BioNanoScience (2022) 12:627–638
https://doi.org/10.1007/s12668-022-00947-5

Synthesis and Stability of Magnetic Nanoparticles

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Accepted: 31 January 2022 / Published online: 4 February 2022

Abstract

Magnetic nanoparticles are a class of nanoparticle that can be manipulated using magnetic fields. Such particles commonly consist of two components, a magnetic material, often iron, nickel, and cobalt, and a chemical component that has functionality. While nanoparticles are smaller than 1 µm in diameter (typically 1–100 nm), the larger microbeads are 0.5–500 µm in diameter. Magnetic nanoparticle clusters that are composed of a number of individual magnetic nanoparticles are known as magnetic nanobeads with a diameter of 50–200 nm. Magnetic nanoparticle clusters are a basis for their further magnetic assembly into magnetic nanochains. The magnetic nanoparticles have been the focus of much research recently because they possess attractive properties which could see potential use in catalysis including nanomaterial-based catalysts, biomedicine and tissue-specific targeting, magnetically tunable colloidal photonic crystals, microfluidics, magnetic resonance imaging, magnetic particle imaging, data storage, environmental remediation, nanofluids, optical filters, defect sensor, magnetic cooling, and cation sensors.

Keywords Nanoparticles · Magnetic nanoparticles · Co-precipitation · Magnetic resonance imaging · Environmental remediation

1 Introduction

In recent years, many efforts have been made to prepare and synthesize magnetic nanoparticles for their application in various fields such as biotechnology, drug delivery, and computer. In general, the performance and application of these nanoparticles is influenced by their proper design and synthesis [1–5]. So far, various magnetic nanoparticles have been synthesized, including pure metal nanoparticles (Fe, Co, Ni), metal oxides (Fe_3O_4, γ-Fe_2O_3), ferrites (MFe_2O_4, M = Cu, Ni, Mn, Mg, etc.), and metal alloys (FePt, CoPt) [6–11]. During the synthesis of these nanoparticles, some key conditions such as intrinsic magnetic properties, size and shape of nanoparticles, surface coating and surface charge of nanoparticles [12–22], and stability in aqueous environment as well as their non-toxicity must be considered [23–28]. By choosing a suitable synthesis method, the size, shape, surface coating, and colloidal stability of magnetic nanoparticles can be optimally controlled [29–31]. In the choice of magnetic material, iron oxides usually play a key role [32–34]. On the one hand, these oxides have good magnetic properties compared to other magnetic nanoparticles, and on the other hand, they show high stability against degradation [12–14, 35, 36]. These nanoparticles also have lower toxicity [15, 16]. To date, various methods for the synthesis of magnetic NPS have been proposed and improved [17]. In the purpose of this study, magnetic nanoparticles (MNPs) have widespread attention because of their unique features [37–44]. For a few decades, growing development in chemical synthesis of nanomaterials and material surface

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modification have been seen and performed in numerous applications including biomedicine, biotechnology, catalysis, and magnetic chemistry thermoelectric materials. Various methods for fabrication of MNPs which have a controllable size, distribution, and surface modification have been reported [45–49]. In these methods, several techniques containing irradiation, microwave, ultra-sonication, vapor deposition, electrochemical, and microwave are applied to produce MNPs either in bottom-up or top-down processes. Generally, magnetic synthesis of nanoparticles is carried out by using these two processes. Nanomaterials with magnetic properties have wide applications in many fields such as biology, medicine, and engineering [50, 51]. In this paper, the recent developments in the structures, occurrences, most commonly used samples, and common areas of use of the MNPs are given.

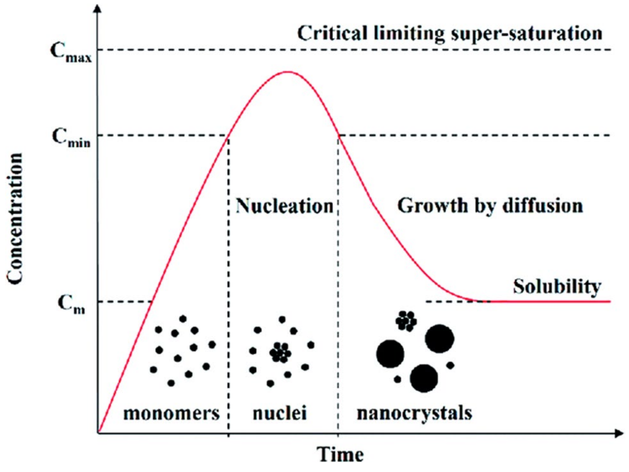


Fig. 1 LaMer diagram [1]

2 Synthesis of magnetic nanoparticles

2.1 Synthesis in liquid phase

Methods of synthesis of magnetic nanoparticles in the liquid phase include precipitation, microemulsion, synthesis using ultrasound, and so on [12, 18–20]. Homogeneous preparation and deposition of high uniformity particles (monodisperses) can be justified by LaMer principles and diagrams (Fig. 1) [1–5]. Particle growth occurs through the penetration of particles on the surface of pre-formed nuclei and the irreversible accumulation of nuclei.

