Published in final edited form as:

J Phys Chem Solids. 2009 March 1; 70(3): 555–560. doi:10.1016/j.jpcs.2008.12.017.

Microwave Assisted Synthesis of Iron(III) Oxyhydroxides/Oxides Characterized Using Transmission Electron Microscopy, X-ray Diffraction, and X-ray Absorption Spectroscopy

J.G. Parsons¹, C. Luna², C.E. Botez³, J. Elizalde³, and J.L. Gardea-Torresdey^{1,2,*}

¹University of Texas at El Paso Department of Chemistry, 500 W University Ave El Paso TX 79986

²University of Texas at El Paso Environmental Science and Engineering PhD program, 500 W University Ave. El Paso TX 79968.

³University of Texas at El Paso Department of Physics, 500 W University Ave El Paso TX 79986

Abstract

Microwave assisted synthesis of iron oxide/oxyhydroxide nanophases was conducted using iron(III) chloride titrated with sodium hydroxide at seven different temperatures from 100°C to 250°C with pulsed microwaves. From the XRD results, it was determined that there were two different phases synthesized during the reactions which were temperature dependent. At the lower temperatures, 100°C and 125°C, it was determined that an iron oxyhydroxide chloride was synthesized. Whereas, at higher temperatures, at 150°C and above, iron(III) oxide was synthesized. From the XRD, we also determined the FWHM and the average size of the nanoparticles using the Scherrer equation. The average size of the nanoparticles synthesized using the experimental conditions were 17, 21, 12, 22, 26, 33, 28 nm, respectively for the reactions from 100°C to 250°C. The particles also had low anisotropy indicating spherical nanoparticles, which was later confirmed using TEM. Finally, XAS studies show that the iron present in the nanophase was present as iron(III) coordinated to six oxygen atoms in the first coordination shell. The higher coordination shells also conform very closely to the ideal or bulk crystal structures.

Keywords

Iron oxide/oxyhydroxide nanoparticles; X-ray diffraction; XAFS; microwave assisted synthesis

Introduction

The synthesis of nanoparticles is plagued by large variations in size for a particular synthesis technique. However, more recent advances in the size control of nanoparticles has been achieved though the use of varying surfactants and capping agents¹⁻³. These new techniques have resulted in controlled growth of nanoparticles in terms of both size and shape¹⁻³. However, another technique that has not been well investigated is the use of microwave assisted synthesis of nanoparticles. There are a few studies on the literature on the use of microwave assisted

© 2009 Elsevier Ltd. All rights reserved.

*Corresponding Author: email: jgardea@utep.edu, phone: (915)747-5359, fax: (915)-747-5847.

Publisher's Disclaimer: This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final citable form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

synthesis of iron oxide nanoparticles⁴⁻⁷. These studies have not shown the full effect of microwave assisted synthesis on iron oxide/oxyhydroxide synthesis.

Microwave assisted synthesis has been used in the synthesis of organic/organometallics, and pseudo peptides, and peptides⁸⁻¹⁴. In addition, the use of microwave assisted synthesis has been used for the synthesis of a number of catalysis, inorganic compounds such as iron oxides and ceramics^{4-7, 15-19}. Generally, microwave assisted synthesis of nanophase compounds and catalysts show better uniformity and dispersion within different matrices²⁰.

The microwave synthesis of iron oxide/oxyhydroxide nanoparticles has shown some interesting results. Jansen *et al.* have shown that gamma-iron(III) oxide nanoparticles have an average size of 4-5 nm²¹. Whereas, Liao *et al.* have synthesized amorphous iron oxide nanoparticles in the size range of 2-3 nm⁵. Kholam *et al.* have prepared submicron magnetite nanoparticles through the use of microwave assisted synthesis⁶. However, most of these studies have been performed at relatively low iron concentrations in the range of millimole concentrations.

In this study, the effects of microwave assisted synthesis were studied on a iron(III) chloride and sodium hydroxide chemical system without a capping agent. The reactions were carried out without modification to the microwave system and closed microwave vessels. The reaction studied was the titration of iron(III) chloride with sodium hydroxide to synthesize iron-oxide/oxyhydroxide nanoparticles. Seven different temperatures were investigated from 100°C to 250 °C in increments of 25°C. The resulting nanoparticles were characterized using X-ray absorption spectroscopy (XAS), transmission electron microscopy (TEM), and X-ray diffraction (XRD).

