International Journal of Current Engineering and Technology
E-ISSN 2277 – 4106, P-ISSN 2347 – 5161

Available at http://inpressco.com/category/ijcet

Review Article

A critical review on preparation of Fe_3O_4 Magnetic Nanoparticles and their potential applications

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Received 10 Oct 2018, Accepted 12 Dec 2018, Available online 14 Dec, **Vol.8, No.6 (Nov/Dec 2018)**

Abstract

Nanotechnology is one of the most powerful techniques which has been spread rapidly in entire world within less time because of wide range of its applications. This review reveals the key methods for the preparation of magnetic nanoparticles systematically. This paper helps us to select the most efficient and economical technique out of many described. In general the properties of magnetic particle differ due to change in its size, shape and crystalline structure, however many factors have been described in this review to obtain the magnetic nanoparticles less than 100nm with wide applications.

Keywords: *Nanotechnology, Magnetic nanoparticles, crystalline structure*

Introduction

Nano Technology is the branch of technology that deals with dimensions and tolerances of less than 100nm. (any one dimension less than 100nm). Nano science and nanotechnology are the study & application of extremely small things and can be used across all the other science fields such as chemistry, biology, physics, material science and engineering. Magnetism play's an important role in electrical, electronic & mechanical engineering because without magnetism components such as loud speakers, motors, generators, transformers, electricity meters, chokes, inductors, coils would not work. Every coil of wire uses the effect of electromagnetism when an electrical current flow through it. Magnets can be found in a natural state in the form of a magnetic core, with the two main types being Magnetite also known as Iron Oxide (Fe_3O_4) & Lode Stone (Leading Stone).

There are basically two forms of magnetism

1. Permanent magnets
2. Temporary magnets

There are many different types of materials available to make magnets such as iron, nickel, nickel alloys, chromium and cobalt. In their natural state some of these elements such as nickel & cobalt show very poor magnetic quantities however, when mixed or alloyed with other materials such as iron or aluminum peroxide they become very strong magnets known as alcomax, alni, alnico and ,hycomax.

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DOI: https://doi.org/10.14741/ijcet/v.8.6.14

Magnetic materials are classified as

a) Diamagnetic
b) Paramagnetic
c) Ferromagnetic

Diamagnetic materials have a weak negative susceptibility to magnetic fields. Diamagnetic materials are slightly repelled by magnetic fields and the material does not retain the magnetic properties when the external field is removed. Cu, silver, gold are example. Ferromagnetic materials have a large possible susceptibility to an external magnetic field. They exhibit a strong attraction to magnetic fields and are able to retain their magnetic properties after the external fields have been removed. Irons, Nickel & Cobalt are ferromagnetic materials

Classification of Nanoparticles: NP'S are broadly divided into various categories depending on their morphology, size and chemical properties.

1. Carbon – based NP's
2. Ceramic NP's
3. Semi-conductor NP's
4. Polymeric NP's
5. Lipid – based NP's

Methods for the preparation of magnetic Nanoparticles

A series of general methods for Nanoparticle synthesis has been developed, most of them can also be used for the preparation of magnetic particles. An essential feature of their synthesis is the preparation of particles with specified size and shape. It is important that the distance between the particles in the matrix should be controllable finally the synthetic procedure should be relatively simple, inexpensive & reproducible.

1. **Physical methods for the preparation of magnetic Nano particles:**

    a) Condensation methods
    b) Methods of Nano dispersion of a compact material

2. **Chemical synthesis of magnetic Nano particles:**

    a) Thermolysis of metal – containing compounds
    b) Decomposition of metal – containing compounds on ultrasonic treatment
    c) The reduction of metal containing compounds
    d) Synthesis in reverse micelles
    e) Sol gel method
    f) Synthesis of magnetic Nano particles at a gas–liquid interface

3. **Specific methods for the preparation of particular types of magnetic Nano particles**

    a) Hetero metallic nanoparticles
    b) Ferrites
    c) Nanoparticles of rare earth elements
    d) Magnetic nanoparticles of anisotropic shapes

