Production of magnetite nanoparticles from Ethiopian iron ore using solvent extraction and studying parameters that affect crystallite size

Kebede Gamo Sebehanie, Alberto Velázquez del Rosario, Abubeker Yimam Ali and Olu Emmanuel Femi

Abstract

Crystallite size is one of the fundamental factors controlling the magnetic property of magnetic nanoparticles. In this study, magnetite nanoparticles were produced from Ethiopian iron ore and the effect of solvent extraction parameters on the crystallite size were investigated by considering classical nucleation theory. First, the iron ore was crushed and unwanted gangues were discarded, then followed by further grinding to get a black powder. Finally, the magnetite nanoparticles were produced using solvent extraction and co-precipitation method. For the solvent extraction, tri-butyl phosphate (TBP) and tri-octyl phosphine oxide (TOPO) were used as an extractant, while 2-ethyl-hexanol, and heptanol were used as a modifier and diluent, respectively. The synthesized nanoparticles was characterized using XRD, SEM (EDS), TGA, and FTIR. The results show that leaching temperature and modifier concentration have a significant effect on the crystallite size of the nanoparticles. Comparison of the crystallite size using the Scherrer equation reveals the mean crystallite size of the sample extracted using TBP is smaller than the sample extracted using TOPO.

1. Introduction

Considerable research has been done on magnetite nanoparticles (MNPs) due to versatile applications in the modern world such as wastewater treatment, as a catalyst, magnetic recording media, ferrofluid, gas sensors, and other applications [1]. In recent times, magnetite nanoparticles are becoming very important in the biomedical field, with applications in targeted drug delivery [2], magnetic resonance imaging [3, 4], biological cell separation [5], and cancer therapy (hyperthermia) [6].

The use of magnetic nanoparticles for a specific application requires, the preparation and design of the particles to meet the desired purpose, since the method of synthesis influences the physical and chemical characteristics of the nanoparticles like crystallite size, shape, size distribution, aggregation, morphology, magnetic properties, stability in the air and dispersibility [7].

Crystallite size is one of the fundamental factors controlling the magnetic properties of magnetite nanoparticles [8, 9]. In crystal growth, which indirectly determines the crystallite size, many factors control the nucleation and growth activity. The presence of impurities considerably influences the nucleation behavior of a solute in the solution. Impurity effects on crystal growth are dependent on both the impurity concentration and solution supersaturation. Some impurities can entirely suppress the nucleation; some may improve nucleation, while others may apply a highly selective effort [10]. For example, ammonium sulfate [11] and potassium sulfate [12] in the presence of Cr(III) impurity and potassium dihydrogen phosphate in the presence of Fe(III) or Al(III) [13] cannot grow below a critical supersaturation.

© 2020 The Author(s). Published by IOP Publishing Ltd
To date, many methods have been developed for the synthesis of magnetite nanoparticles, including coprecipitation \cite{14–17}, sol–gel \cite{18, 19}, thermal decomposition \cite{20}, and hydrothermal synthesis \cite{21, 22} to control the properties of the resulting nanoparticle despite it is challenging.

Among the above alternatives, coprecipitation is the most widely used method by many researchers. However, the technique has some limitations, such as (1) difficulty in controlling oxidation (so that nitrogen gas is used) and pH \cite{23, 24} (2) problems in particle size distribution. Also, chemical reagents have been used in many reports as the starting material for the production of MNPs. This may be difficult for some applications in terms of feasibility, such as wastewater treatment or desalination plants, which requires substantial amount of magnetite nanoparticles.

To overcome the problems mentioned above, especially for mass production, a low-cost approach for the synthesis of MNPs is desirable. The use of iron ore as the primary source of MNPs could be a viable approach, although limited efforts have been devoted so far.

