

Supporting data

Influence of Precursor on Phase evolution of nano iron oxide/oxyhydroxides: optical and magnetic properties

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Table S₁ Some of the recent literature on synthesis of iron oxides/oxyhydroxides

Method	Procedure	Result
Hydrothermal ¹⁹	K ₃ Fe(CN) ₆ , NaOH solutions. Heated to 120-200 °C in autoclave	Flowerlike α-Fe ₂ O ₃
Calcination ²⁰	FeCl ₃ ·6H ₂ O solution heated at 100 °C for 4 h, After drying annealed in a tube furnace at 350 °C for 2h.	α-Fe ₂ O ₃ nanorods with an average L of 400 nm and dia of 75 nm.
Solvothermal ²¹	Ferrous ammonium sulfate, H ₂ O ₂ , pH 3, 24 h stirring.	Crystallization to goethite and/or hematite.
Solvothermal ²²	Ferric chloride, ethanol. Heated to 180 °C for 15 h in autoclave	Peanut-like α-Fe ₂ O ₃ super structures.
Hydrothermal ²³	FeSO ₄ ·7H ₂ O, NaClO ₃ . Heated to 160- 180 °C for several hours in autoclave	Three-dimensional (3D) urchin-like α-Fe ₂ O ₃ nanorods
Solvothermal ²⁴	Ferric acetylacetonate distilled water. Heated at temperatures (140–180 °C) and reaction times (1.5–12 h)	Different product morphologies of α-Fe ₂ O ₃ .
Hydrothermal ²⁵	FeCl ₃ , FeNO ₃ . Urea, NaH ₂ PO ₄ /NH ₄ OH. Solution heated at 90 °C for 3h followed by autoclave 115 °C 24 h,	Nano rod/ellipsoidal α-Fe ₂ O ₃
Calcination ²⁶	FeCl ₃ , FeCl ₂ , HCl, NH ₃ Mixed solutions kept at room temperature for 2h. Calcined in air at 500 °C for 1-2 h.	Nano particle α-Fe ₂ O ₃
Hydrothermal calcination ²⁷	Tri block copolymer (HO(CH ₂ CH ₂ O) 106(CH ₂ -CH(CH ₃)O) ₇₀ (CH ₂ CH ₂ O) ₁₀₆ H) (F127), EG, Fe(NO ₃) ₃ ·9H ₂ O. vigorous stirring 2 h, aged in air at 40 °C for 7 days, gradual heating to 150 °C (1 °C/min) and maintaining at 150 °C for 24 h. Finally, sintered at 400 °C for 5 h	α-Fe ₂ O ₃ nanostructure with different unique morphology.
Hydrothermal calcination ²⁸	FeCl ₃ , NaOH autoclaved 150 °C for 18 h Washed upto pH 7 dried at 100 °C., calcined 600 °C for 1 h	α-Fe ₂ O ₃ nanostructure with different unique morphology

Hydrothermal ²⁹	Fe(acac) ₃ , ethanol. Heated in autoclave at 150 °C for 24 h	Mesocrystalline hematite nanoplates
Microwave-aging- Calcination ³⁰	Fe(NO ₃) ₃ , NaHCO ₃ microwaved until boiling, aged for 3 days. pH adjusted at 12 aged at 90 °C for 30–168 h. heated at 300°C in air for 1 h	Porous Y-shaped hematite rods
Calcination ¹	The ferric nitrate heated up to 800 °C for 4 h.	Pomegranate-like hematite
Hydrothermal ³¹	Ferric chloride, urea were added into the THF– ethanol mixture followed by the addition of PVP under ultrasonic conditions for a few minutes. Heated in autoclave at 180 °C for 12 h. Dried in air at 60°C for 6 h.	3D hierarchical hematite nanostructure,
Surfactant mediated- hydrothermal ³²	CTAB, ferric chloride solution Stirred to form homogeneous clear solution. Heated in autoclave at 120°C for 12 h.	α -Fe ₂ O ₃ nano rhombohedra, nanorods, and nanocubes
Hydrothermal calcination ³³	Ferric nitrate, sodium citrate, and urea solution heated at 160 °C for 10 h in autoclave. Precipitate washed with water and absolute ethanol, and dried at 60 °C for 4 h. Heating the precursor in air at 500 °C for 2 h.	α -Fe ₂ O ₃ porous nanospheres
Precipitation ³⁴	Ferric nitrate, KOH, final pH 6.5 ageing	goethite rods
Precipitation ³⁵	Ferric nitrate, NaNO ₃ , NaOH, pH 8 ageing	Nano ferrihydrite
Precipitation ³⁶	Ferric nitrate, NaOH, pH 7 ageing	Nano ferrihydrite
Precipitation ³⁷	Ferric chloride, NaOH, pH 7	Nano ferrihydrite

Table S₂. Crystallite Size and Lattice Parameters of K₁, K₂, K₃ and K₄ Samples Annealed at 400, 600 and 800 °C.