4. **Methods for the synthesis of stoichiometrically inhomogeneous magnetic particles**

    a) Oxidation of nanoparticles
    b) Chemisorption of small molecular on a nanoparticle surface
    c) Targeted modification of the Surface of magnetic Nano particles

The most widely encountered magnetic Nanoparticles are: 1)Fe 2) BCC-Fe (α -Fe) 3) FCC-Fe (¥ - Fe) 4) Amorphous Fe (metallic glass) 5)Fe_2O_3 6)Fe_3O_4 (magnetic) 7) FeO (wustite) 8)α – FeOOH (goethite) 9) Ferro fluids 10)Fe-Co alloys 11) Fe-Ni 12) Fe-Pt. 13) Co 14) COO (Cubic Cobalt Oxide) 15)CO_3O_4.

Challenges facing or controllable factors

a) Oxidation of magnetic Nanoparticles
b) Agglomeration of magnetic nanoparticles

To improve the protection against the oxidation that usually appears in core shell Nano particles, spherical iron nanoparticles coated with a carbon shell were obtained by modified is discharge reactor, which permits controlling the diameter of the iron core and the carbon shell of the particle. Oxidized Nanoparticles involve a loss of the magnetic characteristics and also changes in the chemical properties. These nanoparticles show super paramagnetic behavior and high magnetic saturation owing to the high purity.

Agglomeration means a mass or collection of Nanoparticles or an assemblage of nanoparticles. There are some methods for the prevention of nanoparticles agglomeration conformably depositing a barrier coating on at least one surface of a substrate provided in liquid form Embedding a plurality of nanoparticles in said barrier coating to a selected depth creating an embedded portion of each of said plurality of nanoparticles

2. Applications of magnetic nanoparticles

- Waste water treatment and reuse is a practice related not only to o number of benefits in regards to water balances and management but also to a number of question marks.
- The spread of a wide range of contaminants in surface water and ground water has become a critical issue worldwide. To combat the problem waste water treatment have been made including photo catalytic oxidation adsorption/ separation processing and bioremediation.
- Magnetism is a unique physical property that independently helps in water purification by influencing the physical properties of contaminants in water. Iron oxide NMS are promising for industrial scale waste water treatment, due to their low cost, strong adsorption capacity, easy separation and enhanced stability & excellent superiority. Current applications of iron oxide NMS in contaminated water treatment can be divided into two groups.

a) Technologies which use Fe_3O_4 nanomaterial as a kind of Nano sorbent or immobilization carrier for removal efficiency enhancement
b) Technologies which use Fe_3O_4 nanomaterial as photo catalyst to break down or to convert contaminants into a less toxic form. Adsorption process is the powerful technique for removal of metal ions from industrial effluent streams. It reduces the operational cost and size of the equipment along with the increase recovery of metal ions.Fe3o4 magnetic nanoparticles modified with 3-amino propyltriethoxysilane (APS), copolymers of acrylic acid (AA)and crotonic acid(CA) are good absorbents used for removing heavy metal ions such as cd-II,zn-II,pb-II and cu-II from aqueous solutions.

- In biomedical field, magnetic nanoparticles and magnetic composites are utilized as the drug carriers to contrast agents for magnetic resonance imaging (MRI) and in magnetic hyperthermia, the utilization of magnetic nanoparticles in separation/pre-concentration of various molecules and cell and their use in diagnosis and therapy are highlighted in his paper the four-application described for MNPs aided in diagnosis and treatment of diseases in the following years.

1. Magnetic separation of biological entities contributed to the development of diagnostics.
2. Magnetic nanocarriers contributed to drug delivery
3. Radio frequency – controlled magnetic nanoparticles provided a new approach for cancer treatment
4. Magnetic resource imaging application (MRI)

