Iranian Journal of Chemistry and Chemical Engineering (IJCCE)

Chemical Methods for Producing Iron Oxide Magnetic Nanoparticles: A Review

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Abstract

In recently magnetic nanoparticles (MNPs) have attracted a lot of attention in the field of biomedicine because of distinctive physicochemical characteristics, chemical stability, biocompatibility, nontoxicity, and ease of external magnetic field guidance. It has been outlined how MNPs with controlled size, shape, and magnetic characteristics are prepared for use in industrial, bioengineering, and commercial applications. In fields of life science such as agriculture, the environment, and healthcare, iron oxide nanoparticles (IONPs) are essential. By coating the IONPs with proteins, starch, polymers, etc., the IONPs can be further enriched. Applications of biomedical are also discussed, including drug administration, hyperthermia, biosensing, and bioseparation. We can treat the tumor by directly injecting IONPs into the organ and using an external magnetic field. The preparation techniques for IONPs, coatings of organic and non-organic compounds, and biological applications have all been outlined in this review. In addition, the surface of iron oxide NPs may be changed by inorganic or organic compounds such as metals, polymers, proteins, silica, etc. The guidelines for the synthesis and surface functionalization of iron oxide NPs are discussed, as well as the problems and major challenges. In-depth discussion has been given in this study of the prospective developments and trends of IONPs in medication delivery and hyperthermia.

Keywords: Iron Oxide, Magnetic Nanoparticles, Hyperthermia, Microemulsion, Bio-sensing

1. Introduction

Iron oxide is widespread in nature and because of its unique properties, this has been used rapidly for the development of various applications like biomedical, environmental remediation, agriculture, and food. Though there are several materials or elements i.e. gold, nickel, cobalt etc., we choose to use iron because it has better magnetic properties than others. Iron oxide is formed by a chemical reaction of iron with oxygen [1–4] and it stands as a pillar of the present-day infrastructure. 16 iron oxides are identified, among those only 3 iron oxides which are commonly used are hematite (α-Fe_2O_3) maghemite (γ- Fe_2O_3), and magnetite (Fe_3O_4). These are popular because of their property of size reduction, tunable shape and size easily designed preparation method, biocompatibility, and biodegradability.

Hematite (α- Fe_2O_3) - It is a widely found mineral on the earth's surface that ranges in color from reddish-brown to black and is composed of ferric oxide. It is a significant source of iron and can be found as crystals or as earthy red ochre. It has low cost and high resistance to corrosion [5].

Magnetite (Fe_3O_4) **-** An iron oxide with equal quantities of iron (II) and iron (III) makes up the majority of the mineral magnetite. Iron (II, III) oxide is a common expression for this compound, which has the empirical formula Fe_3O_4. It was formerly known as ferrous-ferric oxide and tri-iron tetra oxide. It has faced centered cubic structure and is close-packed and the band gap lies in the range 2.2-2.5 eV [6].

Maghemite (γ- Fe_2O_3) **-** Maghemite is a kind of iron oxide mineral commonly known as gamma-Fe2O3. It is highly magnetic and remanence, and its chemical makeup is like ferric oxide (Fe_2O_3). Maghemite is metastable in relation to hematite and joins magnetite to form a continuous metastable solid solution; titanium can take the place of iron to generate titanomaghemite. Magnetite oxidizes naturally to produce maghemite. It has an isometric structure, a flawed spinel shape, and some iron deficiency. It is extremely successful in the breakdown and eradication of pollutants because it functions as both a photocatalyst and a magnetic nanomaterial [7]. The band gap of maghemite is around 2.0 eV [8].

The characteristics of IONPs are considerably influenced by the size and form of NPs. The manufacturing process and chemical composition of IONPs determine their properties. The biomedical applications of IONPs include magnetic imaging, magnetic cell isolation, tissue regeneration, bio-sensing, hyperthermia, and drug delivery. An essential characteristic of IONPs is their tunability, which enables to tailor them for different application [9].

Functionalized IONPs are of great interest due to their diverse applications, especially in nano-medicines. These barriers can be surmounted by surface modification of IONPs, which enhances their physicochemical properties. Levy et.al [10] synthesized 6-18 nm IONPs but there were drastic loss in hyperthermia testing. When size of IONPs was less than 20 nm, it becomes super paramagnetic at room temperature [11]. One-dimensional IONPs are very beneficial due to their unique features and properties [12]. There have been reports on various morphologies of IONPs in particular nanorods, nanocubes, nanohusk beside others. As we know, NPs are forefront of nanotechnology development so they can be synthesized by variety of methods i.e. mechanical, co-precipitation, sol-gel, micro emulsion, reverse micelle, hydrothermal and biological methods divided into two routes firstly aqueous, it has low cost, sustainability, water-soluble MIONPs without size selection. Secondly, non-aqueous, IONPs can be obtained when dissolved in nonpolar solvents, surface modification is very important to increase the compatibility in biosystems. Bowden et al [13] have reported that by using one-step hydrothermal technique highly crystalline Fe_3O_4 nanoparticles can be prepared without the use of surfactant. The saturation magnetization of the nanosized Fe_3O_4 powder (40 nm) produced after 6 hours at 140 °C was 85.8 emu/g, which was somewhat lower than that of the corresponding bulk Fe_3O_4 (92 emu/g). It was also proposed that the higher saturation magnetization in nano-sized Fe_3O_4 is caused by crystallized Fe_3O_4 generated under proper hydrothermal conditions [14]. The chemical reaction for the preparation is given in the Equations (1-4) below.

$$Fe^{3+} + H_2O \xrightarrow{\text{(Deprotonation)}} Fe\,(OH)_x^{3-x} \quad \cdot\cdot\ (1)$$

$$Fe^{2+} + H_2O \xrightarrow{\text{(Deprotonation)}} Fe\,(OH)_y^{2-y} \quad \cdot\cdot\ (2)$$

$$Fe\,(OH)_x^{3-x} + Fe\,(OH)_y^{2-y} \xrightarrow[\text{Dehydration (pH} \sim 9,\ 60^0C)]{\text{(Oxidation)}} \text{Magnetite } (Fe_3O_4) \cdot\cdot (3)$$

$$6Fe(OH)_2 + O_2 \rightarrow 2Fe_3O_4 + 6H_2O \quad \cdot\cdot\ (4)$$

Entropy production and triple diffusive convection have yet to be investigated in a flow through a porous media, taking into account the combined effects of suction and magneto-hydrodynamics [15]. The irreversibility of mass diffusivity caused by a magnetic field has a significant impact on mass and heat transport rates [16,17].

Synthesis Method	**Advantage and Comparison**

Co-Precipitation Method	This method is useful in obtaining smaller size with better morphology. NPs can be easily controlled, by varying different salts like sulphates, nitrates and chlorides.
Sol-gel Method	It is built on condensation and hydroxylation of used precursors in solution which is origin of ‘sol’ of nanometric particles. The microstructure and related properties of gel are affected by concentration, temperature, and nature of solvent and pH value. This method also gives high crystallinity and high magnetic saturation.
Liquid phase method	This method is simplistic and helps in forming the controlled shape and size of NPs. The controlled NPs with super-paramagnetic property were prepared by using alkaline precipitation of $FeCl_2$ and $FeCl_3$. The particles are in the range 4 - 16 nm.
Hydrothermal and Solvothermal method	These reactions are performed using aqueous solution route to develop Fe_3O_4 and α-Fe_2O_3 nanoparticles at temperature 200°C and pressure more than 2000 psi. The key advantage of hydrothermal method over other methods is the better crystal growth and crystalline phases
Sonolysis	High intensity ultra-sonication is required for the preparation of NPs. The sonolysis method provides uniformity of mixing and crystal growth reduction which leads to increase the rate of reactions.
Microemulsion	Microemulsion is stable fluid in terms of thermodynamic, and it is different from kinetically stable emulsion, over the time it gets separated into oil and water. The small sizes of NPs are assigned from higher specific surface area observed in o/w (direct) micro-emulsion.