Annealing Temperature (°C)	Sample	MCD (nm)	a=b (Å)	c (Å)
400	K ₁	16±3	5.0497	13.7983
	K ₂	22±3	5.0494	13.7958
	K ₃	9±2	5.0489	13.8003
	K ₄	35±3	5.0503	13.7978
600	K ₁	46±4	5.0506	13.7996
	K ₂	49±5	5.0508	13.7943
	K ₃	25±3	5.0499	13.7983
	K ₄	57±5	5.0483	13.8018
800	K ₁	96±6	5.0526	13.7966
	K ₂	113±4	5.0523	13.7979
	K ₃	58±4	5.0516	13.7993
	K ₄	106±5	5.0497	13.8003

Table S₃. Mössbauer Parameters I.E., Isomer Shift (IS), Quadruple Splitting (QS), Hyperfine Field (B_{HF}), Line Width (LWD) and Area Ratios Of The Phases Indicated at the Right Most Columns of As-Prepared Samples.

Sample	IS(mm/s) (±0.02)	QS(mm/s) (±0.02)	LWD(m m/s) (±0.02)	B _{HF} (T) (±0.1)	Area (%) (±2)	Phase
K ₁	0.34	-0.23	0.33	37.8	66	Goethite
	0.35	-0.23	0.43	34.3	34	Goethite
K ₂	0.30	0.67	0.42	-	66	Ferrihydrite
	0.36	-0.19	0.36	50.7	20	Hematite
	0.38	-0.29	0.36	37.0	5	Goethite
	0.34	-0.25	0.35	33.4	9	Goethite
	0.31	0.68	0.43	-	100.0	Ferrihydrite
K ₄	0.30	0.67	0.40	-	40	Ferrihydrite
	0.36	-0.19	0.27	51.0	38	Hematite
	0.34	-0.24	0.45	37.4	13	Goethite
	0.26	-0.23	0.44	31.8	9	Goethite

Table S4. Raman Data for Annealed K₁, K₂, K₃ And K₄ Samples

Peak position in Raman spectra for various annealed samples											
400 °C				600 °C				800 °C			
				Peak positions cm ⁻¹							
K ₁	K ₂	K ₃	K ₄	K ₁	K ₂	K ₃	K ₄	K ₁	K ₂	K ₃	K ₄
220	224	224	224	229	229	229	229	229	231	229	229
290	290	290	290	294	294	294	294	297	297	297	297
400	407	407	407	407	407	407	407	411	411	411	411
500	500	500	500	500	500	500	500	493	500	493	493
609	611	611	611	611	611	611	611	612	612	612	612
								659	-	659	-
1305	1314	1314	1314	1316	1322	1316	1316	1322	1322	1322	1322
Literature (hematite)				Assignment							
226				A _{1g} (1) Fe-Osym str							
247				Eg(1) Fe-O sym.bend							
292				Eg(2)+Eg(3)Fe-Osym.bend							
406				Eg(4)Fe-O sym.bend							
495				A _{1g} (2)Fe-Osym str							
600, 613				Eg(5)Fe-O sym bend							
1320				2 nd harmonic vib							