Rivas-Sanchez et al \cite{25} reported that naturally occurring magnetite nanoparticles are non-homogenous with sizes at the micro and nano level. Investigation of the magnetic properties of these naturally occurring MNPs reveals that they are ferromagnetic and paramagnetic. Likewise, Cid et al \cite{26} reported the production of different types of iron oxide nanomaterials, including MNPs using mechanical milling from naturally occurring iron ore. In their work, natural magnetite powder was milled thoroughly in a planetary ball mill. According to SEM images, a surfactant application was found to significantly reduce the particle size, with a particle size of around 100 nm. Darezereshki et al \cite{27} also develop magnetite nanoparticles from iron ore tailing using the reduction–precipitation method. Pregnant leach solution (PLS) of the iron ore tailings was carried out using H\textsubscript{2}SO\textsubscript{4} (5 M)/NaCl (2 M) reagents. The result showed an average particle size of 19 ± 3 nm. Nouioua and Barkat \cite{28} have also studied the solvent extraction of iron (III) from the hydrochloric acid leach liquor of iron ore from tributyl phosphate (TBP) in chloroform. It was found that the extraction of metal ions increased with increasing HCl, KCl, and TBP concentrations. Mao \cite{29} investigated the solvent extraction of iron (III) from acidic chloride solutions by decanol in kerosene. It was found that the extraction of iron (III) increased with increasing HCl concentration in the aqueous phase and alcoholic solvent, decanol was suitable for the extraction of iron (III) in acidic chloride solutions. Mao and Liu \cite{30} showed that TOPO could be used as a potential extractant for the separation of titanium(IV) and iron(III) from titania chloride liquors.

Also, Wei et al \cite{31} prepared ferric iron from acid mine drainage (AMD) by an oxidation-selective precipitation technique under an inert atmosphere and having a particle size in the range of 10 to 15 nm. Cai et al \cite{32} studied the extraction of Fe(III) from HCl solutions using N, N, N’, N’-tetra-2 ethylhexyldiglycolamide (T2EHDGA) as an extractant. They reported that the interaction between the diluents and the extractant has a strong effect on the interfacial activity of T2EHDGA in different diluents and affects the extraction ability. Sakthivel et al \cite{33} in their work prepared magnetite powder using iron ore tailing. A fraction of the extracted FeCl\textsubscript{3} solution was changed into FeCl\textsubscript{2} via metallic iron formation using NaBH\textsubscript{4} as a reducing reagent. The magnetite particles synthesized in different ways showed varying degrees of magnetization behavior, credited to the change in their particle size caused by different precipitating reagents. It was also found that the particle size of the magnetite samples influences the magnetization behavior, and smaller particle size can be obtained by NH\textsubscript{3} treatment as compared to NaOH.

Furthermore, Suh et al \cite{34} extracted Fe from low-grade iron ore by solvent extraction and synthesized MNPs as a nano-adsorbent by coprecipitation. The impurities were removed from the low-grade iron-ore leachate by solvent extraction using tri-n-butyl phosphate (TBP).

Even though there have been some efforts on the extraction of iron(III) from acidic chloride solution from natural iron ore and iron tailing, they cannot be considered sufficient. Besides, minimal research has been made on the synthesis of magnetite nanoparticles by the solvent extraction method using iron ore as the starting material. Further investigation on the effects of leaching temperature and modifier concentration on crystallite size has not been reported.

In this work, preparation of magnetite nanoparticle from Ethiopian iron ore and the investigation of the effects of the leaching temperature, solvent type, and modifier concentration on crystallite size have been carried out.

2. Materials and methods

2.1. Raw material and reagents

The iron ore sample was obtained from the Oromia regional state Wollega zone, Ethiopia. Using EDS, the iron ore’s approximate chemical composition was found to be 15%–40.73% Fe, 0.16%–1% Mg, 3%–8% Si, 1%–3% Al. Chemicals used in the experiment were hydrochloric acid (HCl-35%), sodium hydroxide, sodium borohydride, and hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}–30%); For solvent extraction process, tri-butyl phosphate (TBP),
tri-octyl phosphine oxide (TOPO), 2-ethyl-hexanol, and heptanol were used. All chemicals were of analytical grade.