Table1: Comparison of various synthesis methods

Iron oxide nanoparticles play substantial role in agriculture, the environment, and biomedicine. These nanoparticles can penetrate agricultural land via both direct and indirect routes. This paper provides an overview of the fate and transit of metal-based NPs in agricultural ecological systems, as well as their interactions with plants [18]. Because of their unique properties, microbially synthesised IONPs have significant potential for biological, therapeutic, and environmental applications. The IONPs are made up of an iron oxide core that is magnetised and covered with biocompatible molecules. The small size, high magnetic, and biocompatibility of IONPs make them suitable for use in medical imaging [19]. IONPs can be modified by proteins, starch, and polymers to enhance and modify them for associated benefits. IONPs are resistant to oxidation and agglomeration when coated with hydrophilic natural polymers such as chitosan, starch, and dextran, as well as synthetic polymers such as poly(ethylene phthalate), poly(ethylene glycol), and poly(vinyl pyrrolidone) [20,21]. Because of the unique magnetic properties, biocompatibility, and non-toxicity of iron oxide nanoparticles (magnetite), it has been widely used in biomedical research, magnetic resonance imaging(MRI), drug delivery for cancer treatment, catalysis, sensors, waves absorbing materials and electromagnetic coatings [22].

Magnetic nanoparticles such as Fe_3O_4 NPs are being studied for cancer treatment under clinical hyperthermia conditions. Microwave-based hyperthermia investigations in physiological saline under clinical settings enhanced the samples temperature due to the translation of radiation to heat energy [23]. Magnetic nanoparticles have used in biomedicine such as magnetic cell isolation, hyperthermia, biosensing, tissue regeneration, magnetic imaging, and drug administration. Tunability is an important property of IONPs. It enables them to be customised for each application. It then concentrates on applications like magnetic detection (including imaging), magnetic cell separation, and magnetic particle-based therapies like hyperthermia, magneto-mechanical tumour destruction, localised drug administration, and tissue engineering [24]. Because of their wide range of uses, functionalized IONPs are of tremendous interest, particularly in nano medicine. Surface alteration of IONPs can overcome these barriers by improving their physicochemical qualities. The recent developments in IONP surface modification using inorganic materials, polymers, and small organic molecules are summarized in this work. The preparation methods, workings, and uses of IONPs with different surface modifications are explained. IONPs, however, have two main issues: their large surface area, chemical reactivity, and high surface energy cause them to quickly aggregate and oxidize into the physiological milieu of the tumours, which causes them to lose their magnetism. [35].

To make IONPs biocompatible, proper surface modification is necessary. Coating is the most often used surface modification process for conjugating organic or inorganic compounds onto the surface of IONPs [25,26]. This review paper highlights the synthesis methods of iron oxide nanoparticles, including co-precipitation, microemulsion, sol-gel, and additional chemical methods like hydrothermal, electrochemical, sono-chemical decomposition, and thermal decomposition, because these particles have a wide range of biomedical applications. [27]. There has been extensive discussion of the possible developments and trends of Fe_3O_4 NPs in medication delivery and hyperthermia. A thorough investigation was carried out into the morphological, structural, physicochemical, and magnetic properties of magnetic drug nanoparticles that were based on polymer, layered double hydroxide (LDHs), and drug as coating agents and iron oxide nanoparticles (IONPs) as the core [28]. Magnetic hyperthermia is an oncological treatment that uses magnetic nanoparticles activated by radiofrequency magnetic fields to generate a regulated temperature increase in sick tissue [29].

2. Preparation Methods of iron oxide NPs

IONPs can be obtained by the three various methods that are physical, chemical and biological method. Physical method, it contracts the nanometer range particles size. Some well-known methods of physical methods are Ball milling method, Electron beam lithography, Deposition of gas phase and Laser pyrolysis method. The second method is chemical method. This is a simple and efficient method to control shape, size and composition. The some of the well-known methods are sol-gel method, micro-emulsion, electrochemical, chemical co precipitation and oxidation. The third way, the biological approach, provides high yield, cheap cost, and good reproducibility, but it is a bit of a long procedure, so we avoid employing it. Among all these methods, chemical method is most preferable method because it has low production cost and high yield. There are different routes of synthesizing nanoparticles as shown in **Figure 1**.

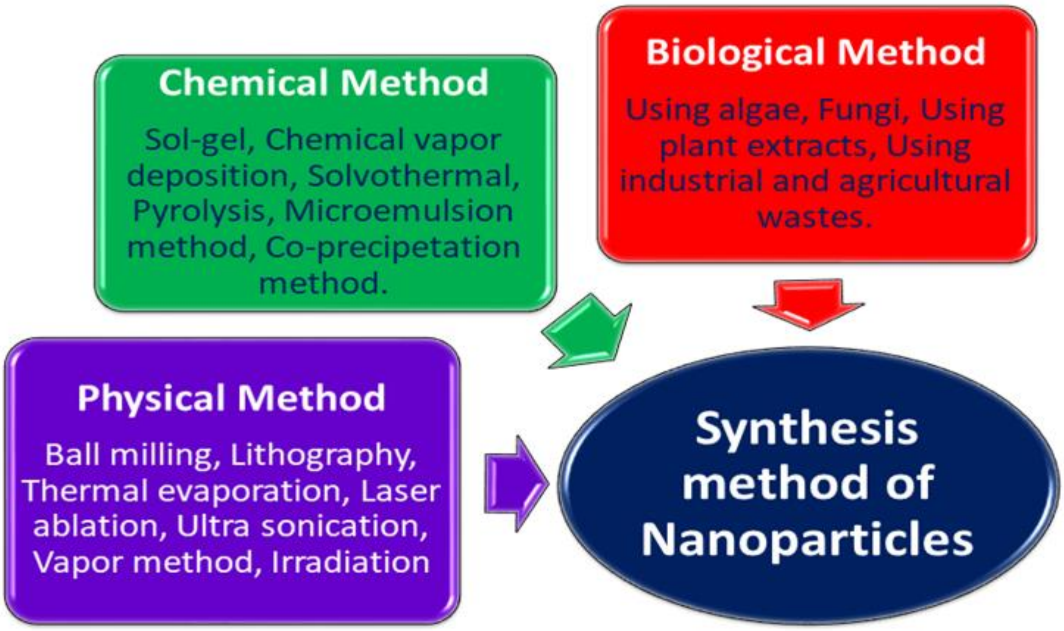

Fig. 1. Different routes for synthesizing IONPs

2.1 Co-precipitation Method

In the co-precipitation method, a basic solution containing ferric and ferrous ions is mixed in a 1:2 ratio at a high temperature [30]. This process makes it simple to manage the form and size of the nanoparticles by first adjusting the various salts, such as sulphates, nitrates, and chlorides. Secondly, using the variation in the ratio of Fe^{2+} /Fe^{3+} and, thirdly, by adjusting the solutions pH level and temperature [31]. The entire co-precipitation process can be carried out in a N_2 environment. A black suspension is eventually acquired. The precipitated particles can be separated from the suspension using an external magnetic field, then they can be washed repeatedly with ethanol and three times with distilled water [32]. Magnetic nanoparticle targeting, namely magnetite-based nanoparticles, is mainly recognized as a promising targeted delivery approach among the several methods used for this purpose [33]. The effects of a number of variables, including pH on adsorbent synthesis, contact time, adsorbent volume, and the ability of magnetic iron oxide nanoparticles coated on sand (MIONCS) to adsorb arsenic, were investigated [34].