Methodology

Synthesis of the Iron/oxide/oxyhydroxide nanophases

A 1.0 M iron(III) chloride was made from iron(III) chloride (hydrate salt) ACS grade in 18 MΩ Millipore water. The iron(III) chloride solution was prepared by dissolving 157.89g of iron(III) chloride hexahydrate in 1.0 L of water. A 1.0 M solution of NaOH was prepared using ACS grade sodium hydroxide. The sodium hydroxide solution was prepared by adding 4.01g of NaOH to 18 MΩ Millipore water and diluting to 100 mL with 18 MΩ Millipore water. Serial dilutions of the stock solutions were performed to make 0.75, 0.5, and 0.25 M solutions of both the FeCl₃ and NaOH solutions again using 18 MΩ Millipore water.

Subsequent to solution preparation, the sodium hydroxide was added slowly into the iron(III) chloride solution at an approximate rate of 0.1 mL/min using a burette. The titrations were performed until the moles of hydroxide in solution were equivalent to the number of moles of iron(III) in solution. The titrations were performed at a slow rate (approximately 0.01 mL/min) to avoid the precipitation of Fe(OH)_{3(s)}.

The prepared solutions were then microwaved in a PerkinElmer Multiwave 2000 system (Waltham, MA) at varying temperatures including: 100, 125, 150, 175, 200, 225, and 250 °C. The program used for the synthesis was temperature controlled meaning that the samples were pulsed with microwaves to maintain a constant temperature. The temperature was ramped initially to the required temperature within 3 minutes and held constant for 30 minutes. The initial power used in the study was 1000 W. After the solution reached the programmed temperature, the power was pulsed to maintain a constant temperature. After microwaving, the samples were oven dried at 100 °C until they were completely dry. The samples were then weighed and ground for further analysis using either: XRD, XAS or TEM.

XRD Analysis

Powder diffraction patterns were collected using a Siemens D5000 powder diffractometer (New York, NY) equipped with a position sensitive detector (PSD) in the θ 2 θ geometry by scanning 2 θ from 20 to 55°. 2 θ steps of 0.01° were employed and a counting time of 10 seconds was used to increase the signal-to-noise ratio. All spectra were background corrected and normalized. The samples were then fitted using FullProf Suit 2006 (CNRS, France) with input files created using crystallographic data from the literature. The full width at half max (FWHM) from 3 different peaks were used in Scherrer's equation to determine the average size of the nanoparticles.

XAS Data Collection

The iron nanophase samples were ground with boron nitride to give a one absorption unit change across the absorption edge. Subsequent to grinding, the samples were packed into 1.0 mm Al sample holders with Kapton® tape windows for XAS analysis. The XAS spectra were collected at Stanford Synchrotron Radiation Laboratory (SSRL) on beamline 10-2. The sample spectra were collected using the on the iron K edge (7.112 keV) using an internal Fe(0) metal foil standard. The conditions of the data collection were as follows: a beam energy of 3.0 GeV, a current of 100 mA, 1.0 mm vertical slit, a 10.0 mm horizontal slit, a Si 220 double crystal monochromator (ϕ 90), a detuning of 30 percent, and N₂ filled ion chambers.

XAS Data Analysis

The XAS data analysis was performed using the WinXAS V 3.1 software and standard data analysis techniques^{22, 23}. The spectra were first energy calibrated using the internal Fe(0) foil spectra E_0 (7.112 keV). The calibration energy was calculated using a second degree derivative of the Fe(0) internal standard and the difference between the calculated and the literature value was applied as a shift factor to the data. After calibration, the data was background corrected and normalized. The background correction was performed using a two polynomial fitting of the sample spectra, a first degree polynomial was fitted to the pre-edge region and a fourth degree polynomial was fitted to the post edge region. The background corrected sample spectra were then normalized to one absorption unit. At this point, the XANES regions of each of the sample spectra were extracted by sectioning the spectra from 7.07-7.22 keV.