2.2. Experimental procedure

First, the iron ore was crushed and unwanted gangue was discarded, then it was further grinded to get a black powder. Next, the elemental composition of the ore was characterized using EDS, and the different phases present in the ore was characterized using XRD. The final step was to extract the iron from the ore using solvent extraction and synthesis of magnetite nanoparticles by coprecipitation method. The solvent extraction process consists of two main steps: extraction and stripping. Details of this process can be found elsewhere [34].

In this work, 60 g of iron ore powder and 90 ml of HCl were mixed in a beaker and stirred at different leaching temperature (60 °C, 80 °C, and 100 °C) by keeping the time of extraction at 3 h. The black leachate was then centrifuged at 3000rpm for 10 min so that the residue would be discarded, then, 80 ml of 30% H2O2 was added as an oxidant to 60 ml of the obtained supernatant. Fe3+ ions were extracted from the leachate by mixing 50 ml of an aqueous solution of the leachate and 50 ml of solvent extractant containing (34 ml of TOPO/TBP, 10 ml of 2-ethyl-hexanol, and 6 ml of heptanol) in a separating funnel. The mixture was then stirred for 10 min to maintain uniformity and then left for 10 min until the organic phase and the aqueous phase were separated.

The aqueous solution was centrifuged at 3000 rpm for 10 min to separate and remove the silica particles. The effect of modifier concentration (percent by volume), 2-ethyl-hexanol (30%, 40%, 50%) was investigated by keeping the other parameters constant. The stripping process was carried out by mixing 50 ml of distilled water with 25 ml of the iron-extractant complex, keeping the iron-extractant complex’s volume ratio to the aqueous solution at 1:2. The mixture was stirred vigorously for 20 min and then left for 10 min in a separatory funnel until the phase separation. Then, the aqueous phase was separately collected to obtain the iron solution.

To synthesize MNPs, aqueous coprecipitation of Fe3+ and Fe2+ at a ratio of 2:1 was carried out in the presence of a base (pH 9–12). Then, 18 ml of NaBH4 (0.033 wt.%) aqueous solution was added to 60 ml of Fe3+ aqueous solution corresponding to a one-third volume of 180 ml Fe3+ aqueous solution and stirred for 10 min. Finally, 200 ml of the iron aqueous solution containing 106 ml of Fe3+ aqueous solution, 70 ml of Fe2+ aqueous solution, and 24 ml of distilled water was gradually added to 200 ml of 2 M NaOH alkaline solution. The mixed solution was stirred at 40 °C for 10 min to react and produce the MNPs. The synthesized nanoparticles were washed three times with distilled water and collected by a bar magnet.

3. Results and discussion

The elemental composition and phases of the Ethiopian iron ore before extraction was characterized using EDS and XRD techniques. Table 1 shows the weight percentage composition of the ore obtained from EDS. From table 1, the average weight percentage composition of the ore was found to be 15%–40.73% Fe, 0.16%–1% Mg, 3%–8% Si, 1%–3% Al. The Iron ore under investigation is a low-grade Iron ore according to the weight percentage of 15%–40.73% of the Iron ore obtained by EDS. The XRD pattern of the ore was also analyzed with X’pert HighScore plus software (figure 1) and the result was in agreement with titanomagnetite (reference number ICCD-01-074-2034).