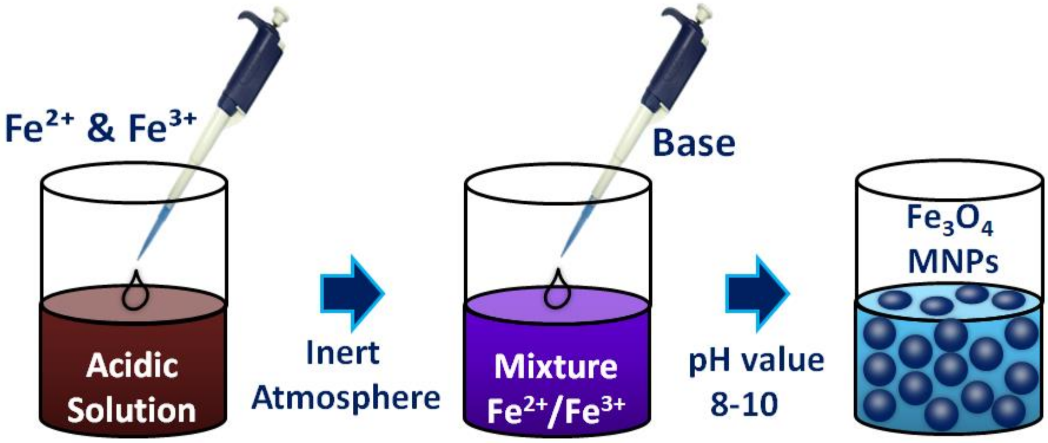

Fig. 2. The schematic showing co-precipitation diagram

The ratio of ionic, bridging, and bidentate coordination modes in the iron oleate precursor is important for improving the synthetic quality of these magnetite NPs. The precursor was dried for 21 hours to obtain a suitable ratio of weakly and highly coordinated ligands. As a result, a decomposition window was formed, supporting several nucleation and growth processes, and producing NPs that were impressively homogeneous in size and shape [35]. The success of this NP system is due to the adjustment of three key parameters: saturation magnetization, magnetic anisotropy, and dipolar interactions [36]. It is impossible to overstate the importance of octahedral shape for dynamical hysteresis loop coercivity amplification and, consequently, for the heating capacity of the NPs [37,38]. The development of the Fe_3O_4 nucleus is simpler at pH values higher than 11, whereas the nucleation of the Fe_3O_4 nucleus is simpler at pH values lower than 11. The co-precipitation method's schematic diagram is shown in **Figure 2**.

Chemical Reaction

$$2Fe^{3+} + Fe^{2+} + 8OH^{-} \rightarrow 2Fe(OH)_3 + Fe(OH)_2 \rightarrow 4H_2O + Fe_3O_4\downarrow$$

Wu et.al. [39], have reported that by using co-precipitation method Fe_3O_4 nano-powders can be synthesized with 15 nm in size using ultrasonic chemical co-precipitation method which also gives better separation of iron oxide particles with excellent purity.

Today we don't need protection gas for the synthesis method; it flexible gives super paramagnetic iron oxide nanoparticles (SPIONs) of Fe_3O_4 NPs. As a result, we have gained size in the range 4.9 - 6.3 nm of Fe_3O_4 NPs by using simple one step aqueous co-precipitation method by using alkanolamines as base. This method is easy, adaptable, practical, profitable way for high efficiency IONPs with modified properties and smaller in size [40]. Generally small size shows low magnetic properties using aqueous co-precipitation as alkanolamines as base shows magnified properties with smaller size. Liu et.al. [41], have reported, that by using 2% chitosan in place of water in acetic acid solution Fe_3O_4 NPs coated with chitosan was prepared by co-precipitation route under the influence of 0.45 T magnetic fields. Suh et. al. [42] have prepared the non-spherical MIONPs in presence of carboxyl putted into polymer matrix where the iron oxide ions disperse into polymer particles and in polymer particles IONPs are increased. All these are the surfactants and bimolecular which are straightly induced in co- precipitation process. The co-precipitation method is a simple, versatile, and successful technique and can be used to add multiple particles, like adding of bimolecular in succeeding reactions. Ortega D et.al. [43], synthesized coating free IONPs by slow reaction of the result by using Sodium carbonate with co-precipitation method. Pereira et. al. [40], have synthesized the different super paramagnetic NPs of Fe_3O_4, Co-Fe_2O_4 and Mn-Fe_2O_4 by one-step aqueous co-precipitation method by using different agent like alkaline, disopropanolamine, alkanolamines and isopropanolamina. As a result, the obtained NPs are small with respect to NaOH and this method also increased saturation magnetization. It is noteworthy that this method is useful in obtaining smaller size with better morphology. Therefore, more attention should be given to short coming like variation in of particle size and utility of strong base in reaction.

2.2 Sol-gel Method

Sol-gel is wet route suitable for synthesis of nano-structured metal oxides [44]. This method is mainly used for the materials fabrication. It is generally built on condensation and hydroxylation of used precursors in solution which is origin of 'sol' of nanometric particles. To obtain 3D metal oxide the sol is dried or "gelled" using a chemical reaction or by the solvent removal. A base or an acid can hydrolyze the precursors, but water is the solvent of choice. The acid catalysis forms of polymeric form of gel and the basic forms colloidal gel [45]. All this process is performed at room temperature and it acquire or form the crystalline state [46]. Temperature, solvent type and concentration, and pH level all affect the structure and related properties of gel. [47]. Additionally, we have demonstrated that supercritical ethyl alcohol (EtOH) can be used to create Fe_3O_4 nanoparticles via a sol-gel method. In order to regulate the final nanoparticles' size, the esterification reaction is preferred for the production of nanostructured metal oxides [44].

The advantages of sol-gel methods are-

1) Probability to attain predetermined structure according to experimental condition.

2) Prospect to get pure phases, mono dispersity, and fine particles.

3) Good control of microstructure and uniformity of the products.

4) Control over the characteristics and stability of the nanoparticles.

Iron oxide silica aero-gel is more conventional than iron oxide because it has more magnitude hence greater reactive of 2 to 3 orders [48]. The silica aero-gel helps in increasing the reactivity of iron oxide nano particles because it attributes to iron oxide nanoparticles with large surface area [49,50]. In alcoholic aqueous solution the TEOS and Fe(III) precursors were dissolved and to get final material they are dried and heated [51,52].