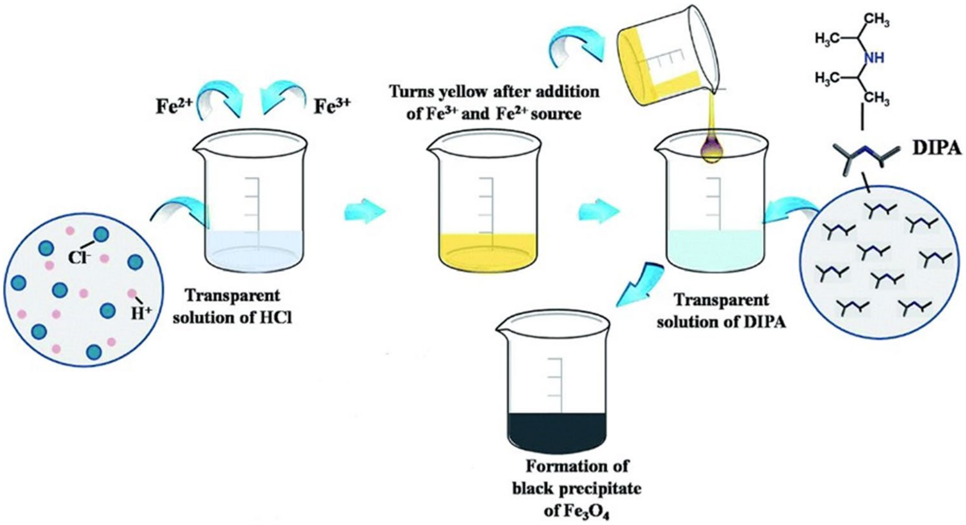
2.1.1 Co-precipitation

The co-precipitation method is the simple and the maximum effect chemic method for the synthesis of MNPS [24]. The main advantage of co-precipitation is its ability to synthesize large numbers of NPS. However, particle size repartition control is limited in this method, and kinetic factors control particle growth [25]. Figure 2 shows the schematic of synthesis of Fe3O4 magnetic nanoparticles using co-precipitation method; first, a solution of iron ions in hydrochloric acid is prepared and then this solution is poured on a solution of diisopropylamine (DIPA) which results in the formation of a precipitate of iron oxide nanoparticles [21, 22].

2.1.2 Arc Discharge

This method is commonly used to synthesize magnetic nanoparticles enclosed in a carbon layer (carbon-encapsulated) or magnetic nanoparticles made of metal carbide. In this method, the metal precursor is placed in a cavity on a graphite electrode and evaporated by arc discharge

Fig. 2 Schematic of synthesis of Fe_3O_4 magnetic nanoparticles using co-precipitation method; first, a solution of iron ions in hydrochloric acid is prepared and then this solution is poured on a solution of diisopropylamine (DIPA) which results in the formation of a precipitate of iron oxide nanoparticles [2]



[26]. This method can also be used to coat the surface of metal nanoparticles with boron nitride. Unfortunately, due to limitations such as low efficiency and difficulty in controlling the size and thickness of synthesized nanoparticles, this method cannot be used on an industrial scale [2]. In addition to these methods, laser light can also be used to synthesize nanoparticles with a size distribution of less than 10 nm (Fig. 3) [4, 27, 52–54].

3 Protection Methods

Although several methods have been proposed to improve the methods of synthesis of magnetic nanoparticles, the stability of these nanoparticles for a long time against their accumulation and deposition is an important issue. Because the stability of these nanoparticles is important in their application [28], magnetic nanoparticles are very sensitive to oxidation and accumulation as well as chemically reactive due to their large surface area. At normal temperature and pressure, the surface of the nanoparticles oxidizes rapidly, resulting in the formation of a thin layer of oxide on it, which drastically changes their properties [29]. Natural aggregation of nanoparticles is another problem that limits the dispread use of magnetic nanoparticles (Fig. 4) [4, 30]. The following methods can be used to stabilize magnetic nanoparticles [31, 55, 56]:

i) Equilibrium between repulsive forces and gravity between nanoparticles
ii) Placing inorganic coatings on the surface of magnetic nanoparticles