The extraction of the EXAFS from the spectra were performed on the background corrected and normalized XAS spectra. First, the spectra were converted to k space based on the energy of the photoelectron ejected from the sample. The energy of the photoelectron ejected from the samples was calculated using a second degree derivative of the sample spectra edge. Once the samples were in k-space, a μ fitting was performed using a k weight of 3 and a spline of 4 knots from 2.0 to 12.2 Å⁻¹. The data was then Fourier transformed over the same range and subsequently back transformed from 0.55 to 3.86 Å and fitted. The fitting of the sample spectra was performed using least squared fitting techniques and calculations from FEFF 8.00²⁴. The fittings were used to determine the coordinating atoms, the interatomic distances and thermal disorder parameters in the samples. The input files for the FEFF calculations were performed using the ATOMS software and crystallographic data of akaganeite and hematite from the literature as determined from the XRD fitting results²⁵⁻²⁷.

TEM

The iron solutions were dried after synthesis onto 200 mesh copper TEM grids coated with silicon monoxide/Formvar. The samples were analyzed using a JOEL 2010 HRTEM (University of New Mexico, Albuquerque, NM.) with a voltage of 200 keV.

Results and Discussion

Figure 1 shows the diffraction patterns for the iron products synthesized from 100°C to 250°C using microwave assisted synthesis. As can be seen in the diffraction patterns, there are two different products synthesized in the reactions. The two lowest temperatures, 100 °C and 125 °C, have one product, whereas, the reactions at 150 °C to 250 °C form a second product. Through the use of XRD fitting in FullProf using a Le bail fitting, it was determined that the product formed at 100°C and at 125°C is an iron(III) oxyhydroxide chloride commonly known as akaganeite²⁶. The fitting of the diffraction pattern is shown in Figure 2, which had a chi squared value of 2.78. Whereas, at temperatures from 150°C through 250°C, the final product synthesized was iron(III) oxide or hematite²⁷, which had a final chi squared value of 4.98 (as determined from the Le Bail fitting), as shown in Figure 3.

The starting materials were the same for all reactions investigated in this study, including the actual times for the reaction and the heating times. In addition, the reactions were all carried out in closed microwave reaction vessels. The only difference in the reactions was the heating temperature. It has been proposed that the reaction that occurs in the iron(III) chloride/ sodium hydroxide system is as follows:

However, the conversion kinetics for the transformation of the iron(III) hydroxide to iron oxyhydroxide are rather slow taking from 24 hours to several days²⁸⁻³⁰. The second transformation from the iron(III) oxyhydroxide to iron(III) oxide are rather slow requiring increases in temperature and the presence of oxygen²⁸⁻³⁰. However, in the microwave assisted synthesis these conversions were achieved in less than 1 hour in closed vessels. The chloride adduct of the FeOOH is formed at lower temperatures (100°C and 125°C) in the presence of high concentrations of Cl⁻ ions in solution. In addition, at the higher temperatures, the Cl⁻ adduct should be formed initially but the higher pressure and temperature force the conversion of the FeOOH-Cl to the Fe₂O₃ product.

The average grain sizes of the different products from the synthesis were determined from the FWHM of the XRD peaks using the Scherrer equation. The results of the calculations for the average grain size in nanometers are shown in Figure 4. The average size of the nanoparticles were determined using three independent peaks from the diffraction patterns and the error bars included in the figure are the standard error of this calculation. As can be seen in Figure 4, generally as the temperature increases the average size of the nanoparticles for both of the products increases. However, the average grain size of the nanoparticles stays within a very small range of the average as indicated by the small error on the grain size calculations. This indicates that this synthesis technique has a good control over the average particle size, which is desired in nanoparticle synthesis. For the iron(III) oxyhydroxide chloride nanoparticles, the initial size of the nanoparticles is approximately 17 nm and it increases to approximately 21 nm. However, at 150°C the average size of the iron(III) oxide nanoparticles is approximately 13 nm and increases to approximately 27 nm at 250 °C. The decrease in the average size of the iron oxide nanoparticles at 150°C is caused by the phase change in the crystal structure from FeOOH-Cl to the Fe₃O₄ product. In addition, the effect on the average size of the nanoparticles from 0.25M to 1.0 M solutions at 30 minute reaction times from 150 through 200 °C was investigated. The effects of the concentration of the starting material are presented in Figure 5. As can be seen in Figure 5, the concentrations of the starting materials apparently

have little effect on the average grain size of the nanoparticles. The average grain size of the nanoparticles remains around 16, 26, and 30 nm indicating very small changes in the average grain size of the nanoparticles. Overall there is a small effect on the average size of the nanoparticles with increasing moles of reactants approximately from 0.25 M to 1.0 M of initial FeCl_3 at all the temperatures investigated.