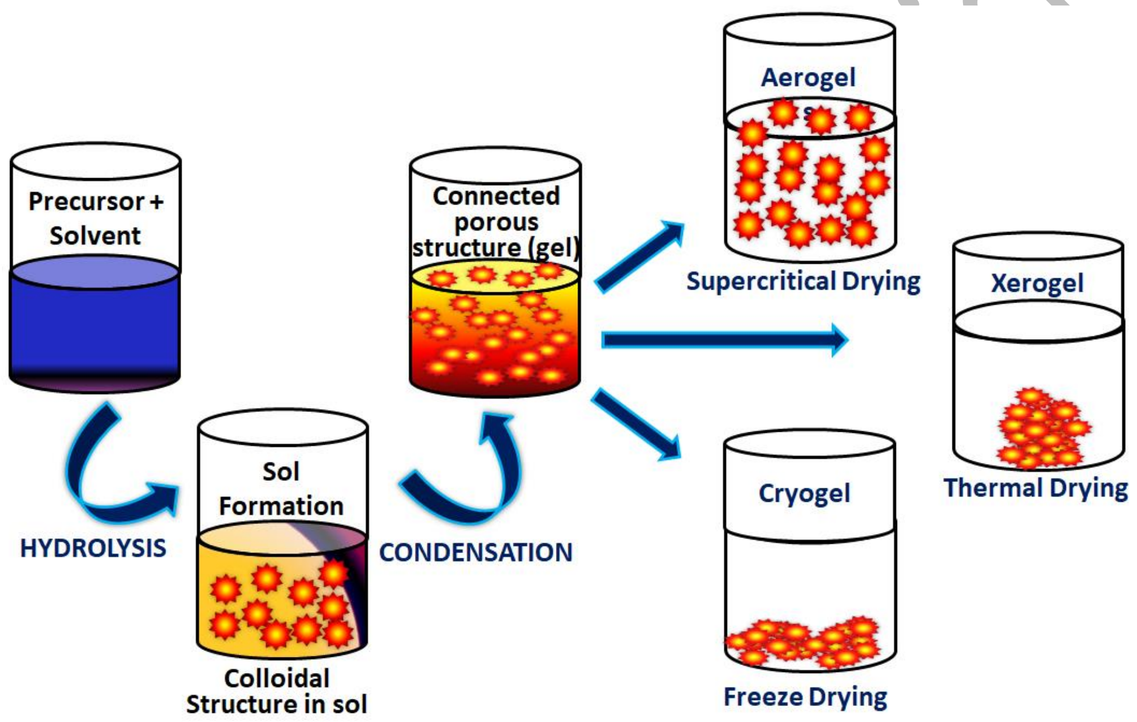

Fig.3. Experimental flow chart of Sol Gel Method

There are different precursors which are important for monitoring size and shape of iron oxide nanoparticles for example Erin Camponeschi et. al. [53] reported that by adding sodium benzene sulfonate (Na DDBS) as surfactant has resulted in constant size of iron oxide nano particles not the 3D gel network. The properties of iron oxide and particles depend on structure created by the soldier process. Lemine et.al. [54], have produced 8 nm-sized particles using the sol-gel technique at room temperature, with a saturation magnetization of roughly 47 emu/g. Qi et al. [50] have reported that the non-alkoxide sol-gel process was used to create particles with a size range of 9~12 nm. With this method, ethanolic solution can be used to produce polymeric gel materials without the need for alkoxide. The manufacture of nanorods with phase control relies on circumstances was described by Woo et al. [51]. Temperature, gel hydrous condition, and environment all affect phase. Additionally, during the gelation process, the oleic-acid ratio can be used to track the diameter and length of the manufactured nanorod. The sol-gel method's experimental methodology is depicted in Figure 3. The polyol approach, which produces iron oxide nanoparticles with varying sizes and shapes, is the opposite of the sol-gel method. [55].

This polyol functions as a reducing agent and solvent, aiding in particle development and preventing the buildup of inter-particles [56]. High temperatures are not necessary for this process; the iron precursor is dissolved in liquid polyol, then heated to a specific temperature to reach the boiling point of the polyol. The size, magnetic characteristics, shape, and colloidal stability of IONPs are all influenced by the polyol solvent [57]. The sol-gel method and polyol method are observed better as compared to co- precipitation method [58]. The only disadvantage of sol-gel process is high priced and discharge of alcohol during calcinations. Though the surface of IONPs constitutes hydrophilic legends, it can be dispersed in polar and aqueous solvents. This method also gives high crystallinity and high magnetic saturation [59].

2.3 Liquid phase method

This method is simplistic and helps in forming the controlled shape and size of MIONPs [60]. In this method nucleation and growth of nuclei takes place through homogeneous precipitation reaction which leads to particles with uniform sizes [61]. Lamer et.al. [62], proposed the classical model in which nuclei is allowed to diffuse slowly as a result it grows uniformly until the final size is obtained. Nucleation should be avoided during the growth period to obtain mono dispersity. Generally, we use aqueous solutions in co-precipitation but there are other liquid solvents which can also be used. By reaction of Fe (II) salt as base and lenient oxidant in aqueous solution gives 30 nm to 100 nm range of spherical magnetite particle [63]. **Figure 4** shows the liquid phase method diagrammatically. The particles size can be monitored by varying the pH, ionic strength and can be obtained particle from 2-15 nm [64].

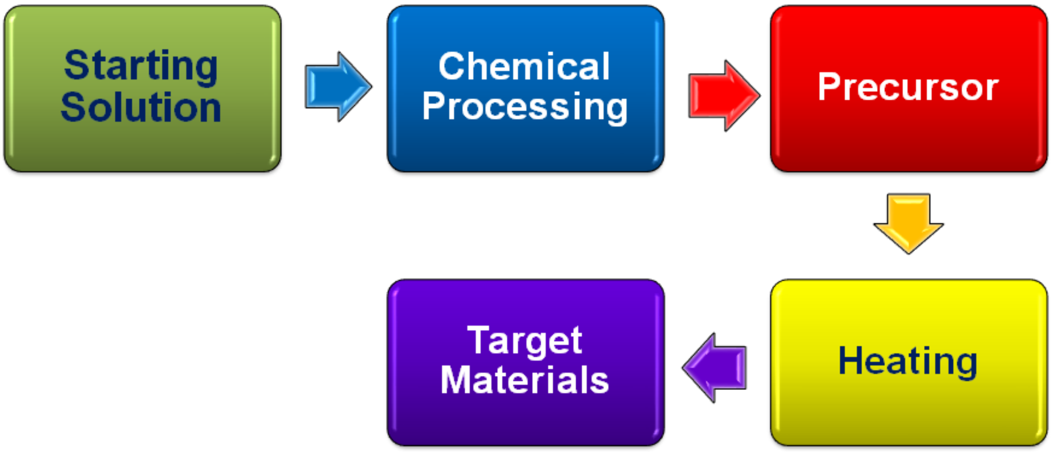

Fig. 4. Flow chart of typical liquid phase method

To stabilize the interruption of NPs anionic surfactants are added as dispersing agents [65,66]. Coating the surface of NPs with electrolytes, proteins [67], starches [65,68] can be done for its stabilization. The purpose of coating is to stop from coagulation to occur at electrolyte concentration. Massart [69] was the first, who prepared the controlled iron oxide particles with superparamagnetic property by using alkaline precipitation of $FeCl_2$ and $FeCl_3$. The shape of the Fe_3O_4 particles were around spherical with 8nm of diameter measured using X-ray diffraction technique [70]. The parameter which effect the precipitation are base (ammonia, NH_3), pH, cations added (K^+ , Li^+ , Na^+ and $NH^+{}_4$) and ratio of Fe^{+3}/Fe^{2+} in the reaction, In this method, particles of the range 16.6 nm to 4.2 nm can easily be obtained [71].

2.4 Hydrothermal and Solvothermal method

These reactions are performed using aqueous solution route to develop Fe_3O_4 and α-Fe_2O_3 nanoparticles at pressure more than 2000 psi and temperature 200°C. Though there are other non-aqueous solutions are also used to synthesize monodisperse, crystalline and controlled shape are used as precursors [72]. In this method Hao and Teja [73] studied the effects of precursor's concentration, residence time and temperature on size of particles and morphology. **Figure 5** shows schematic diagram of apparatus used by Teja for Hydrothermal method. As the concentration of precursor is increased the size of particles also increased and hence size distribution increases. The residence time also have significant of average particle size, as short the residence time, monodispersed particles are produced [74]. They observed that the shape of particles was rhombic, and some are small spherical particles with concentration 0.03M to 0.06M and radius of particles 15.6± 4.0nm to 27.4±7.0 nm. The hydrothermal and solvothermal method both include different wet chemical techniques. The substance is sealed in container at high temperature 130° to 250°C in aqueous and non-aqueous solution with 0.3-4 MPa vapor pressure [75]. These methods are generally used to obtain highly crystalline IONPS than the other methods. Main advantage of hydrothermal method over other methods is the better crystal growth and crystalline phases. The hydrothermal process makes it simple to manufacture materials with high vapor pressure close to melting point [76].