The elemental (weight percentage) composition of the synthesized magnetite nanoparticles from the Iron ore is shown in table 2. The main impurities (that hinders the formation of magnetite nanoparticles) like silicon

| Element | Weight % | Atomic % |
|---------|----------|----------|
| O       | 40.59    | 66.26    |
| Mg      | 0.34     | 0.36     |
| Al      | 2.97     | 2.88     |
| Si      | 7.38     | 6.86     |
| P       | 0.23     | 0.19     |
| K       | 0.23     | 0.15     |
| Ca      | 2.28     | 1.49     |
| Ti      | 4.11     | 2.24     |
| V       | 0.51     | 0.26     |
| Fe      | 40.73    | 19.05    |
| Zn      | 0.63     | 0.25     |
| Total   | 100.00   | 100      |
was significantly reduced from 7.38 (table 1) to 0.18 (table 2), but the weight percentage of iron increases from 15 to 61 (table 2).

Figure 2 shows the magnetite nanoparticles extracted directly from the ore and the standard (with reference number ICCD-01-080-0390). Except at around $2\theta = 35^\circ$, there were slight shifts with the peaks, which may be due to lattice strain; the pattern is very similar. The XRD pattern shows the nature of the synthesized material which is magnetite phase and the average crystalline size. In this study, MNPs were synthesized by varying parameters and the average crystallite size was the resulting nanoparticles was calculated using the Scherer formula and was found to be between 14 to 42 nm.

The FTIR spectra (figure 3) shows a distinct absorption band for different bonds. Below 1000 cm$^{-1}$ mostly there are 2 peaks in magnetite. The peaks at 441 and 557 cm$^{-1}$ are related to Fe–O bonds and are attributed to octahedral and tetrahedral sites of magnetite, respectively [35, 36]. The stretching and deformation vibrations of hydroxyl groups connected to nanoparticles’ surface produce absorption bands nearby 1630 cm$^{-1}$ and 3406 cm$^{-1}$ [37].
SEM analysis was used to investigate the morphology of the synthesized magnetite nanoparticles, as shown in figure 4. At different magnification, the magnetite nanoparticles are agglomerated. This is one of the main problems in synthesizing magnetite nanoparticles due to the high surface energy resulting from a large specific surface area (surface-to-volume ratio) [38]. Therefore, it was difficult to see the shape of the synthesized individual particles.

The effect of solvent types
In the process of solvent extraction, two types of organic solvent (TOPO and TBP) were used to compare their effect on the crystallite size, keeping other parameters constant. As can be seen from the XRD diffractogram (figure 5) of the two samples, there was some phase difference, which is probably due to the different extracting capability between the two solvents. Furthermore, the comparison of their crystallite size using the Scherrer equation gave the mean crystallite size of the sample extracted using TBP (10–18 nm), which is smaller than sample extracted using TOPO (26–42 nm). The difference in crystallite size is believed to be dependent on the amount of impurity present. This has a significant effect on the nucleation and growth process [39].

Figure 6 shows the XRD pattern of the magnetite nanoparticles prepared with and without using an organic solvent and the iron ore powder without further purification. The phases present were also compared. Results show that the phases are very different, and the result was as expected. The use of an organic solvent (TBP) will help selectively extract iron (III) in the process and indirectly remove most of the impurities. However, without the organic solvent, the process only removes elements that cannot be dissolved by HCl, that is the reason for the difference in the types of phases present in the sample. Also, using the ore without any purification reveals more phases in the sample.
The effect of leaching temperature

Figure 7(a) shows the XRD pattern of samples prepared by varying leaching temperature ($60\,^\circ\mathrm{C}$, $80\,^\circ\mathrm{C}$, $100\,^\circ\mathrm{C}$). As the temperature increases, some elements like potassium, calcium, and magnesium dissolve more with hydrochloric acid than iron, and the XRD pattern confirms a phase difference between the samples. Therefore, more impurities will be present in the solution. This will affect the nucleation rate leading to different crystallite sizes. The result indicated that as the leaching temperature increases, the crystallite size increases. However, at $100\,^\circ\mathrm{C}$, the peak intensity becomes large. This suggests that the magnetite nanoparticles phase present is large compared to the lower temperatures ($60\,^\circ\mathrm{C}$, $80\,^\circ\mathrm{C}$).
The effect of modifier concentration

To study the effect of modifier concentration on crystallite size, XRD measurement was carried out, as shown in figure 7(b). From the XRD pattern, as the modifier concentration increase, there is no significant phase difference between the samples. The crystallite size of the nanoparticles was found to increase with increasing modifier concentration. This may be due to an increase in alcohol concentration in the organic phase will help transfer most of the iron ion to the organic phase. This may have an impact on the crystal growth process. Furthermore, samples prepared without diluent produces different phases, which needs further investigation.

