^{57}Fe Mössbauer, FT-IR and TEM Observations of Oxide Phases Precipitated from Concentrated Fe(NO$_3$)$_3$ Solutions

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Concentrated Fe(NO$_3$)$_3$ solutions (final Fe^{3+} concentration of 1 M) were completely or partially neutralized by addition of concentrated NaOH solution, and then aged at various temperatures and times. The oxide phases were investigated using Mössbauer spectroscopy, FT-IR spectroscopy and transmission electron microscopy. In a highly alkaline medium, single phase α-FeO(OH) was obtained by the aging of Fe(OH)$_3$ suspension at room temperature (RT) for 135 days. The Mössbauer spectrum of this sample at 298 K showed superposition of the central quadrupole doublet and a collapsing sextet, while at 80 K a sextet corresponding to α-FeO(OH) was recorded. In the highly alkaline medium, an amorphous material was obtained after heating the Fe(OH)$_3$ suspension at 333 K for 1 day. Spectroscopic investigations provided a certain evidence of the presence of α-Fe$_2$O$_3$ in this material. With prolonged time of heating, α-FeO(OH) appeared at 333 K and its fraction showed a tendency to increase. It was suggested that, in a highly alkaline medium, nucleation and crystallization of α-Fe$_2$O$_3$ occurred inside the amorphous gel, while α-FeOOH crystallized by a dissolution/reprecipitation mechanism. After neutralization of 90% of 1 M Fe(NO$_3$)$_3$ solu-

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tion, lemon-shaped α-Fe_2O_3 particles were produced at 398 K between 6 and 24 h. Interpretations of the spectroscopic and electron microscopic measurements are given.

INTRODUCTION

The term ***iron oxides*** is often used in the literature to describe the group of iron compounds with hydroxide, oxyhydroxide and oxide structures. These compounds have been the subject of numerous investigations from different standpoints. In the past, the attention of researchers was significantly focused on the precipitation of colloidal iron oxides. Because of their desirable colloidal and surface properties, iron oxides were used as model systems in a general study of the colloid and surface properties of metal oxides. The kinetics and mechanisms of the phase transformations of iron oxides in aqueous media were also investigated. The route of chemical synthesis has a significant influence on the chemical, structural and physical properties of iron oxides. For this reason, many researchers have investigated the relation between the experimental conditions of the synthesis of iron oxides and their properties. Understanding of these relations is very important for the practice, since iron oxides are widely used as pigments, catalysts, magnetic recording media, gas-sensors, *etc.*

Iron oxides can be precipitated from Fe(II)- or Fe(III)-salt solutions, as well as from solutions containing mixtures of Fe(II)- and Fe(III)-salts. The chemical and structural properties of iron oxides formed from $FeSO_4$ solutions are strongly dependent on the $[Fe^{2+}]/[OH^-]$ concentration ratio at the beginning of the precipitation process, on the rate of oxygenation, time of precipitation, temperature and kind of alkali (NH_4OH or $NaOH$).[1,2] Hydrolysis of urea at elevated temperature was used[3] for generation of OH^- ions during the process of precipitation from $FeSO_4$ solutions. Phase compositions of the precipitates were strongly dependent on the concentrations of $FeSO_4$ and urea, as well as on the rate of oxygenation. The analysis of precipitates showed the presence of different oxide phases, such as goethite, lepidocrocite, hematite and magnetite, and in one sample of a small amount of siderite. Significant differences in the Mössbauer spectrum of goethite were observed due to the very small particle size, the degree of crystallinity and/or different content of structurally bonded water. Precipitation from $FeSO_4$ solution can be utilized in the synthesis of δ-FeOOH.[4]

