Size-Controlled Synthesis of Iron and Iron Oxide Nanoparticles by the Rapid Inductive Heating Method

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ABSTRACT: Inductive heating synthesis is an emerging technique with the potential to displace the hot-injection synthesis method to prepare colloidal particles very rapidly with a narrow size distribution, controlled size, and high crystallinity. In this work, the inductive heating synthesis is applied to produce a short-temperature jump to mimic conditions like the hot-injection method to prepare traditional iron and iron oxide nanoparticles (IONPs) in the 3–11 nm size range within various solvents, precursors, and reaction time conditions. Moreover, this inductive heating technique can be used under unique experimental conditions not available for hot-injection reactions. These conditions include the use of very high initial monomer concentrations. Considering benefits over conventional methods, the inductive heating technique has the potential to provide an industrial level scale-up synthesis. The magnetization of these particles is consistent with the magnetization of the particles from the literature.

INTRODUCTION

Colloidal synthesis is an important tool in the toolbox of chemists to develop new nanomaterials such as iron oxide nanoparticles (IONPs), gold nanoparticles, semiconducting materials, and so forth for the demands of today’s industries. Among various nanomaterials, magnetic IONPs have received significant attention because of their numerous advantages such as inexpensive method of synthesis, physical and chemical stability, biocompatibility, and low environmental impact of manufacturing, thereby offering many unique advantages over other materials.1–3 Out of eight different forms of IONPs discovered so far, hematite (α-Fe2O3), magnetite (Fe3O4), and maghemite (γ-Fe2O3) are very common and interesting due to their polymorphism property that involves temperature-induced phase transition.10 Moreover, magnetite and maghemite exhibit many unique features such as superparamagnetism, high coercivity, low Curie temperature, and high magnetic susceptibility.15,11 These properties make them outstanding candidates in multiterabit storage, catalysis, biosensors, targeted drug delivery, magnetic hyperthermia, high-sensitivity bimolecular magnetic resonance imaging, bioseparation, and thermal ablation.10,15 χ−Fe2O3 and Fe3O4 are synthesized mainly using the hot-injection (HI) method.16–17 This well-established method is used to synthesize uniformly sized colloidal IONPs in laboratory settings by giving scalable yield with precise size and shape control.16–21 However, the HI method is limited due to uneven nucleation during the rapid injection of the precursor during initial mixing, which limits heat and mass transport of the molecules.21,22 Additionally, this method strictly requires the use of a solid precursor and is difficult to scale up to large amounts of nanomaterials.21

To address the issues of the HI method, Sun et al. have shown highly uniform magnetite NPs through iron (Fe)−

acetylacetone decomposition by annealing at 320 °C in high-boiling octadecene. The authors have demonstrated as much as 40 g of monodisperse IONPs generated without any size-selection process in a single reaction. Furthermore, Hyeon et al. have reported the nonhydrolytic method of synthesis of monodisperse and highly crystalline γ-Fe2O3 ranging from 4–16 nm diameter in size.6 However, these synthetic protocols are complex and required a mixture of multiple solvents (octyl ether and oleic acid) and long heating time followed by refluxing. On the other hand, the inductive heating (IH) method for the synthesis of colloidal nanomaterials achieves high heating rates (100–200 °C/s), thus producing similar outcomes as expected for hot-injection methods.21,22 This methodology relies on a specialized IH reactor with steel balls placed inside where the reactor sits within an induction coil that rapidly heats up the steel balls producing a boiling solvent and decomposition of precursor molecules. The boiling point of the solvent can be used as built-in temperature control for achieving various final temperatures without the need for annealing and reflux. Since the glass reactor itself is not heated, the temperature of the apparatus can drop quickly following the heat up process, allowing the synthesis of very small nanomaterials or proto nanocrystals. Here, we have explored the use of IH for IONP synthesis proving the techniques’ ability to control size and size distribution. In addition, the use
of heated steel balls allows creating a reductive environment to reduce oxygen contamination for the preparation of oxygen-sensitive nanomaterials such as iron, as shown in this work.

From the synthesis point of view, an important consideration is to prepare nanoparticles (NPs) in shorter reaction times, avoiding aging, and annealing. The motivation for this work comes from the technological need for the rapid single-step synthesis of monodisperse and uniform IONPs. In this paper, we demonstrate a rapid, relatively safer, and single-step alternative approach to synthesize IONPs within seconds via the IH technique. The size, magnetic behavior, and crystallinity of the synthesized IONPs using different precursors and solvents at different reaction times are studied. The average size of the synthesized IONPs ranges from 3 to 11 nm in diameter. Importantly, the size of IONPs is dependent on the reaction time and type of the solvent used. Considering benefits over conventional methods, this method can be considered simple, safer, and has the potential to provide an industrial level scale-up synthesis.