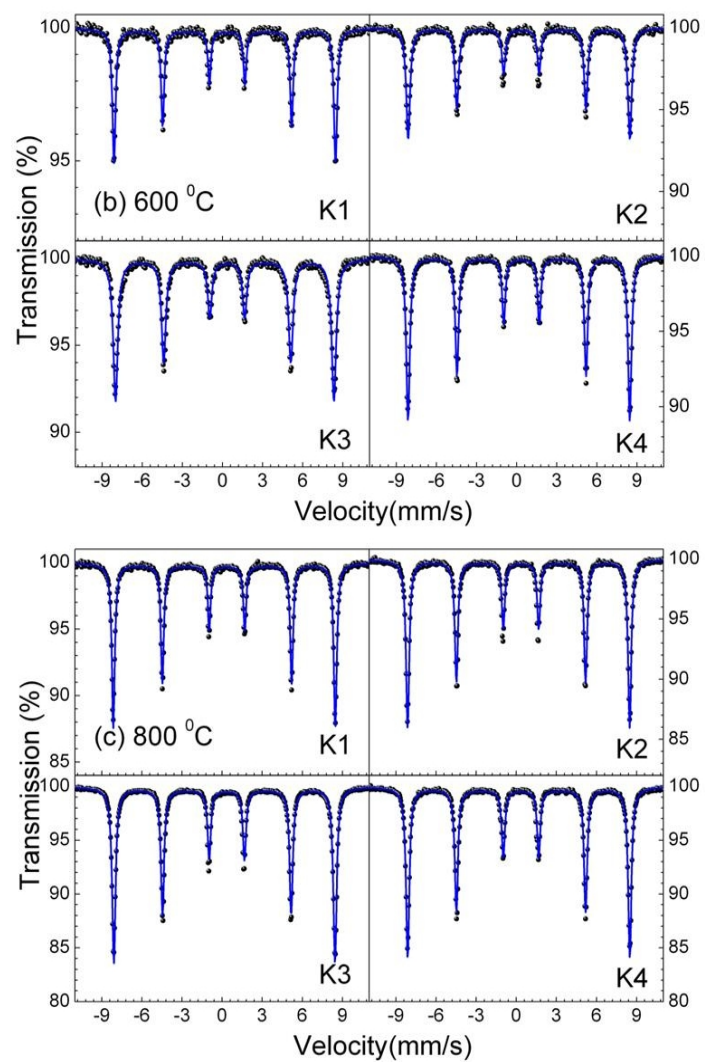


Fig. S1. Mössbauer spectra of the samples annealed at 600 °C and 800 °C.

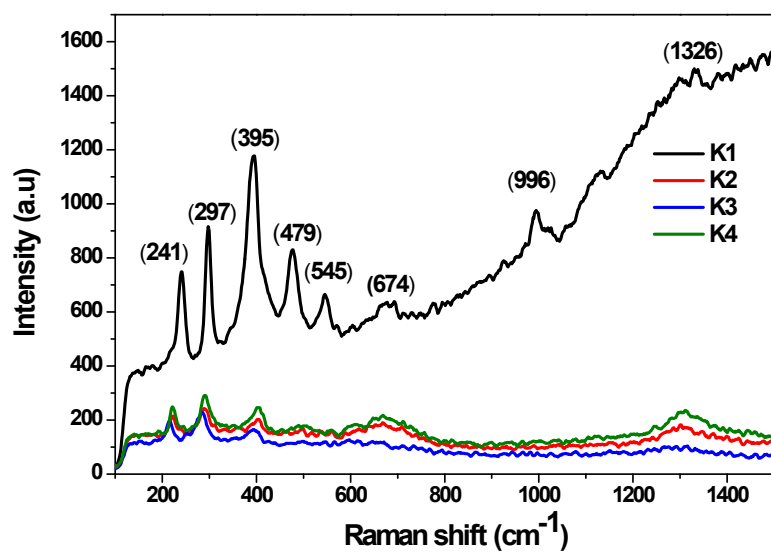


Fig. S₂. Raman spectra of as-prepared nano iron oxide samples.

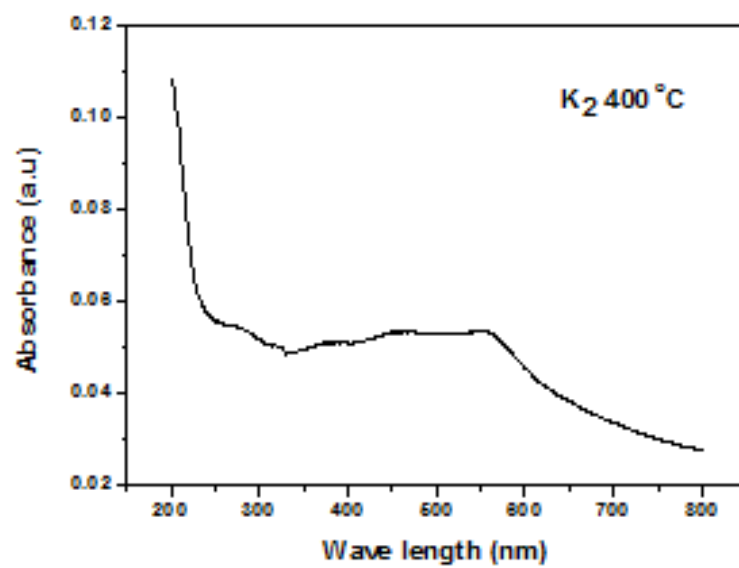


Fig. S₃. UV-Visible spectra of a typical annealed sample (K₂ sample annealed at 400 °C).