3.1 Organic Coating

Organic coatings are corrosion barriers between the underlying metal and the corrosive environment. They maintain durability of structures and provide resistance to weather, humidity, abrasion, chemical resistance, toughness, and aesthetic appearance. Organic coating efficiency depends on the mechanical properties of the coating system, type and concentration of suspended inhibitors [1, 2], pretreatment of the metal surface [3], adhesion of the coating to the underlying metal base [4], and other additives that inhibit substrate corrosion. Coating formulation usually contains solvent, resin (binder), pigment, filler, and additives. When applied to the underlying metal, they provide a continuous, homogeneous coating that prevents cracking and structure breakdown during stress, water permeability, and physical aging. Protective coatings should possess low permeability, good corrosion stability, and appearance over a long period of time to justify the cost [57–59]. Organic coatings are classified according to the resin's chemical composition. The resin is dissolved or suspended in the solvent. The content and density of the resin are critical for corrosion barrier properties and oxygen and water permeability. The common resins used to manufacture single-component organic coatings are vinyls, acrylics, chlorinated rubber, alkyd (oil base), modified alkyd-silicon, amino-modified alkyd, phenolic alkyd, and epoxy ester [60–64]. Two component organic coating systems are manufactured using phenolic and polyurethanes. Coating properties such as color and opacity, mechanical, and barrier properties and water transport depend on the chemical composition of the dispersed pigment, pigment volume concentration, and critical volume concentration. Besides color and opacity, the pigments protect the cured resin against UV radiation. Resins control coating properties

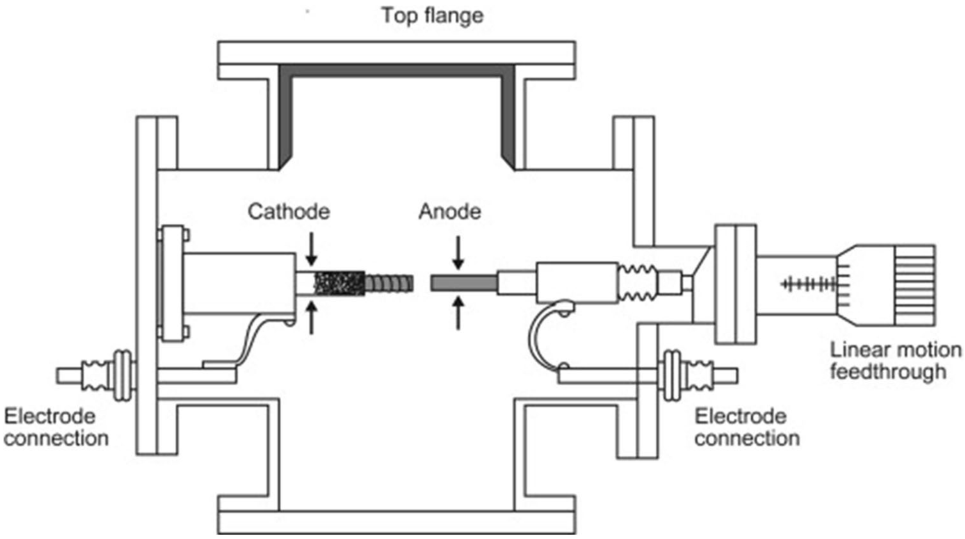


Fig. 3 Arc discharge method synthesis of magnetic nanoparticles [4]