Iron oxides can be also produced by hydrolysis of acidic Fe(III)-salt solutions. This process generally involves several stages, such as the formation of Fe(III)-hydroxy complexes, hydroxy polymers (olation), oxo-bridges (oxolation) and, finally, a visible solid phase. On the basis of Mössbauer spectro-

scopic characterization of the hydrolytical products, Musić *et al.*[5] proposed a mechanism of precipitation by hydrolysis of acidic Fe(III)-salt solutions containing Cl^-, NO_3^- or SO_4^{2-} anions. Structural properties of precipitates, formed by hydrolysis of Fe^{3+} ions, in aqueous solutions at 393 K, were also investigated.[6] α-FeO(OH) and α-Fe$_2$O$_3$ were the solid hydrolytic products in solutions containing NO_3^- ions, while β-FeO(OH) and α-Fe$_2$O$_3$ were typical hydrolytic products in solutions containing Cl^- ions. In mixed Fe(NO$_3$)$_3$ + FeCl$_3$ solutions, the phase composition of the solid hydrolytic products was determined by the concentration of the dominant Fe(III)-salt. Microstructural properties of the precipitates, formed by hydrolysis of Fe^{3+} ions in Fe$_2$(SO$_4$)$_3$ solutions at 363 or 393 K, were also investigated.[7] Concentration regions of Fe$_2$(SO$_4$)$_3$ were determined for the precipitation of α-FeO(OH) or hydronium jarosite, H$_3$OFe$_3$(OH)$_6$(SO$_4$)$_2$. Superparamagnetic behavior was observed in the α-FeO(OH) particles. Hydrolysis of 0.1 M Fe$_2$(SO$_4$)$_3$ solution at 393 K generated the hydrolytic products H$_3$OFe$_3$(OH)$_6$(SO$_4$)$_2$ and basic sulphate, Fe$_4$(OH)$_{10}$SO$_4$. Musić *et al.*[8] also determined the concentration regions of NH$_4$Fe(SO$_4$)$_2$ for precipitation of goethite or ammonium jarosite (NH$_4$Fe$_3$(OH)$_6$(SO$_4$)$_2$) by forced hydrolysis of NH$_4$Fe(SO$_4$)$_2$ solutions at 363 or 393 K.

The size and morphology of the end-products of the hydrolysis of Fe^{3+} ions can be modified by addition of foreign ions and molecules to the precipitation system,[9,10] such as inorganic ions (phosphates, sulphates, *etc.*), organic ions (citrate, tartarate, oxalate, *etc.*) or big organic polymers. Ozaki *et al.*[11] described a procedure for the preparation of monodispersed spindle-type α-Fe$_2$O$_3$ particles by forced hydrolysis of FeCl$_3$ solutions. The particle shape was modified by a small addition of phosphate or hypophosphite ions at the beginning of the precipitation process. These α-Fe$_2$O$_3$ particles were used[12] to prepare spindle-type γ-Fe$_2$O$_3$ (maghemite) particles *via* Fe$_3$O$_4$ as the intermediate phase. Musić et al.[13] showed the influence of sodium polyanethol sulphonate on the morphology of β-FeO(OH) particles during FeCl$_3$ hydrolysis. In the absence of this polymer, different shapes of submicron particles, such as needles, stars, X- and Y-shaped particles were produced. When sodium polyanethol sulponate was added to the precipitation system, big spherical particles of β-FeO(OH), in the range of micron dimensions, were obtained.

Musić *et al.*[14] also investigated the effects of urotropin (hexamethylenetetramine) on FeCl$_3$ hydrolysis in aqueous solutions. During the short time (5 h) of FeCl$_3$ hydrolysis, it was observed that, with an increase of urotropin concentration, there were changes in particle size and morphology, associated with the amorphization of β-FeO(OH) particles. Recently, Mössbauer spectroscopy showed[15] that β-FeO(OH) particles considered amorphous in

our previous work[14] are actually of very small dimensions. After longer times (7 days), the chemical degradation of urotropin and changes of pH had the dominant effect on the phase composition of the precipitates.