RESULTS AND DISCUSSIONS

IONPs were synthesized at atmospheric pressure with various heating times, precursors, and solvents to explore how nanoparticles are formed during IH and determine the level of size and size distribution that can be easily achieved under the operating conditions of the inductive heater. Our goal was to focus on short-term heating effects to explore the impact of the heating rates on the nanoparticle formation. Due to the rapid heating time of the inductive heater, the solvent temperature quickly rises until it reaches the boiling point of the solvent. The final temperatures of the solution are determined by the boiling point of the solvent, which acts as a built-in temperature control during synthesis. Here, we utilize solvents such as 1-octadecane ($T_{bp} = 314\, ^\circ C$), oleylamine ($T_{bp} = 350\, ^\circ C$), and trioctylamine ($T_{bp} = 367\, ^\circ C$). In addition, we demonstrate the use of a high concentration of traditional hot-injection precursors such as Fe(CO)$_5$. The concentrations of Fe(CO)$_5$ are almost 44 times higher here than typically used in the literature for typical hot-injection methods.

The fast nucleation rate depletes the precursors from the solution resulting in a smaller average size of the NPs (but larger yield). When the reaction is performed in two steps by heating the reaction mixture for 7 s twice with a break between heating cycles, the size increased to 1.4 nm, as shown in Figure 1D, H. As the reaction time is increased, the crystallinity of the synthesized nanoparticles changed from amorphous to highly crystalline particles, as shown in Figure 2. The HRTEM images of IONPs (Figure 2A–C) indicate 032, 117, and 022 lattice planes of $\gamma$-Fe$_2$O$_3$ nanoparticles, respectively. These lattice planes are consistent with literature values.

**Figure 1.** Transmission electron microscopy (TEM) images of IONPs produced using 0.8 M Fe(CO)$_5$, and oleylamine at different reaction times (A) 5 s, (B) 7 s, (C) 10 s, and (D) 14 s (7 + 7 s). (E)–(H) are the particle size distribution plots, respectively.

**Figure 2.** High-resolution TEM (HRTEM) images of IONPs produced using 0.8 M Fe(CO)$_5$, and oleylamine at different reaction times (A) 7 s, (B) 10 s, and (C) 14 s (7 + 7 s), indicating 032, 117, and 022 lattice planes of $\gamma$-Fe$_2$O$_3$ nanoparticles, respectively.
supported by lattice spacing data from the crystallography open database (COD) as well as density functional theory (DFT) calculations as reported by Grau-Crespo et al. While comparing γ-Fe$_2$O$_3$ and α-Fe$_2$O$_3$ (hematite), diffraction patterns from the COD show that γ-Fe$_2$O$_3$ has characteristic peaks resulting from 2.95, 3.30, and 3.40 Å lattice spacing, whereas α-Fe$_2$O$_3$ does not. The HRTEM analysis yields several lattice spacing measurements of 2.95, 3.30, and 3.40 Å, indicating the material must be in the γ phase. It is also observed that with the increase in reaction time, IONPs changed from multidomain to a single domain. Additionally, the powder X-ray diffraction (PXRD) spectra of IONPs with heating 7 + 7 s confirm γ-Fe$_2$O$_3$ (Supporting Information, Figure S3A), which is in close agreement with previously reported results.

Next, the impact of the monomer concentration on the NP size and size distribution is investigated. The concentration of the monomer was increased from its initial 0.8 to 3.35 M and to 7.5 M that is about 10 times higher than typically used in hot-injection synthesis methods. The average size of IONPs almost doubled after a short period (5 s) of heating time. When the concentration of the precursor molecule is increased by almost four times (Figure 3C), then crystalline nano-

![Figure 3. TEM images of IONPs produced using 3.35 M Fe (CO)$_5$ and oleylamine at different reaction times (A) 5 s and (B) 10 s. (C) and (D) are the particle size distribution plots, respectively.](image)

precursor concentrations since a larger portion of the reaction mixture is held above the decomposition temperature of the precursors.

**Synthesis of IONP from Fe (CO)$_5$ in Trioctylamine.** A similar experiment was performed using the same precursor under identical conditions of concentration and reaction time but with the different solvent having a higher boiling point than that of oleylamine (Figure 4).