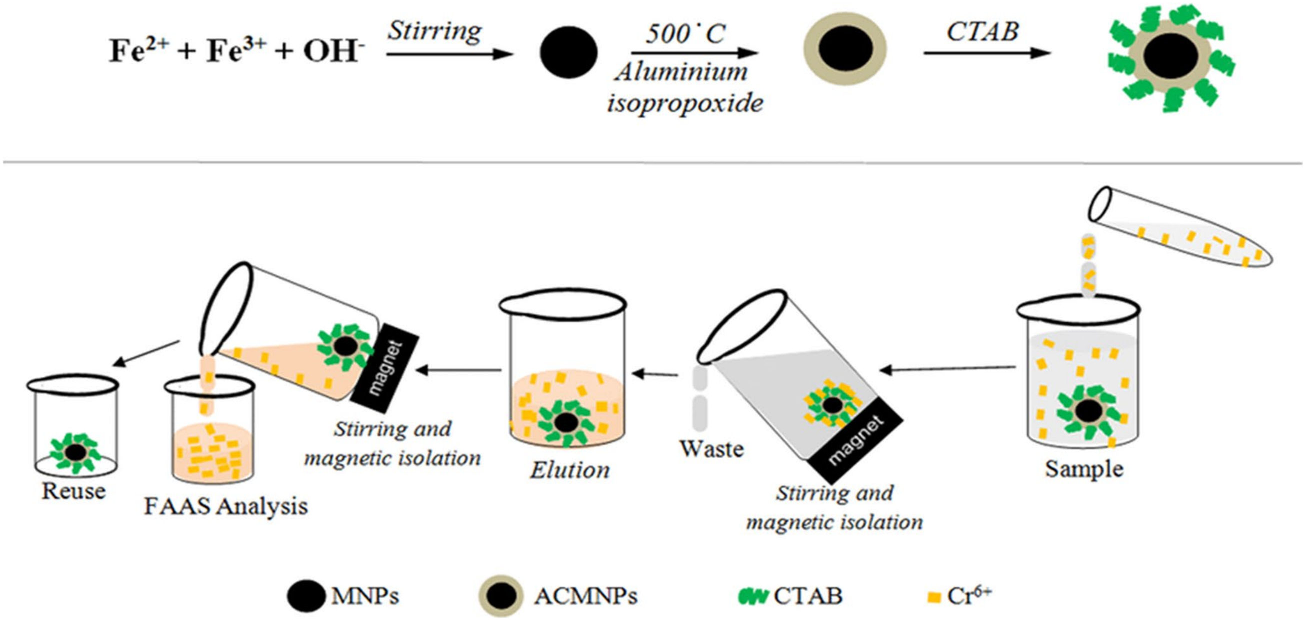


Fig. 4 Protection methods synthesis of magnetic nanoparticles [4]

including toughness, flexibility, time of curing, service performance, exterior weathering, and adhesion [5]. Organic solvents perform several functions. They dissolve the resin, control coating viscosity and evaporation for film formation, and affect film adhesion and coating durability. Other additives and fillers provide coating uniformity and improve coating flow, surface drying, or decrease the permeability of water and oxygen [65–67]. Metal surface preprinting treatments such as phosphate and chromium conversion coatings are applied to increase adhesion of the organic coating. Before applying the top coat, it may be necessary to apply a primer coat that possesses inhibitive properties and good surface adhesion [68–70]. More than one coat provides good mechanical properties, pleasant color and opacity, and good barrier properties (resistance to water and oxygen diffusion to the interface between the underlying meal and the coating). Metal corrosion rate should not exceed more than 1.2–5.0 mm/year with applied liquid coatings [6]. To date, most studies have focused on the development of coatings with surfactants, but today more attention has been focused on coating with polymers due to the repulsion. Numerous methods have been proposed for the stability of magnetic nanoparticles using surfactants and polymers both during and after the synthesis of nanoparticles [32, 33, 71–74]. As shown in Fig. 5, by creating one or two layers on it, they cause the magnetic nanoparticles to remain dispersed. To prevent oxidation of magnetic nanoparticles, the coating should be dense, because one or two thin layers in an acidic environment are easily separated from the surface of the nanoparticles and cause loss of magnetic property [1].

3.2 Inorganic Coatings

Inorganic coatings can be produced by chemical action, with or without electrical assistance. The treatments change the immediate surface layer of metal into a film of metallic oxide or compound which has better corrosion resistance than the natural oxide film and provides an effective base or key for supplementary protection such as paints. In some instances, these treatments can also be a preparatory step prior to painting [13]. The surface of magnetic nanoparticles can be coated with mineral coatings (Fig. 6) such as metal oxides, silica, precious metals, and carbon [34]. A very simple way to protect magnetic nanoparticles is to use metal oxides different from the core as their coating [1, 75–77]. Precious metals such as gold, due to their low reactivity and ability to bridge with other functional groups, can also be used to protect magnetic cores [3]. In this field, the use of coatings made of silica and carbon due to issues such as low cost, low toxicity, good biocompatibility has attracted a lot of attention [2, 78–81].

3.3 Green Synthesis of NPs

Recently, with the development of modern technologies of the nanomaterial synthesis, there was interest in studying the properties of metals at ultra-disperse range as a powder,