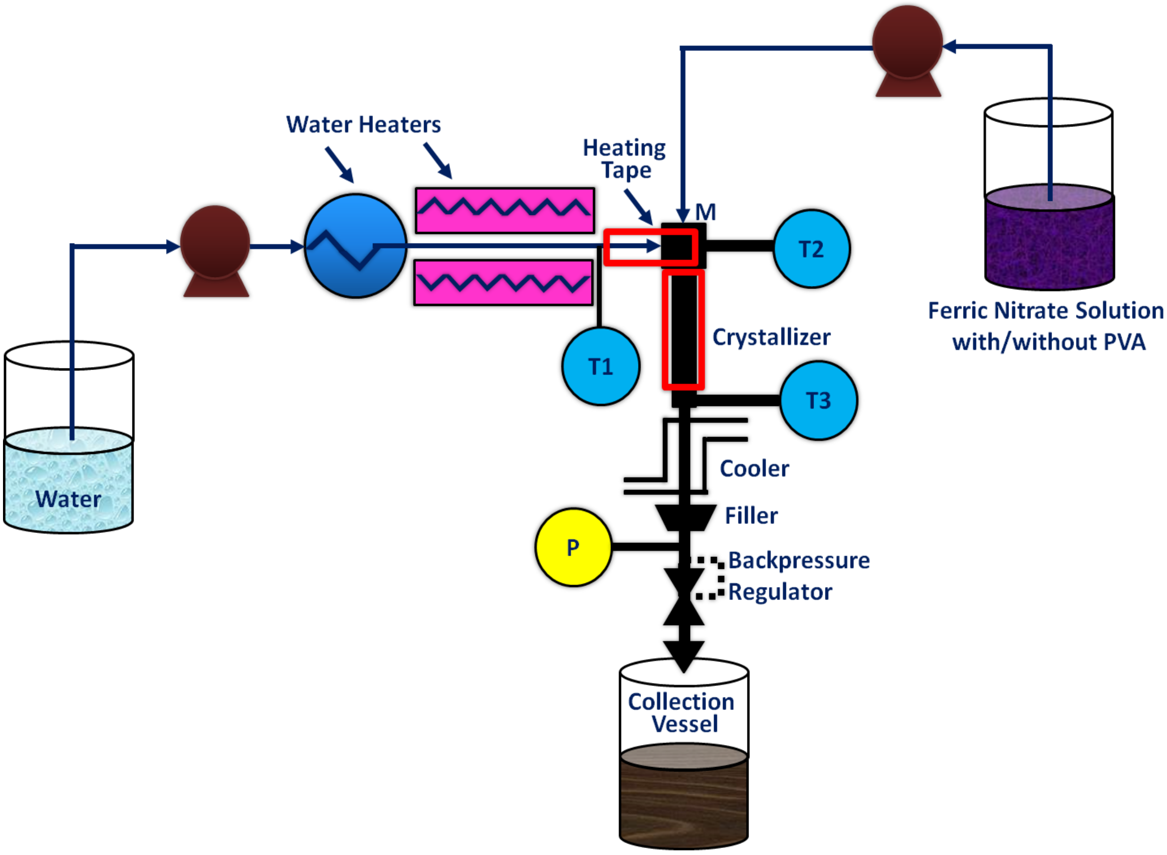

Fig. 5. Schematic diagram of apparatus used by Teja for hydrothermal method, (Adapted from [73]).

This method is particular about good quality iron oxide nano crystals growth which also maintains their composition. The ideas behind the hydrothermal process led to the development of the solvothermal method, which uses an organic solvent in place of water to produce non-aqueous solutions. Using the hydrothermal and solvothermal methods, we can readily produce hollow iron oxide nanoparticles with regulated size and shape [77]. For example, Ma et. at. [78], have reported that there are certain parameters such as reaction time and solvent which plays a vital role in synthesizing the controlled nano structures. Tian et.al. [79], have approached solvothermal method for very small synthesizing monodisperse iron oxide nanoparticles with size control 1 nm (Fe_3) acted as source of iron, reductant is n-octylamine and solvent is n-octanol. Thus, to obtain the shape controlled IONPs, it is beneficial to use hydrothermal and solvothermal route by adjusting the parameters like temperature and pressure hence we can easily obtain highly crystalline iron oxide nano particles including Fe_3O_4, α-Fe_2O_3 and γ- Fe_2O_3 nanoparticles.

2.5 Sonolysis

The ultrasound generated in acoustic cavitations from chemical is the basis of the sonolysis method. The high temperature, long time period in reaction and high pressure are not needed to obtain the novel structure, only high intensity ultrasound is required for the preparation of required nanoparticles [80]. The potential broad and compact acoustic waves during elevation ultrasonography cause bubbles to form and oscillate. The oscillating bubbles collect ultrasonic energy while increasing to a certain size. There are certain circumstances bubble can grow and later it collapses, while releasing the stored energy in bubble in a short period of time. By the sonication of ferrous salt and aqueous ferro solution, anyone can synthesize different types of unadorned and functionalized IONPs under the ambient temperature [81]. Usually, the sonolysis method is used to produce biocompatible IONPs. Theerdhala et. al. [82], have focussed on the surface of IONPs there is combining of semi essential amino acid, L-arginine which creates constant aqueous suspension by using sono-chemical synthesis. Thus, the iron oxide and particles become promising drug delivery vehicle [83]. Zhu et.al. [84], have reported that by using sono-chemical method, produced Fe_3O_4 NPs in the size range 30-40 nm, which were then evenly distributed on the sheets of reduced graphene oxide (Fe_3O_4/RGO). Later, Fe_3O_4/RGO was compromised with haemoglobin to create a biosensor for sensing H_2O_2 . The biosensor gives fast result for sensing H_2O_2 and displays the good linear relation. **Figure 6** shows the flow chart of sono-chemical procedure of IONPs.

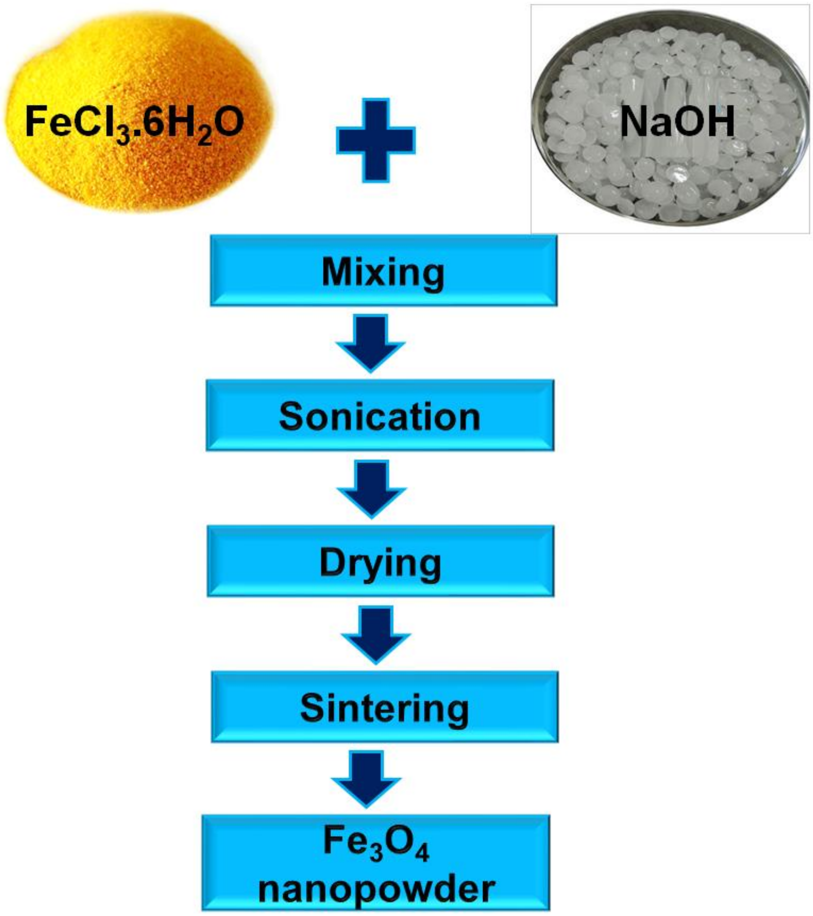

Fig. 6 Flow chart of sono-chemical procedure of IONPs

In latest technology, we can produce nanocomposites with different range, which contains multiple combination of various polymers and summarize materials. Toe et.al. [85], have reported a simple, easy, and efficient approach for synthesizing 100nm latex beads which is loaded with high contented of Fe_3O_4 NPs under ultrasound-initiated effect. The prepared nanoparticles have excellent colloidal stability (they are stable for more than a year in aqueous solution) with very good magnetic properties (24 emu g^{-1} saturated magnetization) and of various desirable size. The sonolysis method is not much good in fabrication of iron oxide nanoparticles of controllable shapes and dispersity but beneficial in uniformity of mixing and crystal growth reduction which leads to increase the rate of reactions.

