A review of synthesis roots of iron nanoparticles

T Anjam*, N U H Altaf, W Ahmad, M A Sajid, M A Munir and M Aslam

Department of Physics, University of Agriculture Faisalabad, 38040 Faisalabad, Pakistan.

*E-mail: tauseefanjam8@gmail.com

Abstract. Nanoscience is a standout amongst the most significant innovative work action in the outskirt of current science. From most recent two decades the nanotechnology and nanoscience have increased more significance in the field of research because of the enormous scale applications. The present work audits crafted by various researchers for the planning of iron nanoparticles by various procedures. Iron is a standout amongst the richest and generally utilized components on earth. Iron nanoparticles have a tremendous potential for various applications including magnetic liquids, magnetic resonance imaging (MRI) differentiate operators, catalyst for carbon nanotube development, nickel-iron batteries, and impetuses and sorbents for natural remediation. The preparation of iron nanoparticles by different methods, pulsed plasma, chemical reduction, chemical vapour condensation, one-step reduction, Green synthesis and Biosynthesis, its advantage over other processes are described. In this review article attempt has been made to compare each process by categorizing them specifically.

1. Introduction

Porous In the recent year, Nano science and technology are most important developing area of research. Nanotechnology implies the formation of nanoparticles in shape, size, controlled diversity, their potential and chemical composition uses for human benefits [1]. Today, nanotechnology is a developing area of investigation on planet. In this field the research explosion is because nanoparticles have remarkably better distinctiveness than their microscope counterpart [2]. The properties of materials change radically in their nano-form. In magnetic recording media (e.g. video tape, audio, data storage devices, magnetic fluid, and recording discs) the magnetic nanoparticles attract much attention as compared to other material due to their unique property. The various applications of magnetic nanoparticles such as bio-separation, targeted drug delivery, NMR imaging and cancer hyperthermia are uses in catalysis and other industries. To fabrication of electronic devices at nanoscale the application of magnetic nanoparticles is also used [3].

In nanoscale natural advance the innovation of iron nanoparticles is one of the first-generation of nanoparticles. In most recent couple of year, to deliver iron nanoparticles the various engineered strategies have been created, nanoparticles surface properties also adjust and improve the production for field transference and responses. Iron is plentiful and extensively used elements. Iron nanoparticles have potential for various application as well as catalysts for carbon nanotube configuration, nickel-iron batteries, sorbents for environmental remediation and magnetic fluid [4].

In the field of medicine, it also plays a vital role. For example, iron NPs are significant for absorbing material and waste water treatment. It also effectively controls the allotment of medicine powder which can targeted achieve liberation of chemotherapy drugs. It is important for absorbing material and waste
water treatment [5]. It has high magnetization at room temperature as compared to another element. It has soft magnetic material because of cubic crystalline structure [6].

Due to their specific activity and large surface area in the field of magnetic material synthesis of iron NPs is one of the most attractive challenge. For preparation of magnetic nanoparticles, the common methods are one step reduction [5], Pulsed plasma [6], Chemical reduction [3], Biosynthesis [1], ball milling [7], green synthesis for green tea [8], microwave plasma [9] and chemical vapour deposition [10]. All these various methods take into account their environmental safety sufficiency, cost-effectiveness, simplicity and occasion for controlling the symphony and properties of iron NPs. The different methods are discussed for the synthesis of iron nanoparticles.

The aim of the study of pulsed plasma in liquid method is to synthesis the pure iron NPs. During the synthesis of iron NPs through pulsed plasma in liquid a number of factors medium applied voltages, temperature, pulse duration, applied liquid condition and medium type are affected by it. For size control and purity of nanoparticles are using the water-toluene. There are four different samples prepared from iron electrode submerge in liquid. The morphology, structure and magnetic properties were investigated obtained from these samples [6].