As shown in Figure 5, larger-sized NPs (5.0 ± 1.3 nm as opposed to 3.7 ± 0.6 nm) were formed using Fe(CO)$_5$ and trioctylamine vs oleylamine even in a shorter heating time (5 s). This increase in size is attributed to the higher boiling point of trioctylamine (bp = 367 °C) compared to oleylamine (bp = 350 °C). Most likely the trioctylamine lowered the decomposition rate of Fe(CO)$_5$, hence, larger nanoparticles were formed as compared to the use of oleylamine in a shorter heating time. The size increased as the heating time increased from 5 to 7 to 10 s. The seed growth method is also used in this experiment, and it resulted in a larger size of NPs (size 7.1 ± 1.5 nm, Figure 5D,H) as compared to the size of nanoparticles produced after heating only 7 s. Park et al. have reported ultra-large-scale synthesis of 22 nm-sized IONPs using trioctylamine. Thus, synthesized IONPs were prepared by mixing iron oleate complex, oleic acid, and trioctylamine by ramping 340 °C at 3.3 °C/min under reflux for 1 h. However, their approach requires longer reaction time and multiple precursors for synthesis. In contrast, the IH method used here produced IONPs using a single precursor and ultrashort reaction time. Additionally, particles also changed from amorphous to crystalline with an increase in reaction time, as shown in Figure 6. Figure 6A–C is the HRTEM image of corresponding NPs indicating 222, 02S, and 026 lattice planes of γ-Fe$_2$O$_3$ nanoparticles. In the corresponding PXRD spectra of Figure 6C, the diffraction peaks of 2θ corresponds to [012] and [013] planes, which indicate the pure phase of γ-Fe$_2$O$_3$ nanoparticles (Supporting Information, Figure S3B). These values are closely matched with the previously reported work.

**Synthesis of IONP from Fe (CO)$_5$ in 1-Octadecane.** The straight chain of hydrocarbon in 1-octadecane makes it weaker binding ligand as compared to oleylamine and trioctylamine. Experiment series was performed using the same precursor in 1-octadecane under identical conditions of concentration as before (Figure 7).
The data in Figure 7 show that the IONPs synthesized using 0.8 M Fe(CO)₅ and 1-octadecane are double in size than synthesized using Fe(CO)₅ and oleylamine (7.4 ± 2.5 nm as compared to 3.7 ± 0.6 nm), with the same concentration and the same heating time, i.e., 5 s. These particles are also larger in size as compared to those synthesized using trioctylamine as a solvent (7.4 ± 2.5 nm as compared to 5.0 ± 1.3 nm) under the identical conditions of concentration, i.e., 0.8 M and reaction time, i.e., 5 s. The size of nanoparticles increased with an increase in reaction time as discussed in previous sets of experiment that was carried out using oleylamine and trioctylamine. However, this set of experiments did not produce monodisperse IONPs as discussed in the literature, but iron NPs.³ Unni et al. also synthesized monodisperse magnetic IONPs by the extended LaMer thermal decomposition route in the absence of oxygen using iron oleate complex and 1-octadecane by mixing at 350 rpm at a controlled temperature of 350 °C for 5 h.³¹

The HRTEM analysis of these particles shows 011, 011, and 002 lattice planes of γ-Fe₂O₃ nanoparticles, respectively.

Figure 5. TEM images of IONPs produced using 0.8 M Fe(CO)₅ and trioctylamine at different reaction times (A) 5 s, (B) 7 s, (C) 10 s, and (D) 14 s (7 + 7 s). (E)–(H) are the particle size distribution plots, respectively.

Figure 6. HRTEM images of IONPs produced using 0.8 M Fe(CO)₅ and trioctylamine at different reaction times (A) 7 s, (B) 10 s, and (C) 14 s (7 + 7 s), indicating 222, 025, and 026 lattice planes of γ-Fe₂O₃ nanoparticles, respectively.

Figure 7. TEM images of IONPs produced using 0.8 M Fe(CO)₅ and 1-octadecane at different reaction times (A) 5 s, (B) 7, (C) 10 s, and (D) 14 s (7 + 7 s). (E)–(H) are the particle size distribution plots, respectively.

Figure 8. HRTEM images of iron NPs produced using 0.8 M Fe(CO)₅ and 1-octadecane at different reaction times (A) 7 s, (B) 10 s, and (C) 14 s (7 + 7 s), indicating 011, 011, and 002 lattice planes of Iron NPs, respectively.