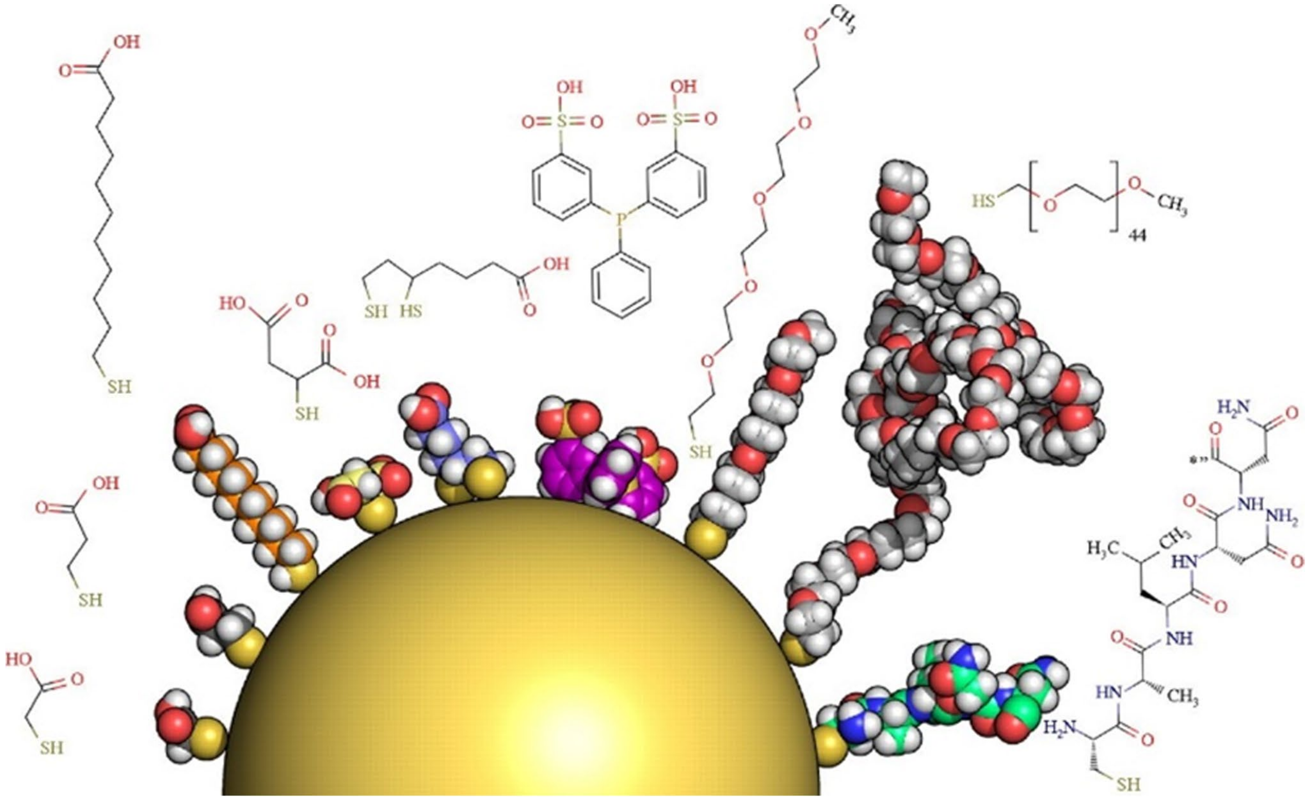


Fig. 5 Some organic coatings used to ensure the stability of magnetic nanoparticles [5]

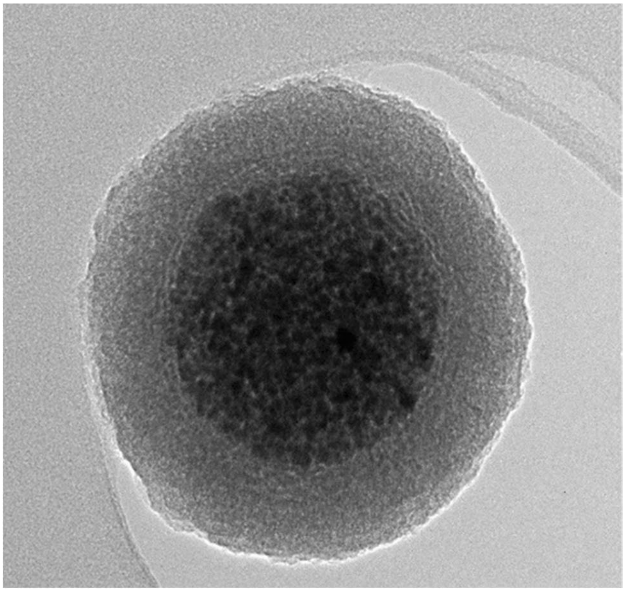


Fig. 6 TEM image of silica-coated magnetic nanoparticles [2]

solution, and suspension. As a rule, the nanoparticles (NPs) may easily form complexes with different substances due to their high chemical activity [14–16]. These complexes have new properties such as good solubility and high biological activity. In this regard, the water dispersion of metal NPs that was obtained by biochemical synthesis using plants shows the ability to absorb, accumulate, and restore inorganic metal ions from the environment. The various organic components, particularly, secondary metabolites that are present in plant tissues, are able to act as stabilizing and reducing agents in the process of NPs synthesis [82–84]. Reduction and formation of NPs occur in the water core of micelles formed by surfactant molecules using natural biologically active substances such as plant pigments from the flavonoid group which ensures long-term stability of NPs and makes this process as safe as possible for the environment [17]. The highest activity and final morphology of NPs is ultimately reached in the last step of green NPs synthesis, when they are coated with plant metabolites (polyphenols, tannins, terpenoids, etc.). Many biological systems of plants can convert inorganic metal ions into metal NPs through the reductive abilities of secondary metabolites present in these organisms. The ability of plants to accumulate and detoxify heavy metals is well proved. Bioactive compounds of plants such as polyphenols, flavonoids, vitamin C, alkaloids, and terpenoids reduce silver (Ag) salts from positive oxidation state (Ag +) to zero oxidation state (Ag0); the mechanism for reduction of Ag + to Ag0 is shown (Fig. 7). Secondary