For the synthesis of iron nanoparticles in aqueous medium the different method has been reported. In this technique utilize the ascorbic corrosive for decreasing the Fe(acac)$_3$. The room temperature and saturation magnetization of iron is very high. The curie temperature of iron is also high, and this application is most important. It is simple, basic and reproducible method to incorporate iron nanoparticles. Chemically synthesize of iron nanoparticles using the ascorbic acid to reduce the Fe(acac)$_3$ in controlled condition. In the nonexistence of water yields this reaction were carried out. Iron nanoparticles were synthesized in the relative study [3]. The chemical vapour condensation is another method to synthesized iron nanoparticles by using the Fe(CO)$_5$ as a precursor. The effect of annealing process and processing parameter on the size and microstructure of iron nanoparticles were investigated [10]. The one step reduction method is also using to synthesize the iron nanoparticles [5]. Iron nanoparticles were removed from green tea leaves [8]. Because of their large quantity and low caste iron-based nanoparticles are also used in industry [11]. Iron nanoparticles are utilized to expel poisons from soil and water in nature and from modern waste streams [12]. Iron nanoparticles are utilized for a wide scope of uses including magnetic recording, media [13], ferrofluids [14], attractive cell separation [15], MRI differentiate agents [16], natural remediation [17], energy storage materials [18], bioimaging system [19] and genetic engineering [20].

2. Methods for the synthesis of iron nanoparticles

2.1. Synthesis of iron nanoparticles through pulsed plasma technique

In the year of 2014, Kelgenbaeve [6] studied that by using the method of pulsed plasma in liquid the iron nanoparticles were synthesized with 10 nm sizes. By using arc discharge method in which water toluene interface used as medium the iron nanoparticles were prepared. In very high potential difference the space between the electrodes were breakdown due to appearance of pulsed plasma from inter-electrode. The diameter of iron electrodes was 5 mm and purity 99.9%. These electrodes were immersed in 20 mm deionized water. The duration of electrical current between pulsed plasma discharges was 10 microsecond and gap between them 1 mm. For continue the discharge process one electrode was connected with vibrator. A magnetic stirring bar was used with rotational speed 900 rpm.

Different samples were ready to study the effect of toluene content by using pulsed plasma. The water-toluene (1) 95-5 (2) 20-80 (3) 70-30 (4) 60-40% were used respectively. In each experiment the (100 V, 6 A) of AC electric power was used for 1 hour. By using the evaporation and centrifuge the black precipitate was separated from liquid. The different ratio of toluene-water in emulsion was control the size and purity of iron nanoparticles. As the toluene fraction decrease from 40 to 5% the size of nanoparticles decrease from 21 to 9.1 nm and also increase the purity from 48 to 98%. The toluene-water ratio 95-5% was found for more purity formation of iron nanoparticles. The lattice parameters of iron sample calculated from XRD. The XRD found (a = 0.2927) and previously reported (a (BCCFe) =
0.2866). The surface morphology of iron nanoparticles were examined through SEM as shown in figure 1 at three different resolution of ×700, ×6,500 and ×100,000 [6]. The spherical agglomerates were evidently display. Because of chemical bond and high surface energy of iron nanoparticles, tendency to form agglomerate was rather strong. The magnetic dipole force was contribute to inter particulate forces.

![Figure 1. FESEM Iron nanoparticles images at different magnifications: (a) ×700; (b) ×6,500; (c) ×100,000 [6].](image)

2.2. Iron nanoparticles via chemical reduction of Fe(acac)₃ by ascorbic acid

In the year 2014, Nene [3] studied that iron nanoparticles were chemically synthesized by using ascorbic acid for the reduction of Fe(acac)₃. For the synthesized of iron nanoparticles different method has been used but this method was used in first time. The chemical used in this method were diphenyl-ether, ascorbic acid, Fe(acac)₃ (purity 99%), dehydrated ethanol and ultrapure deionized water. The solution was prepared by 50mL Fe(acac)₃ and 30 mM diphenyl-ether mixed. The solution of Fe(acac)₃ and diphenyl-ether was kept under stirring in atmosphere of nitrogen gas at 70 °C.