Figure 9. As can be seen from Supporting Information Figure S4A,B, the diffraction peaks of 2θ corresponds to [011, 002] and [001] planes, which confirms the formation of pure iron NPs.³² Thus, synthesized iron NPs were heated in an atmospheric oven at 70 °C for a week. Interestingly, the PXRD of these NPs after 1 week also showed diffraction peaks of 2θ corresponding to [011, 002] and [001]
Figure 9. Magnetization vs magnetic field plot for IONPs produced using 0.8 M (A) Fe(CO)₅ and oleylamine (B); Fe(CO)₅ and trioctylamine; (C) Fe(CO)₅ and 1-octadecane at 5, 10, and 7 +7 s heating time.

Comparison of Magnetic Properties of IONPs. The magnetic properties of IONPs and iron NPs were studied using a superconducting quantum interference device (SQUID). Tadic et al. have reported that magnetic properties are directly dependent on size, distribution, shape, and orientation of IONPs.33–37 In their work, they reported that hematite with a size around 10 nm produced blocking temperature and particles with a size around 20 nm produced Morin transition.33 Furthermore, they have reported hematite saturation magnetization (Mₘ) around 1.93 emu/g.33 In another study, they concluded that shape anisotropy affected the coercivity of hematite NPs.36 They observed that ellipsoid three-dimensional (3D) superstructures of hematite are much higher than that for irregularly shaped hematite NPs.36 Similarly, physical orientation can be used to achieve either superparamagnetic or ferromagnetic maghemite NPs.38 Superparamagnetic properties in NPs are attributed to the random orientation of particles, whereas ferromagnetism is related to parallel orientation of particles.34,35,37 Figure 9 shows the magnetization (emu/g) vs magnetic field, H (Oe) graph for IONPs obtained using Fe(CO)₅ and different solvents at different reaction times, at room temperature (298 K) as well as the Fe NPs; data are summarized in Table S1 in the Supporting Information.

Figure 9A shows that IONPs with a size 3.7 ± 0.6 nm has almost zero magnetization. We have observed that with the increasing size from 3.7 ± 0.6 to 7.3 ± 1.4 nm, the shape of M–H loops for IONPs does not change appreciably, but their Mₘ increases from 0 to 20 emu/g. This value is close to Mₘ of 11 nm-sized γ-Fe₂O₃ synthesized via thermal decomposition of Fe(CO)₅ as reported in literature.38 However, it should be noted that the obtained Mₘ values are much smaller than those expected considering the bulk Mₘ values (74 emu) of γ-Fe₂O₃.39 This could be due to the presence of magnetically disordered atoms at the surface of the NPs, which is common in smaller magnetic NPs.40 As the particle size increases, the particles change from multidomain to a single domain that can be seen from HRTEM images discussed in Figures 2, 6, and 8 and the value of Mₘ also increases. Furthermore, a similar trend is observed in Figure 9B, but the Mₘ of iron NPs using Fe(CO)₅ and 1-octadecane (Figure 9C) increases from 5 to 90 emu/g with increasing size. The increase in Mₘ with an increase in size is attributed to a decrease in a surface spin in binding ligands oleylamine, trioctylamine, and 1-octadecane with the increase in particle size.31–33 This value is also smaller than bulk iron NPs (221 emu/g), but its data are consistent with the observation from the structural analysis that 1-octadecane synthesized samples are composed of iron. Those particles also showed smaller hysteresis at different reaction times (Supporting Information, Figure S6).

CONCLUSIONS

A new simple and facile approach has been developed for the preparation of IONPs and air-stable iron NPs by the rapid inductive heating method. The synthesis involved Fe(CO)₅ as a precursor, a clear advantage over the conventional HI method, using oleylamine, trioctylamine, and 1-octadecane as the solvents. These solvents played the role of binding ligand and reducing agents. The size of the synthesized NPs was dependent on the boiling point of the solvent and reaction time. As the reaction time and boiling point of the solvent are increased, the magnetization of NPs also increased to some extent. Also, this method can be used in the synthesis of a range of nanoparticles sizes (as small as 3 nm to as large as 11 nm), which is another key advantage over the traditional HI method. Interestingly, we have been successfully making stable iron NPs when 1-octadecane is used as a solvent. Overall, it is expected that the inductive heating method will result in further exploration of the topic due to faster, easier, and safer preparation methodology and could easily be scaled up to a gram scale.