- Magnetic nanoparticle devices or magnetic composites are attractive for drug delivery due to their ability to respond to exogenous stimuli via a magnetic field: this allows controlling drug release in spatial temporal and dosage-controlled fashion.
- Super paramagnetic iron oxide nanoparticles (SPION) with appropriate surface chemistry have been widely used experimentally for numerous in vivo applications such as MRI contrast enhancement, tissue repair, immunoassay, detoxification of biological fluids, hyperthermia, and drug delivery and in cell separation.
- Magnetic iron oxide nanoparticles are the basic components of remote controlled nanoparticle systems for nano medicine that seem to fulfil most of biocompatibility and specific formulation requirements related to magnetic field guided drug delivery and hyperthermia systems as well as contrast agents there are envisaged optimized synthesis procedures to ensure large scale and reproducible production of IONP systems with optimal surface properties, shape, size, bio compatibility and high magnetic moment. The main challenge is dual involving the design of magnetic core and 115 surface engineering to provide appropriate core – shell IONP's for theragnostic purpose.
- Based on SPIONS unique mesoscopic physical, chemical, thermal and mechanical properties, super para magnetic nanoparticles offer a high potential for several biomedical applications.

a) Cellular therapy such as cell labelling, targeting as a tool for cell – biology research to separate and purify cell populations
b) Tissue repair
c) Drug delivery
d) Magnetic resonance imaging (MRI)
e) Hyperthermia
f) Magnetoceptionetc

3. Literature Survey

Deepa Thapa (Deepa Thapa ,*et al*, 2011) conducted an experiment simple preparation route to synthesis magnetite (Fe_3O_4) nanoparticles with controlled size. The study of these nanoparticles indicates an enhancement in saturation magnetization with reduction in size down to 10nm.In this experiment magnetic ferrosoferric hydroxide was precipitated by mixing solutions of ferrous chloride ($Fecl_24H_2O$)and molar ammonia solution (NH_4OH) at 80-90^{0}C the precipitate obtained was filtered, dried over night at room temp here concentration of precursor solution and precipitation rate were two factors that controlled the particle size ranging between 5-100nm.The advantages are the particle size decreases as the concentration of the precursor solution decreases and the disadvantages include the reduction in particle size down to 10nm creating negative pressure on the lattice leading to a lattice cell volume expansion, and also leads to decrease in magnetic transition temperature. Yan Wei, conducted an experiment on synthesis Fe_3O_4 NP's Co-precipitation method using sodium citrate and oleic acid as modifiers the magnetic behavior's reveal that in this process there is a decrease in saturation magnetization of Fe_3O_4 NP's due to the surface effect, which shows excellent dispersion capability by which dipolar attraction of the Nano particles reduces. The advantages are the use of (dispersion) sodium citrate and oleic acid in deionized water has great effect on the crystallization of Fe3O4 MNP's, which makes MNP's Fe_3O_4 as a promising biomedical material and these nanoparticles prepared are non-toxic and biocompatible. The disadvantages include the saturation magnetization decreases evidently when the Fe_3O_4 MNP's were modified with sodium citrate & oleic acid.

Rodrigo Fernandez (Rodrigo Fernandez , *et al*, 2012)– Pacheco prepared highly magnetic silica–coated iron nanoparticles by arc – discharge method. The preparation of encapsulated magnetic nanoparticles consists of a metallic iron core and an amorphous silica shell by using arc discharge method. The advantages include the Silica Coating of the particle is a very important issue. It helps to make the particles biocompatible, preventing their aggregation and the degradation of the metallic core, and reducing the extent of clearance by the reticuloendothelialsystem and the inorganic amorphous silica is biocompatible, non-toxic and possesses hydroxyl surface groups. Amorphous silica is a heat resisting material, with a low specific gravity, high surface area, and good mechanical strength theses NP's has high magnetization saturation and strong response to magnetic fields of their ferromagnetic core with adsorption properties and chemical versatility.

N.V.Tarasenko(N.V.Tarasenko, *et al*, 2009) developed a technique for preparing metallic & metal – containing NPs with modification of metal micro powders in liquids. In this technique, the optimal conditions for the production of titanium carbide and copper NPs embedded in carbon layers were found. The advantages are the maximum diameter of nanoparticle did not exceed 50nm, while the minimum was around 2nm and the disadvantages include nanoparticles prepared by are discharge method were in agglomerated form i.e. surrounded by grey regions, which is probably graphite layers, due to agglomeration, it was difficult to measure their size correctly.