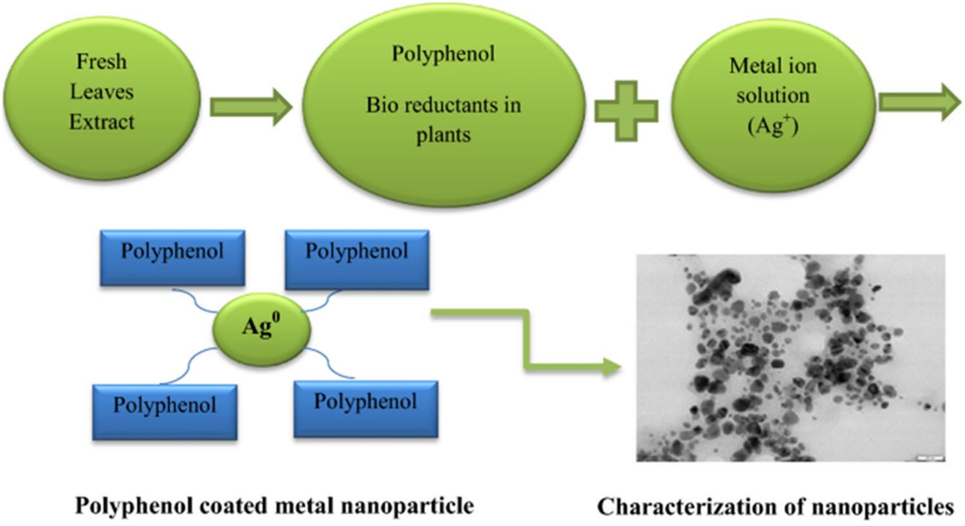


Fig. 7 Pattern of green synthesis. The chemical reaction of NPs synthesis includes several steps. Polyphenols convert positive Ag$^+$ into the zero Ag^0 valent metal, and in the last step of green synthesis, the polyphenols coat metal NPs and affect the morphology and size of NPs [18]

metabolites present in the plant extract affect the size and shape of metallic NPs [12, 18]. These biologically active compounds possess antioxidant activity and are of great interest in the biomedical field as alternative antibacterial agents.

3.4 Chemical Vapor Deposition (CVD)

Chemical vapor deposition (CVD) is a deposition method used to produce high-quality, high-performance, solid materials, typically under vacuum. CVD is the process involving chemical reactions taking place between an organometallic or halide compounds to be deposited and the other gases to produce nonvolatile solid thin films on substrates [85–87]. The key distinguishing attribute of CVD is that the deposition of material onto the substrate is a multidirectional type of deposition, whereas PVD is a line-of-site impingement type of deposition. Microfabrication processes widely use CVD to deposit materials in various forms, including monocrystalline, polycrystalline, amorphous, and epitaxial. In contrast with PVD, in CVD, there is an actual chemical interaction between a mixture of gases and the bulk surface of the material, which causes chemical decomposition of some of the specific gas constituents, forming a solid coating on the surface of the base material. CVD is employed in a wide range of industry applications, such as the deposition of refractory materials (nonmetallic materials that can withstand extremely high temperatures) on turbine blades to greatly increase the wear resistance and thermal shock resistances of the blades [88–90]. Some CVD techniques are atmospheric-pressure CVD, low-pressure CVD, ultrahigh vacuum CVD, plasma-enhanced CVD, microwave plasma-assisted hot filament CVD, metal–organic CVD, photo-initiated CVD, atomic layer deposition, spray pyrolysis, liquid-phase epitaxy, etc. [19]. Chemical vapor deposition (CVD) is a widely used material processing technology. The majority of its applications involve applying solid thin-film coatings to surfaces, but it is also used to produce high-purity bulk materials and powders, as well as fabricating composite materials via infiltration techniques. It has been used to deposit a very wide range of materials. In the late 1970s, it was first found [20] that CVD could deposit diamond films at a pressure lower than 1 atm. Since then, the research on the formation of thin films on different biomaterials by the CVD method has been deepened.

CVD has a number of advantages as a method for depositing thin films. One of the primary advantages is that CVD films are generally quite conformal, i.e., that the film thickness on the sidewalls of features is comparable to the thickness on the top. This means that films can be applied to elaborately shaped pieces, including the insides and undersides of features, and that high-aspect ratio holes and other features can be completely filled. In contrast, physical vapor deposition (PVD) techniques, such as sputtering or evaporation, generally, require a line-of-sight between the surface to be coated and the source[91–94]. Another advantage of CVD is that, in addition to the wide variety of materials that can be deposited, they can be deposited with very high purity. This results from the relative ease with which impurities are removed from gaseous precursors using distillation techniques. Other advantages include relatively high deposition rates and the fact that CVD often does not require as high a vacuum as PVD processes. CVD also has a number of disadvantages. One of the primary disadvantages lies in the properties of the precursors. Ideally, the precursors need to be volatile at near-room temperatures. This is non-trivial for a number of elements in the periodic table, although the use of metal–organic precursors has eased this situation. CVD precursors can also be highly toxic ($Ni(CO)_4$), explosive (B_2H_6), or corrosive ($SiCl_4$). The byproducts of CVD reactions can also be hazardous (CO, H_2, or HF). Some of these precursors, especially the metal–organic precursors, can also be quite costly [95–98]. The other major disadvantage is the fact that the films are usually deposited at elevated temperatures [99]. This puts some restrictions on the kind of substrates that can be coated. More importantly, it leads to stresses in films deposited on materials with different thermal expansion coefficients, which can cause mechanical instabilities in the deposited films.