In addition to recording the XRD patterns of the nanoparticles, we also used TEM to verify the results and to determine the shape of the nanoparticles. The XRD results indicate that the particles are in a spherical geometry as indicated by the similar FWHM calculations for the average grain size^{31, 32}. High preferred growth occurs when nanoparticles grow more along one axis compared to the other two axes in the crystal^{31,32}. The average grain sizes for the particles have a very low error indicating that the growth is the same on all the axes in three dimensions. This was confirmed using TEM, as shown in Figure 6, which is a TEM micrograph of the 100°C synthesis product. As can be seen in this figure, the particles are spherical with an average grain size of approximately 15 nm agreeing with the XRD results.

Further studies into the nanoparticles were conducted using XAS (X-ray absorption spectroscopy), with both XANES (X-ray absorption near edge spectroscopy) and EXAFS (extended x-ray absorption fine structure). Figure 7 shows the XANES spectra for the iron products synthesized for the 30 minute reaction time and the 1.0 M initial iron(III) concentration. As can be seen in the figure, there is not much change in the atomic structure of the samples from the 100°C synthesis through the 250°C synthesis. In addition, all the iron present in the samples is present as iron(III), this can be concluded from the presence of the iron(III); this can be concluded for the presence of the pre-edge in the XANES spectra and the position of the absorption edge. The EXAFS of the iron oxy-hydroxide nanophases synthesized at 100°C and 250° C, shown in Figure 8, are representative of both sets of products obtained from these reactions. As can be seen in Figure 8, there are not many differences in the spectra between the two different species. Even though the 100 °C and 125 °C synthesis products have the presence of Cl^- in the crystal structure, it is so far removed from the iron atoms that it actually has no effect on the local coordination environment. The fittings of the EXAFS are presented in Table 1. All the samples above 150°C show a full first coordination shell consisting of six oxygen atoms split into two different coordination shells. The first oxygen coordination shell consists of three oxygen atoms at approximately 1.98 Å and the second coordination shell consists of three oxygen atoms at approximately 2.1 Å, which has been previously reported in the literature for these types of compounds. However, the lower temperature syntheses at 100° C and 125°C create products that have low coordination numbers in the second oxygen shell. This indicates smaller particle size. The first shell mixtures of oxygen and iron atoms are apparent in the EXAFS. However, after the first shell, the coordination numbers start to increase with increasing particle size as is expected. The larger the particle the higher the coordination numbers until it reaches close to the maximum observed in the crystal structure for each of the coordination shells. The results of the XAS studies complement the results from the TEM and XRD. The nanoparticles are rather large and have a well defined crystal lattice.

Conclusions

The microwave assisted synthesis of iron oxide/oxyhydroxide nanoparticles is feasible using a standard microwave oven, and high concentrations of the starting materials. The nanoparticles formed from the slow titration of FeCl_3 with sodium hydroxide have controlled growth and controlled crystal structure dependent on the temperature of the synthesis. In addition, the nanoparticles synthesized using this techniques have similar growth on three different axes of the crystal structure as the anisotropy in the crystals is low indicating that the nanoparticles are spherical in nature, as was confirmed by TEM.

Acknowledgments

The authors would like to acknowledge funding from SANDIA National Laboratory and the El Paso Water Utilities (Contract Number: 532586). The National Institutes of Health (grant S06 GM8012-33) and the University of Texas at El Paso's Center for Environmental Resource Management (CERM). We also thank the financial support from the Southwest Center for Environmental Research and Policy (SCERP) program, and the HBCU/MI, Environmental Technology Consortium that is funded by the Department of Energy (Grant Number: A0513). Portions of this research were carried out at the Stanford Synchrotron Radiation Laboratory, a national user facility operated by Stanford University on behalf of the U.S. Department of Energy, Office of Basic Energy Sciences. The SSRL Structural Molecular Biology Program is supported by the Department of Energy, Office of Biological and Environmental Research, and by the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program. Dr. Gardea-Torresdey acknowledges the Dudley family for the Endowed Research Professorship in Chemistry.