Zhang Junhao (Zhang Junhao,*et al*,2009) developed a solvothermal reduction method using $K_3(Fe(CN)_6)$ as a raw material synthesized a near mono disperse Fe_3O_4 sub-microspheres with an average diameter of 170nm ethylene glycol plays a key vole both as reducing agent and solvent. The applications of Boron nitride & carbon coated Fe_2O_3 nanoparticles potential applications like high density magnetic recording media magnetic fluids, electromagnetic wave absorbing materials, magnetic carriers in clinical cures and in other novel magnetic devices. These nanoparticles have high saturation magnetization and the disadvantage is that discharge method is not suitable to coat large quantity of nanoparticles for industrial production because of low production yields. Also it is difficult to control the particles size and the thickness of the nano coating in this method. On the other hand oxide coatings made from slight oxidization of metal nanoparticles have a thin oxide layer fabricated in acetone prevents excessive oxidation of Co-nanoparticles these oxide layers are controllable because they are prepared by slow oxidization.

HisatoTokoro,(Hisato Tokoro *et al*, 2014) developed a technique to synthesis iron nanoparticles with boron nitride (BN) and carbon Co Nano coatings, mixture of Fe_2O_3 and boron or carbon powders were employed as starting materials and were annealed at temperature above 123km nitrogen atmosphere.

Jimmy Alexander,(Jimmy Alexander , *et al*, 2017) synthesized hematite Fe_2O_3 nano powder by controlled precipitation method by three stages precursors, precipitation washing and calcinations. Here the precipitation was controlled with ferric chloride as precursor, sodium hydroxide as precipitant. The advantage is that the hematite exhibits high resistance to corrosion therefore used as photo anode for photo assisted electrolysis of water this is due to band gap between 2-2.2eV and disadvantage is that the method require special equipment, high temperatures and tedious removal of impurities, which are all time – consuming and come at high monetary cost. Da Shi, in his experiment of preparing and characterizing core – shell structure Fe_3O_4 @ C magnetic nano particles were synthesized with super paramagnetic Fe_3O_4 Nano sphere as a magnetic core, glucose, phenolic, a soluble starch resin as carbon source via solvothermal method. The advantages include that (100-200) nm diameter & 8-20nm carbon shells are formed because of the stability of carbon shell, the Fe3O4 magnetic core is protected and its strengths about easily recycling and controlled thickness makes it has better application.

Sergey A. Novopasin (Sergey A. Novopasin , *et al* ,2014) A composite Fe-C anode sputtering in a low pressure arc discharge has been used to produce Fe contacting nanoparticles on a carbon matrix & the produced material was calcined stepwise in air from 300 to 1100K this procedure resulted in the formation of Fe oxides and oxidation of carbon and converting it into gas phase.

S.A Novopashis (Sergey A. Novopasin ,*et al* ,2014) used plasma–arc method for the synthesis of MNPs encapsulated in a carbon jacket in this technology the iron oxide nanoparticles composite Fe–C anode sputtering in a low-pressure arc discharge method. Christion Klinke, developed a simple method for fabrication of metal nanoparticles i.e. heating metal organic crystals in vaccum results in the formation of well-defined metal particles embedded in a carbon matrix iron phthalocyamine (FePc) was suspended in ethanol sonicated& dried on silicon oxide surfaces.

Teguhendahsaraswati (Teguh Endah, *et al*, 2004)used a method catalytic chemical vapour deposition (CCVD) using catalyst i,e iron, nickel, generally except the catalyst, carbon source gases as the precursor arc still required the bi-functional catalyst was prepared by submerged arc discharge that simply performed using carbon & carbon / iron oxide electrodes in ethanol 50%. The prepared material was then used as a catalyst in thermal chemical vapour deposition, this catalyst plays dual role as catalyst and secondary carbon source for growing carbon nano tubes. WalidBaaziz : iron oxide nanoparticles with average sizes in the range 4-28nm have been obtained by varying different synthesis parameters of the thermal decomposition of an iron precursor in the thermal decomposition of an iron precursor in the presence of surfactants in high boiling solvents. The synthesis parameters affect the NP's nucleation and growth by modifying the stability of iron stearate on which depend monomer formation and concentration with the lamer model.