Figure 8 shows the thermal properties of the material and the point of weight loss using TGA. It can be seen that for the three samples prepared at different leaching temperature (a = 60 °C, b = 100 °C, and c = 80 °C), a sudden weight loss, in the beginning, was observed with increasing temperature up to 100 °C for the three samples caused by removing water molecules. For sample a, after 100 °C, there is a slight weight gain. This is due to oxidation. For sample b, after 100 °C, the weight loss is gradual, and it is due to dehydration of the β-FeOOH phase in the sample. A significant amount of weight loss was observed for sample c, between 100 °C and 200 °C. This might be because of the decomposition of impurities in the sample, which is also observed in the XRD
pattern. From all the three samples, sample extracted at a leaching temperature of 60 °C has more stable weight loss than the others.

4. Conclusion

Elemental composition and phases present in Ethiopian iron ore were characterized using EDS and XRD, respectively. The result revealed that the iron ore was low-grade and containing with other impurities. From the study, it can be concluded that

1. Magnetite nanoparticle can be synthesized directly from the iron ore, using the solvent extraction method as confirmed by XRD analysis, EDS, and FTIR spectra of having some impurities.

2. Two types of organic solvent extractant (TOPO and TBP) were used to compare the effect on the crystallite size of the magnetite nanoparticles, and the results show that samples synthesized using TBP produce small crystallite size (14 nm) compared with TOPO (20 nm), keeping other parameters constant. MNPs without the use of organic solvent was also synthesized, having more impurities and large crystallite size (40 nm) as expected.

3. The effect of leaching temperature (60 °C, 80 °C and 100 °C) and modifier concentration (30%, 40% and 50%) was also investigated, keeping other parameters constant. The results show that the increase in the concentration of modifier and leaching temperature increases the MNPs crystallite size.

4. The thermal stability of the synthesized sample at different leaching temperature was analyzed with TGA showing thermally stable magnetite nanoparticles at a leaching temperature of 60 °C.

Acknowledgments

The authors would like to thank Ethiopian Biotechnology Institute for financial support, Tezpur University, chemistry department for allowing laboratory facility, Jimma University, Ethiopian Conformity Assessment Enterprise, Geological Survey of Ethiopia and Addis Ababa Science and Technology University.

ORCID iDs

Kebede Gamo Sebehanie  https://orcid.org/0000-0001-8745-3632
Olu Emmanuel Femi  https://orcid.org/0000-0002-6069-7968