The 0.025 M solution was prepared of dehydrated ethanol and ascorbic acid. This solution was added drop by drop at rate of 2 mL/min when temperature reached at 70 °C. After adding this reducing acid solution, the solution heated to 190 °C. Then the solution was cooled down to 25 °C. The solution was washed and filtered 4-5 time by chloroform. Dry powder obtained by using reducing Fe(acac)₃ chemical method. Figure 2 shows the TEM images of iron nanoparticles at different magnifications. The iron nanoparticles size was observed by TEM to be 7 ± 1 nm.

2.3. Synthesis of iron nanoparticles by chemical vapor condensation technique

In the year 2002, Choi [10] studied the iron nanoparticles were synthesis through chemical vapor condensation method. By using the pyrolysis of iron pentacarbonyl (Fe(CO)₅) the iron nanoparticles were synthesized. The influence of CVC parameter for size distribution and formation of nanoparticles was studied. The effect of annealing process and processing parameters on size and microstructure of iron nanoparticles were studies. The liquid iron pentacarbonyl was use as a precursor and helium or argon gas carried through heating bubble. The vaporization temperature was optimized at 150 °C. Flow gas was entered the precursor and pass through the tubular furnace to working chamber.

The precursor decayed in furnace and compress in particles. For conducting the experiment tubular furnace was heated at 400-1100 °C range. All particles were put on the surface of liquid nitrogen-cooled chiller. In working chamber, the powder was collected. The synthesized particles size was nearly equal to 6-25 nm in diameter. The structure of synthesized nanoparticles was core-shell type.
To examine the effect of corrosion temperature on particles size the different samples were synthesized at different temperature from 400-1100 °C by using helium or argon. Figure 3 shows the XRD pattern of samples formed at various disintegration temperatures. It also shows that through CVC iron NPs were also synthesized. With increasing the decomposition its width decreases and peaks intensity increase as shown in figure 3. In helium atmosphere there were no visible BCC peaks at decomposition temperature below 750 °C. BBC peaks was also not shown in X-ray diffraction pattern.

Figure 2. TEM images of iron nanoparticles [3].

Figure 3. XRD patterns of iron NPs with diameter of 19 nm (1), 12 nm (2), and 8 nm (3) for (a) After heating 220 °C, and (b) After heating 320 °C with nanoparticles size of 25 nm in argon atmosphere [10].

2.4. Synthesis of iron nanoparticles via One-Step reduction process
In the year of 2016 Ping [5] studied the iron nanoparticles were synthesized through the method of one-step reduction. The citric acid (C$_6$H$_8$O$_7$·H$_2$O) and ferric nitrate (Fe(NO$_3$)$_3$·9H$_2$O) were used. With sol-gel method the gel was prepared. The 41.6 grams citric acid and 80 grams ferric nitrate were mixed in 122 mL deionized water. This solution was stirred at 17.5 r/s. The stirrer was continued for 30 minutes. After stirring the gel was prepared and again heated to 60 °C. The solution of ammonia was added by drop wise at 60 °C. This solution was controlling the PH at 7.10. This process was continued for 4 hours. After four-hour heating temperature was increased to 95 °C and addition of 10 mL of olefinic acid. The product was washed with ethanol three times after stirring and dried, milled. In the presence of argon
gas atmosphere, the powder heated to 620 °C for 30 minutes. The black powders were obtained by one-step reduction method.

In one-step reduction method carbon source was used as carbon reduction that is provided by citric acid. For breaking of carbon in citric acid too much citric was added and also increase the temperature. Adding citric acid and increasing the temperature also results the formation of single matter C. This is used to condense the Fe$_2$O$_3$ to iron. Figure 4 shows the XRD pattern which showed all peaks was indexed to iron. The figure also shows the bcc structure from iron phase. The peaks or iron phases (110), (200), (211) at 2θ≈44°, 65°, 82°, respectively. This show the high crystallinity and iron nanoparticles.