3.5 Methods of Protection

Three basic methods of protection from chemical hazards exist: engineering controls, personal protective equipment, and administrative controls. Engineering controls are systems and equipment designed to prevent or decrease contact with a chemical. Examples include chemical fume hoods, ventilation fans, and secondary containers. Personal protective equipment (PPE) is protective clothing that is resistant to specific chemicals and acts as a barrier between the wearer and the chemical he or she is handling. Administrative controls are limitations imposed by supervisors to ensure exposures are minimized or eliminated. The supervisor is responsible for ensuring that appropriate controls are in place and used [37–41].

3.5.1 Engineering Controls

Engineering controls are considered the most effective form of exposure control. Before beginning a process or procedure, consider engineering controls that will decrease chemical exposure or risk of harm. Examples include grounding and bonding when transferring flammable liquids; using exhaust ventilation to decrease vapor concentration when using a volatile chemical; and storing hazardous chemicals in cabinets according to hazard class [42].

3.5.2 Personal Protective Equipment

PPE should be worn for protection from hazardous chemicals whenever contact is possible. PPE includes gloves, safety glasses, face shields, Tyvek suits, lab coats, etc. The use of powdered latex gloves is prohibited. A respirator should only be used while engineering controls are being installed or upgraded or when engineering controls are not a feasible option. If respirators are deemed necessary, EH&S must be contacted to determine the correct respirator and provide fit testing, training, and medical screening for users. PPE must be selected according to the chemical hazard involved [100–104].

3.5.3 Administrative Controls

Administrative controls should be used to limit exposure durations. The most common example of administrative control is rotation of workers to minimize the length of time a worker is exposed to a certain chemical. This form of control should only be used under well-documented conditions and after engineering controls have first been considered or used [47–49].

4 Functionalization of Magnetic Nanoparticles

Interactions between NPS and their environment are strongly influenced by the surface groups of NPS [67, 68]. The development of surface modification methods for magnetic NPS to chemically functionalize them and control their solubility is important and strongly influenced by the type of application. For biological applications, for example, the surface of magnetic nanoparticles is often referred to as biomolecules such as proteins [34, 105–109]. Most applications of magnetic NPS require chemical stability, uniformity in size, and proper dispersion in a liquid medium [35, 110–114]. Therefore, the surface of NPS must be modified with appropriate groups. Electrostatic chemical absorption (or addition of a ligand, in ligand chemistry is an ion or molecule that is able to attach to a particular metal or several metals to form a complex) and covalent bonding (ligand exchange) are some of the methods, which are used to change and modify the surface of NPS (Fig. 8) [5, 12, 36–39, 115–120].

5 Conclusion

Biomedical applications like magnetic resonance imaging, magnetic cell separation, or magnetorelaxometry control the magnetic properties of the nanoparticles in magnetic fluids. Furthermore, these applications also depend on the hydrodynamic size. Therefore, in many cases, only a small portion of particles contributes to the desired effect. The relative amount of the particles with the desired properties can be increased by the fractionation of magnetic fluids. Common methods currently used for the fractionation of magnetic fluids are centrifugation and size-exclusion chromatography. All these methods separate the particles via nonmagnetic properties like density or size. The positive charge of the maghemite surface allows its dispersion in aqueous acidic solutions and the production of dispersions stabilized through electrostatic repulsions. By increasing the acid concentration (in the range 0.1 to 0.5 mol l^{-1}), interparticle repulsions are screened, and phase transitions are induced. Using this principle, these authors describe a