MATERIALS AND METHODS

Chemicals. Oleylamine (70%), trioctylamine (98%), and 1-octadecane (tech. grade, 90%) were purchased from Sigma-Aldrich. All solvents were dried and degassed via boiling under vacuum before use. Iron pentacarbonyl (Fe(CO)₅) (>99.99%) was also purchased from Sigma-Aldrich and stored inside a nitrogen-filled glovebox. Methanol, ethanol, chloroform, acetone, andtoluene were purchased from Fischer Scientific (ACS grade).

Method of Synthesis. The synthesis method for IONPs was based on literature with some modifications. The setup for inductive heater and reactor with precursor mix is the same as that of previously reported work by Chikan et al. and Luo et al.21,22 Briefly, in this synthesis, iron pentacarbonyl is used as precursor, and oleylamine, trioctylamine, and 1-octadecane are used as solvents. All of the precursors and solvent were mixed inside nitrogen (N₂)–filled glovebox. In the N₂-filled glovebox, the stock solution was prepared by stirring the mixture until the precursor dissolved into the solvent resulting in a yellowish solution. Then, the reactor was filled with steel balls (25.92 g, Bearing-Quality E52100 Alloy Steel, Hardened Ball, 1/8” diameter) and transported to the glovebox. Next, 4 mL of 0.8 M precursor was transferred from the stock solution to the reactor. Following, the reactor was transported to the inductive heater and connected to tubing, which maintains an argon atmosphere throughout the reaction. Then, the reaction mixture was heated in a standard 10 kW inductive heater set at minimum power for 5, 7, 10, and 14 s to see the change in size, crystallinity, and magnetic behavior of synthesized IONPs.
using varying reaction times, solvents, and precursor molecules. The yellowish solution turns darker black with increasing reaction time, indicating the formation of IONPs.

The synthesized NPs were cooled to room temperature, then isolated by centrifuging using various solvents. IONPs synthesized using oleylamine were isolated by centrifuging with methanol (~20–25 mL) at 8000 rpm for 10 min followed by sonication. This process was repeated over three cycles. The colorless supernatant was discarded, and the precipitated NPs were then dispersed in small quantities of toluene (3–4 mL) for glovebox storage until further use. IONPs synthesized using trioctylamine were isolated by centrifuging with ethanol (~20–25 mL) at 8000 rpm for 10 min and repeated for a total of two cycles followed by sonication between each cycle. Then, 2–3 mL of oleylamine was added for ligand transfer, washed with methanol for two cycles, sonicated between cycles, and dispersed in small quantities of toluene (3–4 mL) for glovebox storage until further use. IONPs synthesized using 1-octadecane as the solvent were separated by centrifuging with hexane and acetone in a 1:4 ratio at 8000 rpm for 10 min followed by bath sonication. The NPs underwent three cycles of this process and then were dispersed in chloroform for glovebox storage. Representative steps of the reactor preparation, product formation, and purification are shown in Figure 10.

![Image](https://dx.doi.org/10.1021/acsomega.0c02793)

**Figure 10.** Inductive heating reactor filled with the precursor and the solvent (left), the same reactor after the reaction (middle), and magnetic IONPs after purification (right).

**Characterization of IONPs.** The size and surface morphology of IONPs were analyzed using a transmission electron microscope (TEM) FEI Tecnai G2 spirit Bio TWIN. The elemental composition of IONPs was measured by energy dispersive spectrum (EDS) using an energy dispersive X-ray analysis (EDAX) detector. The crystal structure of IONPs was analyzed by high-resolution transmission electron microscopy (HRTEM) in an FEI Tecnai F 20X transmission electron microscope at an electron acceleration voltage of 200 kV.

High-resolution transmission electron microscopy (HRTEM) was performed at the University of Kansas’s Microscopy and Analytical Imaging (MAI) facility with an FEI Tecnai F 20X transmission electron microscope at an electron acceleration voltage of 200 kV. The TEM samples were prepared by agitating the nanoparticle solution in an ultrasonic bath for 10 min. Five microliters of the sample was placed onto a copper mesh grid with a lacey carbon film. The wet grids are dried for several minutes prior to being examined under TEM. The particle size and morphology were examined by bright-field and dark-field scanning transmission electron microscopy (STEM). High-resolution images were captured using a standardized, normative electron dose and a constant defocus value from the carbon-coated surfaces. Results were analyzed using TEM Imaging and Analysis (Thermo Fischer Scientific Company, Waltham, MA). The boundary of each measured domain is indicated with red lines (presented in the Supporting Information). The lattice plane of each measurement is shown with red lines along the lattice. Measurements were made by the TIA software’s diffractogram and verified via calculating the lattice spacing from the Fourier transform of the region. The white labels beside each measured crystal lattice indicate the lattice spacings for the respective lattice. The acquired lattice spacings of each sample were then compared with reference data supported by the Crystallography Open Database to characterize the sample by chemical composition and space group identification. After characterization, Miller indices and spacegroups were included alongside the lattice spacing measurements.

