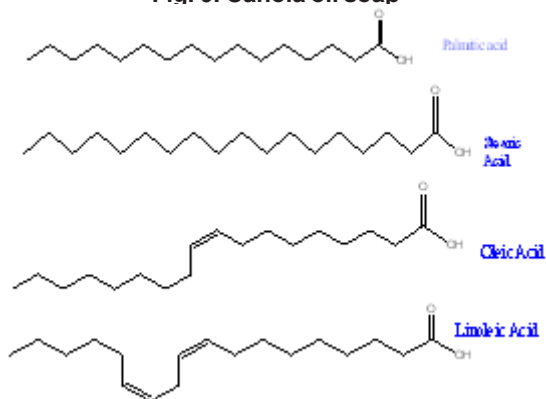
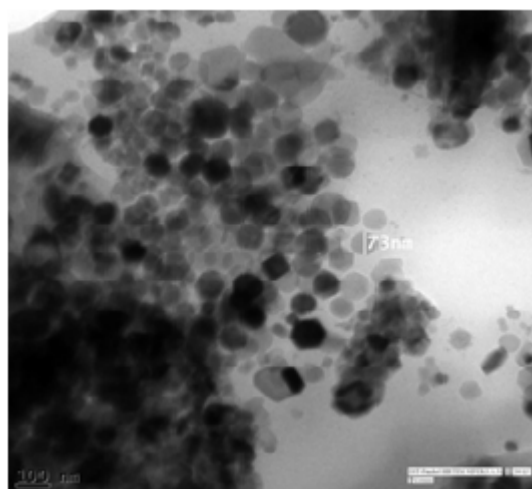
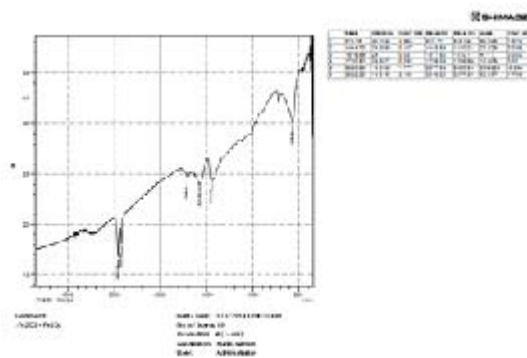


Fig. 7: Nanomagnetite slurry



Instrument name: FEI Tecnai G2 F20, Analysed at 200KV. Courtesy :HRTEM Facility,NIPER, Mohali

3. Nanomagnetite can be used as Encapsulation within polycaprolactone microspheres for bone replacement.
4. Nanomagnetite can be used for biomedical applications.
5. The two-dimensional arrays of nanomagnetite particles can be used for fundamental rock magnetic studies.
6. Nano-sized magnetite can be used for the wastewater treatment for ciprofloxin like biologically non-degradable antibiotics.
7. Nanomagnetite can be used in target drug delivery to enhance the curative effect and minimize the adverse effects of an anticancer drug.
8. Nanoparticles (SPIONs) with poly(d,l-lactide-co-glycolide) PLGA can be used to serve the dual work of MRI contrast improvement as well as direction of polymer particles to particular locations.
9. Magnetite and maghemite nanoparticles and their suspensions are widely used in different industrial applications like magnetic sealing, oscillation damping, position sensing or magnetic storage

media¹⁵. The ferrofluids contain iron oxide magnetite nanoparticles. Recently, ferrofluids have been used in conjunction with microcontact printing and capillary filling to fabricate patterned structures of magnetic materials on the micron scale. The ability to generate patterns of ultrafine magnetic particles has versatile technological applications, because of the information density on tapes, for instance, is inversely proportional to the size of the particles. Research has been done on finding the use of ferrofluids as magnetic inks for ink jet printing. Magnetic inks are presently used in printing USA paper currency, as can be demonstrated by the attraction of a genuine dollar bill to a strong magnet¹⁶.

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