![Figure 4. XRD patterns of iron nanoparticles [5].](image)

2.5 Green synthesis of iron nanoparticles using green tea leaves extract

Gottimukkala in the year 2017 [8] extract the iron nanoparticles from the green tea. The weight of 20 grams tea was mixed with 1000 ml of water in order to extract the tea. For getting the tea extraction, the solution was heated in water bath at temperature of 80 °C. The extract of tea was collected and filtered. This solution was assembled and stored. This solution was dried in a beaker for additional use. In sterilized flask the 1:1 proportion of green tea extract and 0.01M ferric Chloride was added for synthesized of iron NPs. The colour of solution was changed when adding of green tea extract to ferric chloride solution. After changing the coloured the solution centrifuge wash with deionized water.

For synthesis of different the morphology of iron NPs green tea were used as reducing agent. The 4000 species have been found in polyphenols and 1/3rd of total. This species was helps in reduction of salt precursor. The polyphenols consist of catechins and flavonoids. The reduction process was occurred due to the catechin. The standard potential of catechin was 0.57 V which decrease the Fe$^{3+}$ to Fe$^{0}$. The iron ordinary potential was -0.036 V. When precursor was added in solution formed partial bonds due to breaking the -OH bond. This partial bond was made bond with metal ion. The partial bond was also breaking to reduce metal ion into nanoparticles.

In order to study diameter and functional groups of nanoparticles, the size and morphology were characterized through SEM and FTIR analysis. FTIR analysis send synthesis of iron NPs was ready to evaluate and analysis the attach biomolecules. Vibration stretch was also showed the FTIR at 3452 cm$^{-1}$ for O-H and 1632 cm$^{-1}$ for C=C. The C-N and C-H were observed to be 1383 and 2926 cm$^{-1}$, respectively. It was compared to FTIR results it showed broad stretch C=C at 1635 cm$^{-1}$, cm$^{-1}$, C-O-C at 1020 cm$^{-1}$, c-n 1379 cm$^{-1}$ and O-H at 3419 cm$^{-1}$ which match with extract. Polyphenols was function as capping agent as well as reducing agent in green tea extract. Figure 5 shows the average diameter of iron nanoparticles were about 116 nm found from SEM images. While comparing the all different methods for the synthesis of iron NPs, different particle sizes have been observed as shown in the table 1.
Table 1. Summary of the iron nanoparticles synthesized by different methods.

| No. | Method | Study description | Procedure | Resulted iron nanoparticles size (nm) |
|-----|--------|------------------|-----------|--------------------------------------|
| 1   | Preparation of iron nanoparticles via pulsed plasma technique | The pulsed plasma in liquid is the suitable and cheap method for synthesis of nanoparticles. For increasing the demand of preparation of well dispersed nanostructure single-pulse plasma method is the low-cost conventional technique. | By using arc discharge method in which water toluene interface used as medium the iron nanoparticles were prepared. | ≤10 nm |
| 2   | Preparation of Iron nanoparticles via chemical reduction method | It is a simple, reproducible and profitable method to make iron nanoparticles. | Nanoparticles of iron were chemically synthesized by reduction of Fe (acac)₃ using ascorbic acid in controlled condition. | 7±1 nm |
| 3   | Iron nanoparticles via chemical vapour condensation | Iron nanoparticles by chemical vapour condensation (CVC) by the pyrolysis of organometallic Fe (CO)₅ as precursor. The impact of handling parameters and strengthening process on the microstructure and size of Fe nanoparticles were examined. | By using the pyrolysis of iron pentacarbonyl (Fe (CO)₅) the iron nanoparticles were synthesized. | 6-25 nm |
| 4   | Synthesis of iron NPs via One-Step reduction technique | It is easy and simple method to synthesize the iron nanoparticles. Under moderate temperature iron nanoparticles were synthesized. Highly crystalline nanoparticles were obtained | To obtain high crystallinity and pure iron nanoparticle at appropriate temperature (620°C) is using excessive citric acid. The citric acid (C₆H₈O₇·H₂O) and ferric nitrate (Fe (NO₃)₃·9H₂O) were used. | 30 nm |
| 5   | Iron nanoparticles via green tea leaves extract | The advancement over physical and chemical method is providing by green synthesizing. It is environment friendly, easy scaled up and cost-effective method to synthesis the iron nanoparticles. No need to use toxic chemical, high energy, pressure and temperature. | By take 20 g tea in 1000ml of water the extract of tea was prepared. For getting the tea extract the solution was heated in water bath at temperature of 80°C. The extract of tea was collected and filtered. | 116 nm |
3. Conclusion
Nanoscience is a champion among the most noteworthy imaginative work activity in the edge of current science. From latest two decades the nanotechnology and nanoscience have expanded more centrality in the field of research on account of the colossal scale applications. The present work reviews created by different specialists for the arranging of iron nanoparticles by different methods. Iron is a titleholder among the most extravagant and for the most part used segments on earth. Iron nanoparticles have an enormous potential for different applications including attractive fluids, attractive reverberation imaging (MRI) separate administrators, impetus for carbon nanotube advancement, nickel-iron batteries, and impulses and sorbents for regular remediation. The arrangement of iron nanoparticles by various techniques, beat plasma, confection decrease, synthetic vapour build-up, one-advance decrease, Green combination and Biosynthesis, its bit of leeway over different procedures are portrayed. The resulted particles size was observed distinct through all different methods.