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c02793.

HRTEM images of IONPs produced using 3.75 M Fe(CO)5 and oleylamine, EDS of IONPs prepared using Fe (CO)5 and oleylamine, PXRD of IONPs and iron NPs, table summarizing size and magnetization values, and hysteresis of iron NPs using 0.8 M Fe(CO)5 and 1-octadecane (PDF)

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**Notes**

The authors declare no competing financial interest.

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ABBREVIATIONS USED

NPs, nanoparticles; IONPs, iron oxide nanoparticles; TEM, transmission electron microscopy; HRTEM, high-resolution transmission electron microscopy; MAI, microscopy and analytical imaging; STEM, scanning transmission electron microscopy; EDS, energy dispersive spectrum; N, nitrogen; Fe(CO)₅, iron pentacarbonyl; PXRD, powder X-ray diffraction

REFERENCES

(1) Teja, A. S.; Koh, P.-Y. Synthesis, Properties, and Applications of Magnetic Iron Oxide Nanoparticles. Prog. Cryst. Growth Charact. Mater. 2009, 55, 22–45.
(2) Ali, A.; Zafar, H.; Zia, M.; ul Haq, I.; Phull, A. R.; Ali, J. S.; Hussain, A. Synthesis, Characterization, Applications, and Challenges of Iron Oxide Nanoparticles. Nanotechnol., Sci. Appl. 2016, 9, 49–67.
(3) Park, J.; An, K.; Hwang, Y.; Park, J.-G.; Noh, H.-J.; Kim, J.-Y.; Park, J.-H.; Hwang, N.-M.; Hyeon, T. Ultra-Large-Scale Syntheses of Monodisperse Nanocrystals. Nat. Mater. 2004, 3, 891–895.
(4) Marasini, R.; Pitchaimani, A.; Thanh Nguyen, T. D.; Comer, J.; Aryal, S. The Influence of Polyethylene Glycol Passivation on the Surface Plasmon Resonance Induced Photothermal Properties of Gold Nanorods. Nanoscale 2018, 10, 13684–13693.
(5) Vidal-Vidal, J.; Rivas, J.; López-Quintela, M. A. Synthesis of Monodisperse Maghemite Nanoparticles by the Microemulsion Method. Colloids Surf., A 2006, 288, 44–51.
(6) Hyeon, T.; Lee, S. S.; Park, J.; Chung, Y.; Na, H. B. Synthesis of Highly Crystalline and Monodisperse Maghemite Nanocrystallites without a Size-Selection Process. J. Am. Chem. Soc. 2001, 123, 12798–12801.
(7) Aslam, M.; Schultz, E. A.; Sun, T.; Meade, T.; Dravid, V. P. Synthesis of Amine-Stabilized Aqueous Colloidal Iron Oxide Nanocrystals. Cryst. Growth Des. 2007, 7, 471–475.
(8) Gupta, A. K.; Gupta, M. Synthesis and Surface Engineering of Iron Oxide Nanoparticles for Biomedical Applications. Biomaterials 2005, 26, 3995–4021.
(9) Peng, S.; Wang, C.; Xie, J.; Sun, S. Synthesis and Stabilization of Monodisperse Fe Nanoparticles. J. Am. Chem. Soc. 2006, 128, 10676–10677.
(10) Wu, W.; Wu, Z.; Yu, T.; Jiang, C.; Kim, W.-S. Recent Progress on Magnetic Iron Oxide Nanoparticles: Synthesis, Surface Functional Strategies and Biomedical Applications. Sci. Technol. Adv. Mater. 2015, 16, No. 023501.
(11) Lam, U. T.; Mammucari, R.; Suzuki, K.; Foster, N. R. Processing of Iron Oxide Nanoparticles by Supercritical Fluids. Ind. Eng. Chem. Res. 2008, 47, 599–614.
(12) Aslam, M.; Schultz, E. A.; Sun, T.; Meade, T.; Dravid, V. P. Synthesis of Amine-Stabilized Aqueous Colloidal Iron Oxide Nanoparticles. Cryst. Growth Des. 2007, 7, 471–475.
(13) Raj, K.; Moskowitz, B.; Caciari, R. Advances in Ferrofluid Technology. J. Magn. Magn. Mater. 1995, 149, 174–180.
(14) Weller, D.; Moser, A. Thermal Effect Limits in Ultrahigh-Density Magnetic Recording. IEEE Trans. Mag. 1999, 35, 4423–4439.
(15) Weiss, W.; Ranke, W. Surface Chemistry and Catalysis on Well-Defined Epitaxial Iron-Oxide Layers. Prog. Surf. Sci. 2002, 70, 1–151.
(16) De Berti, I. O. P.; Cagnoli, M.; Pecchi, G.; L Alessandrin, J.; Stewart, S.; Bengoa, F.; Marchetti, S. Alternative Low-Cost Approach to the Synthesis of Magnetic Iron Oxide Nanoparticles by Thermal Decomposition of Organic Precursors. Nanotechnology 2013, 24, No. 175601.
(17) Vidal-Vidal, J.; Rivas, J.; López-Quintela, M. A. Synthesis of Monodisperse Maghemite Nanoparticles by the Microemulsion Method. Colloids Surf., A 2006, 288, 44–51.
(18) Hyeon, T. Chemical Synthesis of Magnetic Nanoparticles. Chem. Commun. 2003, 8, 927–934.