2.6 Microemulsion

Microemulsion is stable fluid in terms of thermodynamic, and it is different from kinetically stable emulsion, over the time it gets separated into oil and water. The variation in particles sizes is observed to be in the range of 10-300 nm and because of this small particle size micro emulsion look clear, stable and translucent solution. Surfactant molecules create a monolayer at the water-oil interface, with the hydrophilic head group of the molecules dissolving in the aqueous phase and the hydrophobic tails dissolving in the oil phase. Direct and reversed are the two elementary mechanism of microemulsion, firstly, oil may disseminated in water and secondly, water disseminated in oil which help in synthesizing of controlled shape and size of IONPs [86] mainly by wearing the droplet size concentration of reactants and surfactants nature we can achieve the controlled size IONPs. Darbandi et. al. [87], have synthesized uniform sized iron oxide nanoparticles with spinal structure of diameter 3, 6 and 9 nm at room temperature using microemulsion route. When direct particle take place then capping agent can prevent the agglomeration effect. Okoli et. al. [88], have synthesized IONPs for the binding of protein and their separation using w/o (reversed) and o/w (direct) microemulsion. The potential of both approaches was investigated and compared to produce nano crystalline magnetic iron oxide having relatively higher surface area. The average surface of both 304 m^2g^{-1} for o/w and 147 m^2g^{-1} for w/o. The small size of nanoparticles is assigned from higher specific surface area observed in o/w (direct) microemulsion. The bare iron oxide nano particles, shows considerable interaction between protein and magnetic IONPs whereas, protein bound IONPs shows considerable reduction in rate of removal of clay particles. However, several washing process and stabilization treatment are required because of presence of surfactants so that it can be used in biomedical application. Since magnetic iron oxide nanoparticles-magnetite or maghemite-are produced from microemulsion systems and are superparamagnetic, nontoxic, and less prone to change when manufactured in a controlled environment, they are the subject of the current study. To increase their efficacy, they can also be custom-made to a particular size with more homogenous nanoparticles and surface specificity. Due to these unique qualities, microemulsion-prepared iron oxide nanoparticles have an advantage over other magnetic nanoparticles. In addition, these materials are being researched extensively for a variety of uses, including drug delivery, protein purification, water treatment, magnetic separation, and catalysis, among others [89,90]. The flow chart of microemulsion method is shown in **Figure 7.**

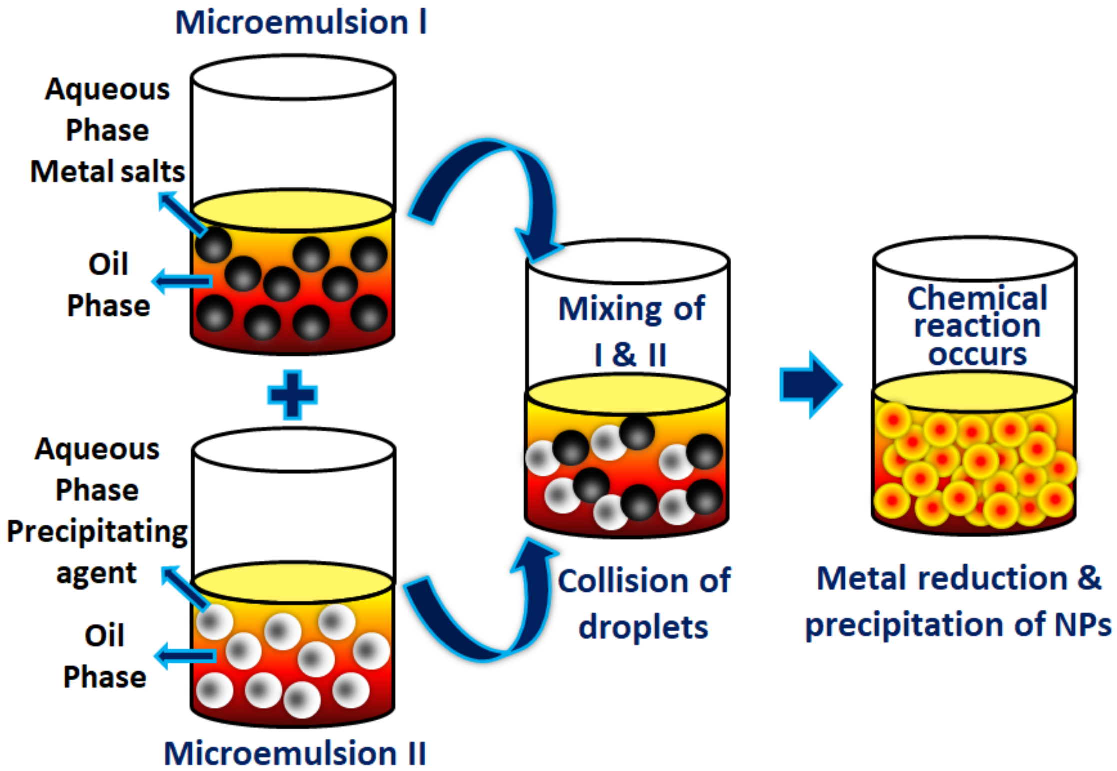

Fig. 7. Flow chart of Microemulsion method

2.7 Surface Coating of Nanoparticles

The surface of iron oxide has tendency to collect because of magnetic attraction between particles, high surface energy and high Vander Waals forces [91]. Highest density of native Fe ions is harmful to organisms from Fe decomposition [92]. The toxic effect of iron oxide when injected into the body can be avoided by coating a shell on the surface of synthesized iron oxide which adds on the properties like biocompatible, hydrophilic and functionalised material [93]. The proper surface coating allows the nanoparticles to directly target the localisation area without affecting the healthy tissues non-toxically and biocompatible. Though so much work has been done for the coating of nanoparticles which gives a perfect size distribution shape, surface and quality of magnetic particles but we have come across very few investigations and development. Surface coating of Nanoparticles play a vital role in determining the overall size of colloid and the bio-distribution of nanoparticles in body [94]. The coating on nanoparticles depends on the application aimed for particular response in biomedical application. In hypothermia proteins, enzymes or antibodies can be used to reach the localized area with the help of magnetic field [95].

In comparison to the organic solvent heating approach, the co-precipitation method is the most efficient way to synthesize iron oxide nanoparticles coated with biological molecules, such as lactobionic acid, gluconic acid, and polyacryl acid [96]. These types of nanoparticles have constricted size distribution and highly soluble in water. These have great capability in biomedical application because of biological molecules coating like liposome coating [97]. To stabilize the iron oxide nano particles high density coating is required. Particular stabilizer, surfactant or polymer is added during preparation to protect from aggregation of nanoparticles. There are various coatings of molecules / polymers for magnetic nanoparticles to stabilize ferrofluids like PEG(it improves biocompatibility by non-covalent immobilization on the surface and it improves blood circulation time) [98], PVP(stable colloidal solution and improves blood circulation time) [99], PVA (increase monodisperse particles and stops coagulation of particles) [100], Gelatin (it is natural polymer, biocompatible and used as a gelling agent) [101], phosphorylcholin (activate the coagulation and colloidal solution) [102] and Poly N-isopropylacrylamide (PNIPAM) (it separates the cells and help in drug delivery) [103].

Various coatings of material which protect the iron oxide from corrosion Like SiO_2, polymers, silver, gold and carbon. The main purpose of using SiO_2 is, it gives modification to colloid surface. It is stable and easy dispersed in organic and aqueous solution without surfactant. Incorporated with dye and drug and with Quantum dots and also compatible with many chemicals. Polymer, it enhances dispersibility in aqueous medium, for the functionalization it gives protective and biocompatible organic surface. Carbon is also used because it takes cells with both single nano particles and small nanoparticles cluster, which affect the evaluation of cytotoxicity. Nowadays silver and gold is mainly used because it protects the iron oxide nanoparticles from low pH corrosion. it also gives additional optical properties to the nanoparticles. The fundamental characteristics of magnetic nanoparticles allow for the variation of certain variables, such as surface chemistry and solubility size. These compounds all prevent corrosion in iron oxide cores.