References

1. Koutzarova T, Kolev S, Ghelev Ch, Paneva D, Nedkov I. Microstructural study and size control of iron oxide nanoparticles produced by microemulsion technique. *Physica Status Solidi C* 2006;3:1302–1307.
2. Iijima M, Yonemochi Y, Tsukada M, Kamiya H. Microstructure control of iron hydroxide nanoparticles using surfactants with different molecular structures. *J. Coll. Interface Sci* 2006;298:202–208.
3. Pileni MP, Moumen N, Hocheplid JF, Bonville P, Veillet P. Control of the size of cobalt ferrite nanoparticles: synthesis and properties. *J. de Phys. IV* 1997;7:C1/505–C1/508.
4. Rigneau P, Bellon K, Zahreddine I, Stuerger D. Microwave flash-synthesis of iron oxides nanoparticles. *Eur. Phys. J* 1999;7:41–43.
5. Liao X, Zhu J, Zhong W, Chen H-Y. Synthesis of amorphous Fe₂O₃ nanoparticles by microwave irradiation. *Mater. Lett* 2001;50:341–346.
6. Kholam, YB.; Dhage, SR.; Venna, S.; Potdar, HS.; Deshpande, SB.; Joy, PA.; Date, SK. Magnetic properties of nanosized ferrite powders synthesized by microwave-hydrothermal method. Proceedings of the 9th International Conference on Ferrites (ICF-9); San Francisco, CA, United States. 2005.
7. Li Q, Wei Y. Study on preparing monodispersed hematite nanoparticles by microwave-induced hydrolysis of ferric salts solution. *Mater. Res. Bull* 1998;33:779–782.
8. Hong RY, Pan TT, Li HZ. Magnetic field synthesis of Fe₃O₄ nanoparticles used as a precursor of ferrofluids. *J. Mag. Mag. Mater* 2006;303:60–68.
9. Lehmann F, Scobie M. Rapid and convenient microwave - assisted synthesis of primary amines via reductive N-alkylation of methyl carbamate with aldehydes. *Synthesis* 2008;(11):1679–1681.
10. Kremsner JM, Jennifer M, Stadler, Alexander, Kappe, Oliver C. The scale-up of microwave-assisted organic synthesis. *Top. Curr. Chem* 2006;266:233–278.
11. Artillo A, Della Sala G, Santis De M, Llordes A, Ricart S, Spinella A. Preparation of organometallic uracil-analog Fischer carbene complexes: Comparative study of conventional heating vs. microwave irradiation. *J. Organometal. Chem* 2007;692:1277–1284.
12. Ardon M, Hogarth G, Oscroft DTW. Organometallic chemistry in a conventional microwave oven: the facile synthesis of group 6 carbonyl complexes. *J. Organometal. Chem* 2004;689:2429–2435.
13. Palasek SA, Cox ZJ, Collins JM. Limiting racemization and aspartimide formation in microwave-enhanced Fmoc solid phase peptide synthesis. *J. Pep. Sci* 2007;13:143–148.
14. Park M-S, Shim H-S, Cho W-M, Lee K-H. Microwave-assisted solid-phase synthesis of pseudopeptide containing reduced amide bond. *Pep. Sci* 2006;43:70–71.
15. Monroc S, Feliu L, Planas M, Bardaji E. Microwave-assisted cyclization of peptides on SynPhase lanterns. *Synlett* 2006;9:1311–1314.
16. Ding S-W, Wang L-Y, Ding Y, Zhang M-H, Wang Z-X. Microwave synthesis and photocatalysis of nano-TiO₂ mix-crystals. *Yingyong Huaxue* 2006;23:651–654.
17. Glaspell G, Fuoco L, El-Shall MS. Microwave Synthesis of Supported Au and Pd Nanoparticle Catalysts for CO Oxidation. *J. Phys. Chem. B* 2005;109:17350–17355. [PubMed: 16853217]
18. Kim K-Y, Ahn WS, Park DW, Oh JH, Lee CM, Tai WP. Microwave synthesis of titanium silicalite-1 using solid phase precursors. *Bull. Korean Chem. Soc* 2004;25:634–638.