M.Kashif (M.Kashif ,*et al*, 2013)Synthesized Zno Nano rods using Sol-gel method on thermally oxidized P-type silicon substrate the electrical characterization was performed using interdigitated silver electrodes to investigate the stability in the current flow of the fabricated device under different (UV) exposure times.

R.Haarindra Prasad (R.Haarindra Prasad *et al*, 2015) synthesized Zno this films of different thickness which were deposited on silicon and glass substrates an increase in the film thickness greatly influences the crystallinity, surface morphology and opto-electronic properties of the thin film. AFM data shows that the RMS surface roughness of this film decreases from 23 to 3.58nm with the increasing film thickness.

Mahdi Hajivaliei (Mahdi Hajivaliei, *et al*,2014) synthesized are discharge method on Ag-TiO_2 nanoparticles high current electrical are discharge of Ag electrodes have been prepared XRD results confirm the formation of a mixture of nano crystalline TiO_2 in rutile phase with silver metals.

Luke Burke(Luke Burke, *et al*,2016) developed a novel high through put method for in–situ synthesis of magnetic iron NP's in electro spun NFs using both conventional needle and free surface electro spinning techniques this work represents a significant step forward for production rates of magnetic NF scaffolds in terms of both particle and fiber.

Mohsen Fatemi (Mohsen fatemi, *et al*,2018) synthesized MIONP's using a newly extracted bacterium supernatant analysis shows the average particle size of very stable spherical MIONP's is about

29.3nm. The biosynthesis of MIONP's using HMH1 bacterial supernatant provides a simple fast cost effective and eco- friendly method for synthesis one of the useful nano material in nano medicine which are low toxic.

ArsalanRavanbakhsh (Arsalan Ravanbakhsh, *et al*,2018) synthesized porous zinc oxide nano – flakes by anodization method on zinc substrate in a 0.025m NaOH and 0.05M NH_4Cl solution with a voltage of 10V at room temperature of 25^0c the FESEM images show the structural evolution during 90 minute of the anodization process they also demonstrate the dependency of growth of ZnO flakes on the grains of the zinc substrate.

C.Y.Wang (C,Y,Wang *et al*, 2000),synthesized novel arc discharge method by using metallic iron filaments in NaCl electrolyte. The average sizes of the spherical particles in this technique are ranging from 25-40nm when voltage applied is 100-50v.in this technique as the voltage increases, the size of the magnetic Fe_3O_4 nanoparticles decreases.

Yuan Ming-Liang (Yuanming-liang, *et al*, 2009)synthesized α-Fe nanoparticle by a simple ethanol system in the presence of surfactant and the average particle size was around 10-40nm.In this process shell silica was produced by the hypothesis and condensation of tetra ethyl orthosilicate. The SiO_2 shell effectively improves thermal stability of Fe nanoparticle from oxidation and these Fe-SiO_2 coated iron nanoparticle are used in many applications such as catalysis, magnetic recording magnetic fluid and many other biomedical applications.

VolkanEskizeybek (Volkaneskizeybek, *et al* ,2012) Synthesized polyaniline (PANI) and PANI/ZnOnano composite by the chemical oxidative polymerization of aniline by using arc-discharge method. In this technique, as experiments are carried out by using different amount of Zno nanoparticle and maintaining the other reaction conditions unchanged. The average size obtained is around 20-100nm.

Conclusions

This paper gives us the clear information for the investigation to be done and the best technique which has to be chosen in future research in a particular area based on the simplest method, effective cost and promising work. To obtain different types and sizes of magnetic nanoparticles, many unique techniques have been discussed here. Out of these method's C.Y .Wang's method of synthesizing Fe_3O_4 powder by novel arc discharge method, which can produce magnetic Fe_3O_4 nanoparticles with an average size of 15nm looks to be economical, effective and also a method by which large quantity of magnetic nano powder can be prepared in very less time and this is the main requirement in today's world. We are now trying to further investigate this method by changing the input parameters and electrode material.

Acknowledgement

The authors express their thanks to Head of the Mechanical Engineering Department, Director, principal and Correspondent of Vidya Jyothi Institute of Technology, Aziz Nagar, Hyderabad for the help and support extended towards this work.

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