(19) Murray, C. B.; Norris, D. J.; Bawendi, M. G. Synthesis and Characterization of Nearly Monodisperse CdE (E = Sulfur, Selenium, Tellurium) Semiconductor Nanocrystallites. J. Am. Chem. Soc. 1993, 115, 8706–8715.
(20) Lu, A.-H.; Salabas, E. L.; Schüth, F. Magnetic Nanoparticles: Synthesis, Protection, Functionalization, and Application. Angew. Chem., Int. Ed. 2007, 46, 1222–1244.
(21) Chikan, V.; McLaurin, E. J. Rapid Nanoparticle Synthesis by Magnetic and Microwave Heating. Nanomaterials 2016, 6, No. 85.
(22) Lu, M.; H.; Kebede, B. A.; McLaurin, E. J.; Chikan, V. Rapid Induction and Microwave Heat-Up Syntheses of CdSe Quantum Dots. ACS Omega 2018, 3, 5399–5405.
(23) Sun, S.; Zeng, H. Size-Controlled Synthesis of Magnetite Nanoparticles. J. Am. Chem. Soc. 2002, 124, 8204–8205.
(24) Hyeon, T.; Lee, S. S.; Park, J.; Chung, Y.; Na, H. B. Synthesis of Highly Crystalline and Monodisperse Maghemite Nanocrystallites without a Size-Selection Process. J. Am. Chem. Soc. 2001, 123, 12798–12801.
(25) Laurent, S.; Forge, D.; Port, M.; Roch, A.; Robic, C.; Vander Elst, L.; Muller, R. N. Magnetic Iron Oxide Nanoparticles: Synthesis, Stabilization, Vectorization, Physicochemical Characterizations, and Biological Applications. Chem. Rev. 2008, 108, 2064–2110.
(26) Geaves, C. A Powder Neutron Diffraction Investigation of Vacancy Ordering and Covalence in γ-Fe₂O₃. J. Solid State Chem. 1983, 49, 325–333.
(27) Grau-Crespo, R.; Al-Baitai, A. Y.; Saadounie, I.; De Leeuw, N.; H. Vacancy Ordering and Electronic Structure of γ - Fe₂O₃ (Maghemite): A Theoretical Investigation. J. Phys.: Condens. Matter 2010, 22, No. 255401.
(28) Finger, L. W.; Hazen, R. M. Crystal Structure and Isothermal Compression of Fe₂O₃, Cr₂O₃, and V₂O₃ to 50 kbars. J. Appl. Phys. 1980, 51, 5362–5367.
(29) Wu, W.; He, Q.; Jiang, C. Magnetic Iron Oxide Nanoparticles: Synthesis and Surface Functionalization Strategies. Nanoscale Res Lett 2008, 3, 397–415.
(30) Pecharmac, C.; Gonzalez-Carreno, T.; Iglesias, J. E. The Infrared Dielectric Properties of Maghemite, γ-Fe₂O₃, from Reflectance Measurement on Pressed Powders. Phys. Chem. Miner. 1995, 22, 21–29.
(31) Unni, M.; Uhl, A. M.; Saviwala, S.; Savitzky, B. H.; Dhavalikar, R.; Garraud, N.; Arnold, D. P.; Kourkoutis, L. F.; Andrew, J. S.; Rinaldi, C. Thermal Decomposition Synthesis of Iron Oxide Nanoparticles with Diminished Magnetic Dead Layer by Controlled Addition of Oxygen. ACS Nano 2017, 11, 2284–2303.
(32) Woodward, P. M.; Suard, E.; Karen, P. Structural Tuning of Charge, Orbital, and Spin Ordering in Double-Cell Perovskite Series between NdBaFe₂O₅ and HoBaFe₂O₅. J. Am. Chem. Soc. 2003, 125, 8889–8899.
(33) Tadic, M.; Panjan, M.; Tadic, B. V.; Lazovic, J.; Damnjanovic, V.; Kopanja, M.; Kopanja, L. Magnetic Properties of Hematite (α – Fe₂O₃) Nanoparticles Synthesized by Sol-Gel Synthesis Method: The Influence of Particle Size and Particle Size Distribution. Journal of Electrical Engineering 2019, 70, 71–76.
(34) Tadic, M.; Kralj, S.; Jagodic, M.; Hanzel, D.; Makovec, D. Magnetic Properties of Novel Superparamagnetic Iron Oxide Nanoclusters and Their Peculiarities under Annealing Treatment. Appl. Surf. Sci. 2014, 322, 255–264.
(35) Tadic, M.; Kralj, S.; Lalatone, Y.; Motte, L. Iron Oxide Nanoclusters Coated with Silica: Synthesis, Surface Effects and Magnetic Properties. Appl. Surf. Sci. 2019, 476, 641–646.
(36) Tadic, M.; Trpkov, D.; Kopanja, L.; Vojnovic, S.; Panjan, M. Hydrothermal Synthesis of Hematite (α-Fe₂O₃) Nanoparticle Forms: Synthesis Conditions, Structure, Particle Shape Analysis, Cytotoxicity and Magnetic Properties. J. Alloys Compd. 2019, 792, 599–609.
(37) Tadic, M.; Kralj, S.; Kopanja, L. Synthesis, Particle Shape Characterization, Magnetic Properties, and Surface Modification of Superparamagnetic Iron Oxide Nanoclusters. Mater. Charact. 2019, 148, 123–133.
(38) Hyeon, T.; Lee, S. S.; Park, J.; Chung, Y.; Na, H. B. Synthesis of Highly Crystalline and Monodisperse Maghemite Nanocrystals...
without a Size-Selection Process. J. Am. Chem. Soc. 2001, 123, 12798–12801.