References
[1] Chandran M, Yuvaraj D, Christudhas L, Ramesh K V 2016 Biotechnol. Ind. J. 12, 112.
[2] Okoth O J K 2016 (Doctoral dissertation, University of Nairobi).
[3] Nene A G, Takahashi M, Somani P R 2016 World J. NanoSci. Eng., 6, 20.
[4] Huang K C, Ehrman S H 2007 Langmuir, 23, 1419-1426.
[5] Ping H, Lu K, Jun Y, Fan F, Kuaisme W, Jingding D, Dongxin L 2016 Rare Metal Mat. Eng., 45, 3112-3114.
[6] Kelgenbaeva Z, Omurzak E, Takebe S, Sulaimankulova S, Abdullaeva Z, Iwamoto C, Mashimo T 2014 J. nanopart. res., 16, 2603.
[7] Kerekes L, Hakl J, Meszaros S, Vad K, Gurin P, Kis-Varga M, Beke L 2002 Czech. j. phys., 52, A89-A92.
[8] Gottimukkala K S V, Harika R P, Zamare D 2017 J. Nanomedine Biotherapeutic Discov., 7, 151.
[9] Hoder T, Kudrle V, Fr gala Z, Janca J, David B 2005 In WDS 5, 300-305.
[10] Choi C J, Tolochko O, Kim B K 2002 Mater. Lett., 56, 289-294.
[11] Wang Ch, Xu L, Wang Q 2003 J. Nat. Gas. Chem. 12, 10–16.
[12] Winkelmann K, German H, Hodes C, Li J, Price M, Termini C, Thiele C 2011 J. Nano. Edu. 3, 62-81.
[13] Jorgensen F, The Complete Handbook of Magnetic Recording; McGraw-Hill: New York, 1995.
[14] Rosensweig R E 1966 AIAA J. 4, 1751.
[15] Molday R S, Mackenzie D 1982 J. Immunol. Methods 52, 353-367.
[16] Tiefenauer L X, Tsegirky A, Kuhne G, Andres R Y 1996 Magn. Reson. Imaging 14, 391 402.
[17] Elliott D W, Zhang W X 2001 Environ. Sci. Technol. 35, 4922-4926
[18] Zhang F, Braun G B, Shi Y, Zhang Y, Sun X, Reich N O, Zhao D, Stucky G, 2010 J. Am. Chem. Soc. 132, 2850
[19] Fredin L A, Li Z, Ratner M A, Lanagan M T, Marks T J, 2012 Adv. Mater. 24, 5946
[20] Shah B, Yin P T, Ghoshal S, Lee K B, 2013 Angew. Chem. 125, 6310