19. Vanetsev AS, Makshina EV, Oleynikov NN, Tret'yakov YD, Romanovskii BV. V. Microwave synthesis of lanthanum strontium cobaltites and study of their catalytic activity. *Doklady Chem* 2005;405:226–229.
20. Wang Z-N, Z.-N. Zhu J-Z, Zhong H. Study on microwave synthesis of CeO₂-catalyzed cobalt phthalocyanine molecular sieve and catalysis of H₂O₂ decomposition. *Xiyou Jinshu Yu Yingzhi Hejin* 2004;32:5–8.
21. Janzen C, Wiggers H, Knipping J, Roth P. P. Formation and in situ sizing of gamma-Fe₂O₃ nanoparticles in a microwave flow reactor. *J. Nanosci. Nanotechnol* 2001;1:221–225. [PubMed: 12914055]
22. Ressler T. WinXAS: a new software package not only for the analysis of energy-dispersive XAS data. *J. de Phys* 1997;IV:269–270.
23. Parsons JG, Aldrich MV, Gardea-Torresdey JL. Environmental and biological applications of extended X-ray absorption fine structure (EXAFS) and X-ray absorption near edge structure (XANES) spectroscopies. *App. Spectros. Rev* 2002;37:187–222.
24. Ankudinov AL, Ravel BJ, Rehr JJ, Conradson DD. S.D. Real-space multiple-scattering calculation and interpretation of x-ray-absorption near-edge structure. *Phys. Rev. B* 1998;7565–7576.
25. Ravel B. ATOMS: crystallography for the x-ray absorption spectroscopist. *J. Synchro. Rad* 2001;8 (2):314–316.
26. Plachinda AS, Chertov VM, Suzdalev VM, Baldokhin IP, Yu V, Marakov EF, Zelentsov VI. Transition of ferrogel γ -ferric hydroxide oxide to α -ferric oxide under hydrothermal conditions syudied by γ -resonance spectroscopy. *Teoreticheskaya i Eksperimental'naya Khimiya* 1974;10:545–9.
27. Post JE, Buchwald VF. Crystal structure refinement of akaganeite. *American Mineralogist* 1991;76:272–277.
28. Blake RL, Hessevick RE, Zoltai T, Finger LW. Refinement of the hematite structure *American Mineralogist* 1966;51:123–129.
29. Mendelovici E, Villalba R, Sagarzazu A. Processings of iron oxides at room temperature. II. Mechanochemical reaction effects on the structure and surface of pure, synthetic lepidocrocite. *Mater. Res. Bull* 1982;17:241–9.
30. Desiraju GR, Rao M. A mild transformation of γ -iron hydroxide oxide to γ -ferric oxide using organic reagents. *Mater. Res. Bull* 1982;17:443–9.
31. Sun Y, Fuge GM, Ashfold MNR. Selenium-containing 15-Mer Peptides with High Glutathione Peroxidase-like Activity. *Chem. Phys. Lett* 2004;396:21–26.
32. Ma G, Zhao X, Zhu J. Microwave hydrothermal synthesis of rutile TiO₂ nanorods. *Int. J. Modern Phys. B* 2005;19:2763–2768.

Figure 1. Diffraction patterns collected for the synthesis products of FeCl_3 reacted with NaOH and heated to temperatures ranging from 100 °C through 250 °C under pulsed microwave irradiation.

Figure 2.
Fitting of 100 °C FeCl₃ sample normalized and back ground corrected data with a chi squared value of 2.78.

Figure 3. Fitting of 225 °C FeCl_3 sample normalized and back ground corrected data with a chi squared value of 4.98.

Figure 4. Average particle size from microwave assisted synthesis from 100 °C to 250 °C using iron(III) chloride and sodium hydroxide at 1:1 mole ratio and a 30 minute microwave time.