(39) Nemati, Z.; Alonso, J.; Khurshid, H.; Phan, M. H.; Srikanth, H. Core/Shell Iron/Iron Oxide Nanoparticles: Are They Promising for Magnetic Hyperthermia? RSC Adv. 2016, 6, 38697–38702.

(40) Rath, C.; Mishra, N. C.; Anand, S.; Das, R. P.; Sabu, K. K.; Upadhyay, C.; Verma, H. C. Appearance of Superparamagnetism on Heating Nanosize Mn0.65Zn0.35Fe2O4. Appl. Phys. Lett. 2000, 76, 475–477.

(41) Berkowitz, A. E.; Kodama, R. H.; Makhlouf, S. A.; Parker, F. T.; Spada, F. E.; McNiff, E. J.; Foner, S. Anomalous Properties of Magnetic Nanoparticles. J. Magn. Magn. Mater. 1999, 196–197, 591–594.

(42) Berkowitz, A. E.; Takano, K. Exchange Anisotropy — a Review. J. Magn. Magn. Mater. 1999, 200, 552–570.

(43) Smolensky, E. D.; Park, H.-Y. E.; Zhou, Y.; Rolla, G. A.; Marjańska, M.; Botta, M.; Pierre, V. C. Scaling Laws at the Nano Size: The Effect of Particle Size and Shape on the Magnetism and Relaxivity of Iron Oxide Nanoparticle Contrast Agents. J. Mater. Chem. B 2013, 1, 2818–2828.

(44) Muthuswamy, E.; Iskandar, A. S.; Amador, M. M.; Kauzlarich, S. M. Facile Synthesis of Germanium Nanoparticles with Size Control: Microwave versus Conventional Heating. Chem. Mater. 2013, 25, 1416–1422.