References

[1] Lu A H, Salabas E L and Schüth F 2007 Magnetic nanoparticles: synthesis, protection, functionalization, and application Angew. Chem. Int. Ed. 46 1223
[2] Sudimack J B A and Lee R J 2000 Targeted drug delivery via the folate receptor Adv. Drug Deliv. Rev. 41 148
[3] Hu F, Wei L, Zhou Z, Ran Y, Li Z and Gao M 2006 Preparation of biocompatible magnetite nanocrystals for in vivo magnetic resonance detection of cancer Adv. Mater. 18 2553
[4] Song H, Choi J, Huh Y, Kim S, Jun Y, Suh J and Cheon J 2005 Surface modulation of magnetic nanocrystals in the development of highly efficient magnetic resonance probes for intracellular labeling J. Am. Chem. Soc. 127 9992
[5] Yang H-H, Zhang S-Q, Chen X-L, Zhuang Z-X, Xu J-G and Wang X-R 2004 Magnetite-containing spherical silica nanoparticles for biocatalysis and bioseparations Anal. Chem. 76 1317
[6] Jordan A, Scholz R, Maier-hauf K, Johannsen M, Wust P, Nadobny J, Schirra H, Schmidt H, Deger S and Loening S 2001 Presentation of a new magnetic field therapy system for the treatment of human solid tumors with magnetic fluid hyperthermia J. Magn. Magn. Mater. 225 120
[7] Kolhatkar A G, Jamison A C, Litvinov D, Willson R C and Lee T R 2013 Tuning the magnetic properties of nanoparticles Int. J. Mol. Sci. 14 15978
[8] Baumgartner J, Bertinetti L, Wildrat M, Hirt A M and Faivre D 2013 Formation of magnetite nanoparticles at low temperature: from superparamagnetic to stable single domain particles PLoS One 8 1
[9] Goya G F, Berquó T S, Fonseca F C and Morelas M P 2003 Static and dynamic magnetic properties of spherical magnetite nanoparticles J. Appl. Phys. 94 3520
[10] Mullin J W 2001 Crystallisation. 4th ed. (Amsterdam: Elsevier)
[11] Kubota M, Yokota M and Mullin J W 2000 The combined influence of supersaturation and impurity concentration on crystal growth J. Crystal Growth 212 485
[12] Zhang S-B, Stepanski M, Yuan J-J and Ulrich J 1990 Investigations of crystal growth rates in the presence of different additives [in] Proceedings of the 11th International Symposium on Industrial Crystallization (Garmisch-Partenkirchen, Germany: A. Mersmann) 690
[13] Rashkovich L N and Kronsky N V 1997 Influence of Fe + and Al3 + ions on the kinetics of steps on the {100} faces of KDP J. Crystal Growth 182 436
[14] Nabiyouni G, Julaee M, Ghanbari D, Aliabadi P C and Safaie N 2015 Room temperature synthesis and magnetic property studies of Fe3O4 nanoparticles prepared by a simple precipitation method J. Ind. Eng. Chem. 21 599

[15] Wu S, Sun A, Zhai F, Wang J, Xu W, Zhang Q and Volinsky A A 2011 Fe3O4 magnetic nanoparticles synthesis from tailings by ultrasonic chemical coprecipitation Mater. Lett. 65 1883

[16] Mascolo M C, Pei Y and Ring T A 2013 Room temperature coprecipitation synthesis of magnetite nanoparticles in a large pH window with different bases Materials Basel6 3530

[17] Tajabadi M and Khosroshahi M E 2012 New finding on magnetite particle size reduction by changing temperature and alkaline media concentration APCBEE Proced. 3 141

[18] Deng Y H, Wang C C, Hu J H, Yang W L and Fu S K 2005 Investigation of formation of silica–coated magnetite nanoparticles via sol–gel approach Colloids Surfaces A: Physicochem. Eng. Asp. 262 87

[19] Lee B and Koo S 2011 Preparation of silver nanoparticles on the surface of fine magnetite particles by a chemical reduction J. Ind. Eng. Chem. 17 764

[20] Grzeta B, Risti M, Nowik I and Musi S 2002 Formation of nanocrystalline magnetite by thermal decomposition of iron choline citrate J. Alloys Compd. 334 305

[21] Lian S, Wang E, Kang Z, Bai Y, Gao L, Jiang M, Hu C and Xu L 2004 Synthesis of magnetite nanorods and porous hematite nanorods Solid State Commun. 129 486

[22] Mizutani N, Iwasaki T, Watanabe S, Yanagida T, Tanaka H and Kawai T 2008 Effect of ferrous/ferric ions molar ratio on reaction mechanism for hydrothermal synthesis of magnetite nanoparticles Bull. Mater. Sci. 31 715

[23] Bandhu A, Mukherjee S, Acharya S, Modak S, Braham K S, Das D and Chakraborti P K 2009 Dynamic magnetic behavior and Mössbauer effect measurements of magnetite nanoparticles prepared by a new technique in the coprecipitation method Solid State Commun. 149 1790