Figure 5. Size of nanoparticles formed at 150-250°C varying the initial iron(III) chloride concentrations using microwave assisted synthesis for 30 min reaction time.

Figure 6.
TEM micrograph of iron oxide nanoparticles synthesized at 100°C, with 30 minutes of pulsed microwave irradiation.

Figure 7. XANES region from 7.07 keV to 7.22 keV of iron oxyhydroxide and the products from the microwave assisted synthesis of iron oxides from 100°C through 250 °C.

Figure 8.

A. EXAFS of the 100°C FeCl₃ 30 minute microwave synthesis product, solid line represents the Fourier transform of the fitting the dotted line represents the Fourier transform of the raw data. **B.** EXAFS of the 250°C FeCl₃ 30 minute microwave synthesis product, solid line represents the Fourier transform of the fitting the dotted line represents the Fourier transform of the raw data. The insert shows the Raw EXAFS (dotted line), the back transformed (dashed line), and the Fitting of the back transform (solid line).

Table 1

EXAFS fittings obtained from the different iron oxide/oxyhydroxides synthesized using microwave irradiation from 100°C through 250°C.

Sample	Interaction	CN	R(Å)	$\sigma^2(\text{Å}^2)$
FeO 100°C MW Synthesis	Fe-O	2.7	1.98	0.0053
	Fe-O	0.8	2.13	0.0010
	Fe-Fe	0.6	2.64	0.011
	Fe-Fe	4.9	3.07	0.010
	Fe-O	5.7	3.33	0.015
	Fe-O	5.5	3.70	0.0012
FeO 125°C MW Synthesis	Fe-O	3.0	1.96	0.0075
	Fe-O	1.5	2.30	0.016
	Fe-Fe	1.6	2.67	0.012
	Fe-Fe	2.2	3.05	0.0026
	Fe-O	5.8	3.50	0.0014
	Fe-O	2.6	3.77	0.0022
FeO 150°C MW Synthesis	Fe-O	2.6	1.97	0.0033
	Fe-O	2.4	2.13	0.0039
	Fe-Fe	3.9	2.98	0.0056
	Fe-Fe	6.0	3.53	0.010
	Fe-O	5.1	3.55	0.0039
	Fe-O	5.4	3.72	0.004
FeO 175°C MW Synthesis	Fe-Fe	4.9	3.75	0.014
	Fe-O	2.6	1.93	0.0035
	Fe-O	2.7	2.07	0.0046
	Fe-Fe	2.9	2.95	0.0047
	Fe-Fe	4.4	3.40	0.0043
	Fe-O	5.3	3.58	0.0069
FeO 200°C MW Synthesis	Fe-Fe	5.2	3.58	0.0054
	Fe-O	4.7	3.75	0.0053
	Fe-O	2.7	1.95	0.0068
	Fe-O	2.5	2.08	0.029
	Fe-Fe	3.7	2.97	0.0065
	Fe-Fe	3.2	3.35	0.0040
	Fe-O	8.5	3.52	0.0040
	Fe-Fe	7.8	3.58	0.017

Sample	Interaction	CN	R(Å)	$\sigma^2(\text{\AA}^2)$
FeO 225°C MW Synthesis	Fe-O	4.2	3.72	0.0014
	Fe-Fe	1.7	3.78	0.0038
	Fe-O	2.2	1.95	0.0039
	Fe-O	3.6	2.13	0.021
	Fe-Fe	2.4	2.91	0.0042
	Fe-Fe	3.5	3.35	0.0013
	Fe-O	7.8	3.48	0.0010
	Fe-Fe	3.5	3.69	0.0089
	Fe-O	10.5	3.71	0.0060
FeO 250°C MW Synthesis	Fe-O	3.35	1.98	0.0059
	Fe-O	2.3	2.15	0.0056
	Fe-Fe	4.3	2.99	0.0064
	Fe-Fe	1.8	3.44	0.0051
	Fe-Fe	2.7	3.69	0.0028
	Fe-O	8.8	3.78	0.0082

Note: MW represents microwave synthesis, CN represent the coordination number, R is the interatomic distance given in Å, and the σ^2 is the Debye-Waller factor given in Å².