3. Application of Iron Oxide Nanoparticles

The IONPs has been taken in account of biomedical application based on biocompatibility and toxicity. It also have strong magnetic properties that is why used in the biomedical application like drug delivery, magnetic hyperthermia and MRI etc [103]. The size of the particle produced, and its coating is the parameters which determine the biocompatibility and toxicity of NPs. The composite iron oxide nano particles should also have the high magnetisation so that it can move inside body by applying magnetic field and reach to targeted area [104]. The size, charge of magnetic particles and surface chemistry persuade bio-distribution of nanoparticles [105]. Magnetic iron oxide nanoparticles has attracted attention now a days as labelling material in life science and various other fields of scientific world [106]. Magnetic iron oxide nano particles are the primarily nanomaterials in biomedical application specially vivo and vitro. The main motivation of using Iron oxide NPs is owing to their excellent magnetic properties were firstly used for the biological purpose and then used in the medicines [107]. The size change and surface chemistry of the magnetic particles influence biodistribution of NPs. From past decades, the use of NPs has been increased because it plays vital role in diagnostics and modalities of treatment [108]. IONPs have a wide variety of applications like biomedical, healthcare, agriculture and food, construction, automotive, textiles and electronics.

3.1 Biomedical: IONPs have a special ability for biomedical application efficiency, including photo-thermal therapy, imaging, and diagnostics. Due to the many uses IONPs have in the biomedical area during the past 20 years, interest in their application has grown, particularly with regard to iron oxide-based materials (magnetite and maghemite). It has long been known that they are used as drug delivery and imaging contrast agent carriers in cancer treatments. This is a result of their unique performances, which stem from their nanoscale structure and magnetic characteristics. In order to achieve the necessary efficiency for the chosen application, regulated surface engineering is essential for the design of multi-functional IONPs. There has been extensive research on the use of IONPs as magnetic resonance imaging (MRI) agents for accurate and sensitive diagnostic instruments as well as for synergistic combinations with other imaging modalities [109,110]. IONPs have been applied to targeted drug/therapeutic agent administration, magnetic separation, magnetic field/light-induced hyperthermia, transfection of different viral/non-viral vectors and nucleic acids (called magnetofection), and diagnostic imaging as contrast agents. Uses for IONPs include magnetic hyperthermia, medication administration, MRI contrast agent, cellular treatment, cell separation and handling, purifying cell populations, and treating pain and disability. [107]**.**

3.2 Healthcare: IONPs are widely available, inexpensive, and essential to numerous biological and geological processes, they are commonly used. Humans also use them extensively for various purposes, in particular hemoglobin, long-lasting pigments, catalysts, healthcare, and drug-delivery. In healthcare humans are treated for various diseases and of the most common and prevalent is related with malignant tumors, because liver cancer is the third largest cause of cancer-related death globally. Prompt diagnosis and accurate lesion placement are essential for extending survival time. IONPs having a hydrodynamic diameter ranging from 50 to 150 nm may be used as particular MRI contrast agents to distinguish between normal liver tissue and malignant hepatocellular carcinoma (HCC). Kupffer cells are phagocytic macrophages found in the parenchyma of healthy livers. They have the ability to selectively absorb and retain IONPs in the lysosome in the form of clusters. However, it is discovered that Kupffer cells are lacking or absent in hepatic lesions, which reduces the uptake of IONPs by macrophages [111].

3.3 Agriculture and food: According to Dola et al. [112], iron-based nano-fertilizers are always advantageous for plants because they either provide nutrients or actively participate in the transfer or absorption of available minerals, hence accelerating plant growth. IONPs are being studied as potential agricultural agents such as fertilizers, growth enhancers, and insecticides. As a result, knowing the mechanisms underlying their impacts is critical. IONPS is a novel, effective, and time-tested solution made up of three essential nutrients for plant growth and development: nitrogen, phosphorus, and sulfur. The fertilizer gives plants a healthy start and assures an abundant yield due to the right nutritional balance and selection. According to the findings, the impacts of IONPs on plants could be either beneficial or negative, depending on the additive amounts. Low concentrations of IONPs appeared to be beneficial to plant growth, whereas high concentrations of IONPs appeared to be detrimental to plant growth in this investigation. Nano-based products (IONPs-fertilizers, pesticides) pyrite-nanoparticles are used for various plants sowing the seeds, larger leaf numbers, broad leaf morphology, enhanced breakdown of stored starch, increased biomass, nano-sensors, nano-food, encapsulation, food packing, nano-coatings [113]**.**

3.4 Environmental- Heavy metals and organic contaminations in soil ecosystems have increased due to the fast expansion of industrialization and these contaminations pose serious health hazards to humans, including the potential for liver and kidney damage as well as cancer [114]. It is becoming more and more important to comprehend the ecological safety and environmental characteristics of IONPs as interest in their application grows. IONPs would initially interact with organic contaminants and inorganic heavy metal ions in polluted soil, both of which could change the IONPs' surface. Finally, the stability, bioavailability, and toxicity of IONPs may be influenced by their environmental behaviors and changes. While some research has noted potential environmental risks associated with IONP uses it is rare to discuss the evolution of IONP material classification and its implications for adsorption, stability, and risk [115]. According to this study, iron (II) chloride salt totally reacts, transforming into IONPs and producing NaCl as a beneficial byproduct. The effect categories were able to be reduced by presuming that neither iron nor chloride ions were released into the environment [116]. Thus, we observe that IONPs has a wide impact on the environment. The potential and problems related to using IONPs for water treatment was also examined [117].

3.5 Energy- Better engineering of NPs might be possible with an understanding of the mechanisms driving the transport of heat created by the NPs, particularly for biomedical purposes. In fact, it might be able to more cleverly utilize the energy flux to trigger the release of the target molecule or to activate the immobilized enzyme. Since IONPs have a higher level of biocompatibility than other magnetic nanoparticles (NPs) such those based on cobalt and nickel oxide, they are preferred in this situation. Thus, our primary attention will be on the IONPs functionalized with a commonly utilized anchoring group. In fact, a successful way of covalently connecting, for example, enzymes to IONPs are to use species like amino-propyl-triethoxysilane (APTES) as anchoring groups. APTES may build bridges between hydroxylated oxide surfaces and (bio)molecules and even with live systems [118]. IONPs have recently been created using a variety of techniques and used in numerous applications, including energy storage and others. In order to demonstrate how the charge and discharge capacities of Li-ion batteries are morphology dependent, Wu et al. [119] manufactured -Fe_2O_3 nanorods and employed them as an anode material. The hydrothermal synthesis of rhombohedral - Fe_2O_3 by Hwang et al. [120] revealed a 1500 mAh/g discharge capacity. Wu et al. [121] examined the most recent advancements in the production of IONPs using the currently used techniques, outlining their advantages and disadvantages.

3.6 Defense & aerospace - The mineralization of chemical weapon agent simulants by oxygen and peroxide is mediated by nano-crystalline metal oxides, which are semiconductors activated by light. Metal oxide nano-crystals have also been used to destructively adsorb warfare chemicals. The nanoparticles, which can be employed on both acids and bases, quickly bind to dangerous chemical molecules and convert them to safer by-products. IONPs is used in composite propellant to speed up combustion, hence it has a significant impact on the aircraft industry as well [122]. Diverse polymorphic forms of iron oxide nanoparticles have garnered significant attention in a range of applications due to their unique electrical, optical, and magnetic properties. IONPs are particularly crucial to the aerospace sector because they accelerate the rate at which composite propellants burn. Numerous research are currently being undertaken on the catalytic effect of nano-Fe_2O_3 on the thermal decomposition of ammonium perchlorate (NH_4ClO_4 or AP) are being conducted since the combustion mechanism of propellants is heavily dependent on this process [123].

3.7 Construction - Experimental research on the structural build-up of cementitious paste containing nano-Fe_3O_4 in time-varying magnetic fields was conducted using the small amplitude oscillatory shear (SAOS) technique. To the cementitious paste, various magnetic field modes were applied, such as constant, linearly changed and sudden changed. The findings showed that the cementitious paste's structural build-up was influenced by both the strength of the magnetic field and the length of the magnetization process. Constant magnetic fields improved the liquid-like behavior during the first few minutes; iron oxide pigments, nano-coatings, nanocomposites, nanoscale sensors, and smart materials then improved the solid-like feature [124].