[24] Gnanaprakash G, Mahadevan S, Jayakumar T, Kalyanasundaram P, Philip J and Raj B 2007 Effect of initial pH and temperature of iron salt solutions on formation of magnetite nanoparticles Mater. Chem. Phys. 103 169

[25] Rivasa-Sanchez M L, Alva-Valdivia I M, Arenas-Alatorre J, Urrutia-Fucugauchi J, Perrin M, Goguitchaichvili A, Ruiz-Sandoval M and Ramos M A 2009 Molina, natural magnetite nanoparticles from an iron-ore deposit: size dependence on magnetic properties Earth Planet. Space 61 158

[26] Ud F R 2017 Manufacturing of Iron Oxide Nanoparticles Through Mechanical Milling [Dissertation] (Barcelona: Universitat Politècnica de Catalunya) 67

[27] Darezereshki E, Darban A. k., Abbodlaha M and Jamshidi A 2018 Synthesis of magnetite nanoparticles from iron ore tailings using a novel reduction–precipitation method J. Alloys Compd. 749 536

[28] Nouioua A and Barkat D 2017 Liquid–liquid extraction of iron (iii) from ouenza iron ore leach liquor by tributylphosphate J. Fundam. Appl. Sci. 9 1483

[29] Mao X 2015 Solvent extraction of iron (III) from chloride acid solutions by decanol, [in] The 3rd International Conference on Material, Mechanical and Manufacturing Engineering, Sichuan 27 131

[30] MAO X H and LIU D J 2013 Solvent extraction separation of titanium(IV) and iron(III) from acid chloride solutions by triethyolphosphine oxide Asian J. Chem. 25 4756

[31] Wei X and Vaidero R C Jr. 2007 Synthesis of magnetite nanoparticles with ferric iron recovered from acid mine drainage: implications for environmental engineering Colloids, and Surfaces A: Physicochem. Eng. Aspects. 294 285

[32] Cai X, Wei B, Han J, Li Y, Cui Y and Sun G 2016 Solvent extraction of iron(III) from hydrochloric acid solution by N,N,N′-tetra-2-ethylhexylidiglycolamide in different diluents Hydrometallurgy 164 5

[33] Saktivel R, Vasumathi N, Sahu D and Mishra B K 2010 Synthesis of magnetite powder from iron ore tailings Powder Technol. 201 190

[34] Suh Y J, Do T M, Kil D S, Jang H D and Cho K 2015 Production of high-purity magnetite nanoparticles from a low-grade iron ore via solvent extraction Korean Chem. Eng. Res. 53 40

[35] Karimzadeh I, Rezagholidpour Dizaji H and Aghazadeh M 2016 Preparation, characterization and PEGylation of superparamagnetic Fe3O4 nanoparticles from ethanol medium via cathodic electrochemical deposition (CED) method Mater. Res. Express 3 33

[36] Peng M, Li H, Luo Z, kong J, Wan Y, Zheng L and Zhang Q 2015 Dextran–coated superparamagnetic nanoparticles as potential cancer drug carriers in vivo Nanoscale 7 11158

[37] Karimzadeh I, Dizaji H R and Aghazadeh M 2016 Development of a facile and effective electrochemical strategy for preparation of iron oxides (Fe3O4 and γ-Fe2O3) nanoparticles from aqueous and ethanol mediums and in situ PVC coating of Fe3O4 superparamagnetic nanoparticles for biomedical applications J. Magn. Magn. Mater. 416 83

[38] Petcharoen K and Sirivat A 2012 Synthesis and characterization of magnetite nanoparticles via the chemical coprecipitation method Mater. Sci. Eng. B 177 421

[39] Kashchiev D 2000 Nucleation – Basic Theory with Applications (Oxford: Butterworth-Heinemann)