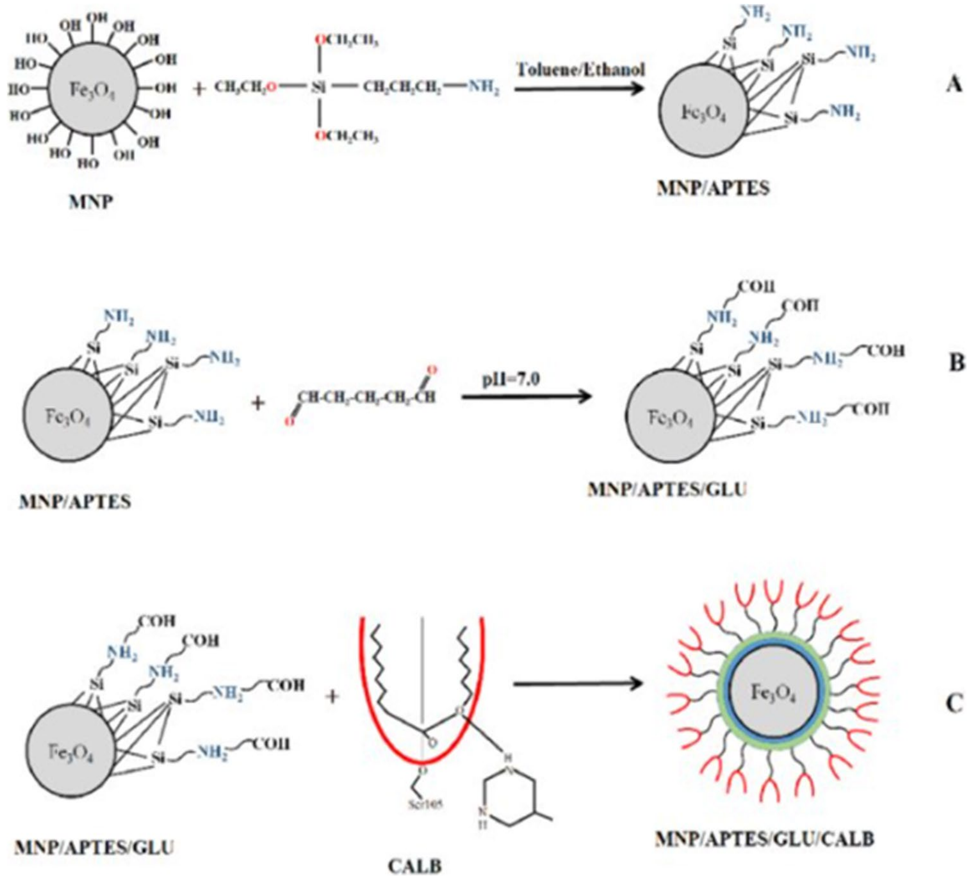


Fig. 8 **A** Functionalization of magnetic nanoparticles with 3-aminopropyl triethoxysilane in toluene and ethanol, **B** glutaraldehyde reticulation of magnetic NPS after 3-aminopropyl triethoxysilane treatment, and **C** *Candida antarctica* lipase B immobilization on 3-aminopropyl triethoxysilane functionalized magnetic NPS after glutaraldehyde reticulation [5]

two-step size sorting process in order to obtain significant amounts of nanometric monosized particles with diameters between typically 6 and 13 nm. As the surface of the latter is not modified by the size sorting process, usual procedures are used to disperse them in several aqueous or oil-based media. Preference should be given, however, to partitions based on the properties of interest, in this case, the magnetic properties. So far, magnetic methods have been used only for the separation of magnetic fluids, for example, to remove aggregates by magnetic filtration. Recently, the fractionation of magnetic nanoparticles by flow field-flow fractionation was reported that field-flow fractionation is a family of analytical separation techniques, in which the separation is carried out in a flow with a parabolic profile running through a thin channel. An external field is applied at a right angle to force the particles toward the so-called accumulation wall. Advances within the synthesis of magnetic NPS, especially within the last 20 years, have led to the event of a good range of those NPS, in numerous sizes and controllable. However, one amongst the unavoidable problems these related to NPS is their inherent instability over long periods of your time. On the opposite hand, issues like very high reactivity and toxicity to some magnetic NPS limit their use. Research during this field has shown well that to beat these problems, coating these NPS using organic and inorganic molecules is one amongst the foremost effective solutions. In recent years, the functionalization and modification of the surface of magnetic NPS has significantly increased the potential of using these NPS in several fields.

Acknowledgements The authors acknowledge the Department of Chemical Engineering, Arak Branch, Islamic Azad University, Arak, Iran, and the Young Researchers and Elite Club, Gachsaran Branch, Islamic Azad University, Gachsaran, Iran. The authors acknowledge the support of the Deanship of Scientific Research at Prince Sattam bin Abdulaziz University.

Author Contribution “I wrote to you in regard to your question about naming some people in my article, I must point out that in some cases, help was sought from people and it was necessary to mention the names of these people in order to maintain professional ethics in research issues.”

Therefore, on this basis:Mohammad Javed Ansari, Mustafa M. Kadhim, and Baydaa Abed Hussein: investigation, concept and design, experimental studies, writing—original draft, reviewing, and editing, Holya A. Lafta, Ehsan kianfar: investigation, concept and design, data curation, conceptualization, writing—original draft, reviewing, and editing.

Funding None.

Declarations

Conflict of Interest None.

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