3.8 Automotive - Additives for fuel, batteries, nano-coatings, catalysis, and smart materials. Adhesively bonded joints are paving the way for light weighting in automotive applications because they spread the load over large areas, minimize the need for drilling holes, associated stress concentrations, and delamination, and can combine heterogeneous material substrates. Many people in the automotive industry are interested in learning more about IONPs with exceptional mechanical and tribological properties. It is essential to look into the viability of a workable strategy for recycling these materials given the high potential of IONPs in the automotive industry and the requirement to recycle materials to lessen their environmental impact. [125,126].

3.9 Textiles- Applying IONPs to cotton fibers resulted in the production of a magnetic textile. IONPs have been produced using reverse co-precipitation. Due to their distinct physicochemical characteristics, affordability, and low toxicity, IONPs have attracted a lot of attention lately. IONPS are having efficient magnetic properties, and they have been thoroughly explored and used in the production of nanowires, textiles, polymers, nanofiber coatings, tissue restoration, and the remediation of contaminated industrial sites [127]. Scientists who seek to apply IONPs in industrial settings are still drawn to them. Attention has been drawn to these materials' controllable dimensions of up to 100 nm as well as their capabilities in chemical, magnetic, electrical, and mechanical fields. Due to their excellent recyclability, separation efficiency, thermal resilience, and durability they also make textile textiles recyclable, smart materials, nanofibers, coatings, and sensors. [128]. A substantial amount of IONPs were deposited on textiles to enable their straight forward magnetic separation from vast volumes of solutions; if required, the magnetic sensitivity of the modified textiles can be simply augmented by incorporating a piece of magnetic iron wire [129].

3.10 Electronics- Due to their distinctive characteristics, metallic oxides like iron oxide (magnetite, maghemite, and hematite) have seen a boost in interest in their nanoscale transition in recent times including photoluminescence, optical, magnetic, electrical, and opto-electronic properties. These properties find application in a variety of fields, opto-electronics, inorganic pigments, printable solar cells, progress in gas sensors, spintronics, information storage and ferrofluid technology. We provide a thorough analysis of various magnetite surface terminations, Fe_3O_4 (111), an important iron oxide with numerous uses in electronics and spintronics. IONPs alter the values of electrical conductivity, including DC and AC electric conductivity. To ascertain these changes, the electrical conductivity of composites was examined using different theories using nanowires, carbon nanotubes, nanoscale memory, printed electronics, and quantum dots We also observed that when the charge carriers are provided by Co and Ni ions, which replaces Fe ions. It was found that there are more charge carriers and more photo-excited electrons that participate in electronic conduction are produced when light photons decrease. This results in a decrease in resistivity and an increase in electrical conductivity, confirming that IONPs doped with metals are a promising option for enhanced performance. [130].

3.11 Biosensing and Bioseparation: The principle on which it works is the magnetic field generated by Magnetic particles which modify the magnetic fields of sensor which changes the electrical current or resistance in the sensor [131]. It has many other advantages as compared to other technique like long term stability, gives high sensitivity and biomaker detection etc [132]. Biosensing has application in environmental control, clinical diagnosis and food industry [133]. Magnetic separation in IONPs, inexpensive and efficient in nature, it is better solution to problems encountered by conventional tedious and time-consuming biomedical separation methods which includes centrifugation and filtration [134,135]. By applying magnetic field, the complexes can be easily attracted from the immaculate mixture thus it is convenient and time saving approach for bio-separation. It is also used to purify the complementary DNAs with the help of IONPs comportment incapacitated single isolated DNAs [136]. This technique has variously applied in the separation process and purification of cells [137], bacterial [138], proteins [139] as well as virus.

3.12 Drug Delivery and Hyperthermia: During cancer treatment, compounds with excessive cytotoxicity activities are delivered in tumor cell to damage the cell. This method is beneficial because it does not affect the healthy tissue but damage the unhealthy tissue [140]. The molecules of targeted drug delivery of IONPs improve the specific drug and reduce the side effect [141]. The IONPs are injected in body through blood stream and guided by applying magnetic field externally [142]. It is medical treatment that kills the tumor cells by heating tumor at temperature between 42-46°C [143,144]. There are two types of hyperthermia. Firstly, full body hyperthermia which includes heating of whole body to treat cancer cells spread across the body. Secondly, localized hyperthermia involves heating a particular area of tumor. Techniques available for deduction of hyperthermia are, infrared radiation, radiofrequency, magnetically excitable thermoseeds and microwaves [145]. In hyperthermia treatment, IONPs based ferromagnetic or superparamagnetic nanoparticles are involved to treat tumor tissue by heating effect produced by alternating current (AC) magnetic field [149]. Main advantages of using magnetic nanoparticles are (1) to assure the protection of hyperthermia, only tumor cells are affected by heating [147]. (2) to provide particularity in hyperthermia, destroys only unhealthy tissue in the body [148]. (3) increase the efficiency of hyperthermia, the NPs used are of very small size in nm so that NPs can be easily injected in body and can be detected by magnetic field [149].

4.0 Conclusions and Future Perspectives

- In order to produce trustworthy superparamagnetic nanoparticles having ideal size, surface charge, shape, biocompatibility, colloidal stability in biological environment, and saturation magnetization, this review article has been written.

- The current review on the synthesis of iron oxide magnetic nanoparticles is primarily focused on developing new techniques or optimizing the existing ones. Particularly in the last ten years, significant advancements have been made for the development of magnetic nanoparticles, which can have a wide variety of compositions and tunable sizes.

- Numerous chemical synthetic techniques, including co-precipitation, sol-gel method, liquid phase method, hydrothermal and solvothermal, sonolysis, and microemulsion, have been used to create various types of mono-disperse spherical nano particles with their desired sizes and compositions.

- Designing magnetic nanoparticles with efficient surface coatings that can offer top performance for in vivo and in vitro biological applications is a significant problem for all approaches. Toxicity, safety and scale-up of nano-particle production techniques are additional difficulties.

- It has been accomplished to change hydrophobic nature with respect to water and biocompatible using coated monolayer polymer with organic ligand. In addition, iron nanoparticles coated with other biomolecules have improved their biocompatibility, winning approval from regulatory bodies like the food and drug administration.

- As a result, the usage of iron nanoparticles is widespread in many different industries, including MRI, cancer treatments, target-specific medication delivery, in vitro diagnostics, and many more. The field will advance with the adoption of more efficient ways to increase our thoughtful of NP toxicity.

- The difficulty is in practical application of NP probes, along with in biocompatibility, toxicity, and in vitro and in vivo targeting effectiveness. That being said, we are confident that surface functionalization and modification of magnetic iron oxide nanoparticles (NPs) to introduce novel functionality will continue to attract attention.

- Additionally, developed active site multifunctional magnetic iron oxide composite nanoparticle systems have promise for a range of applications, including catalysts, bio-separation, magnetic recording, bio-detection, etc.

- Future research in this field must concentrate on determining the toxicity and biodegradability of functionalized or surface-functionalized iron oxide nanoparticles (NPs) and manufacturing them using green chemistry to minimize environmental damage. A successful development in this field would boost the expansion of numerous scientific studies or commercial applications while also enhancing the standard of living for the populace.

Conflicts of Interest

The authors declare that they have no conflict of interest.

Acknowledgments

Madhavi is thankful to SGT University for the financial support in the form of fellowship. MK thankful to Shree Guru Gobind Singh Tricentenary University Gurugram and JRA gratefully acknowledge the support, co-operation and financial assistance from Yonsei University, South Korea.

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List of Abbreviations

Deoxyribonucleic Acid	:	DNAs
Iron Oxide Nanoparticles	:	IONPs
Magnetic Nanoparticles	:	MNPs
Magnetic Resonance Imaging	:	MRI
Poly Ethylene Glycol	:	PEG
Poly N-Isopropylacrylamide	:	PNIPAM
Poly Vinyl Pyrrolidone	:	PVP
Reduced Graphene Oxide	:	RGO
Sodium Benzene Sulfonate	:	Na DDBS
Solid Lipid Nanoparticles	:	SLNs
Super Paramagnetic Iron Oxide Nanoparticles	:	SPIONs
Tri Ethyl Ortho Soilicate	:	TEOS

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