The hydrolysis of Fe^{3+} ions was mainly investigated in relatively low concentration regions of Fe(III)-salt solutions. In the present work, we focus on the formation of crystalline iron oxides from concentrated $Fe(NO_3)_3$ solutions that were partially or completely neutralized with alkali. These reactions are of interest for their utilization in chemical technology. However, in these precipitation systems it is not so easy to control phase composition, particle size and morphology. In the present work, solid hydrolytic products were investigated using ^{57}Fe Mössbauer and FT-IR spectroscopies and transmission electron microscopy. Mössbauer spectroscopy has found important applications[16–18] in the investigations of iron oxides, as well as mixed metal oxides containing Mössbauer active nuclides.

EXPERIMENTAL

Chemicals of analytical purity and doubly distilled water were used. Precipitation of the amorphous hydroxide phase was performed using vigorous stirring of the precipitation system with an electrical stirrer. The experimental conditions for the precipitation of iron oxides are given in Table I. After a proper aging time, the solid phases were separated from the mother liquor using an ultra-speed centrifuge (operation range up to 20000 r.p.m.). The solid phase was subsequently washed with doubly distilled water and then dried.

TABLE I

Experimental conditions for preparation of samples

Sample	mL 2 M $Fe(NO_3)_3$	mL 6 M NaOH	mL 8 M NaOH	mL H_2O	Temp. (K)	Time of aging
S1	50		50		RT	135 d
S2	50		50		333	1 d
S3	50		50		333	3 d
S4	50		50		333	7 d
S5	50		50		333	10 d
S6	50		50		333	21 d
S7	25	22.5		2.5	398	6 h
S8	25	22.5		2.5	398	1 d

Key: RT = room temperature, h = hour, d = day

^{57}Fe Mössbauer spectra were recorded using the standard equipment and a ^{57}Co/Rh source. The spectra recorded were resolved into subspectra using the procedure of mathematical deconvolution.

The FT-IR spectra were recorded at room temperature using a Perkin-Elmer spectrometer (model 2000). IRDM (Infrared Data Manager) program, supplied by Perkin-Elmer, was used to process the spectra. The specimens were pressed into the KBr matrix.

The particle size and its morphology were monitored by transmission electron microscopy (TEM), (Opton, model EM–10). Before TEM observation, the powder was dispersed in doubly distilled water by ultrasound and then a drop of the dispersion was put on a copper grid, previously coated with a polymer film.

RESULTS AND DISCUSSION

^{57}Fe Mössbauer Spectroscopy

Figure 1 shows the Mössbauer spectra of sample S1. At 298 K, the spectrum showed superposition of a central quadrupole doublet and a sextet
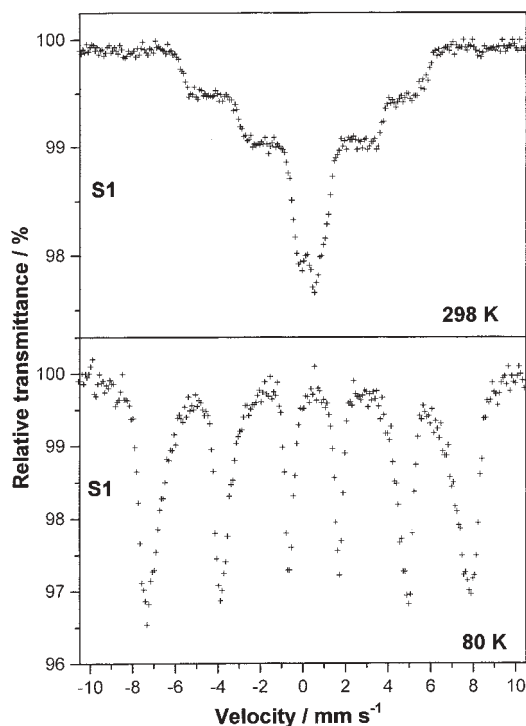

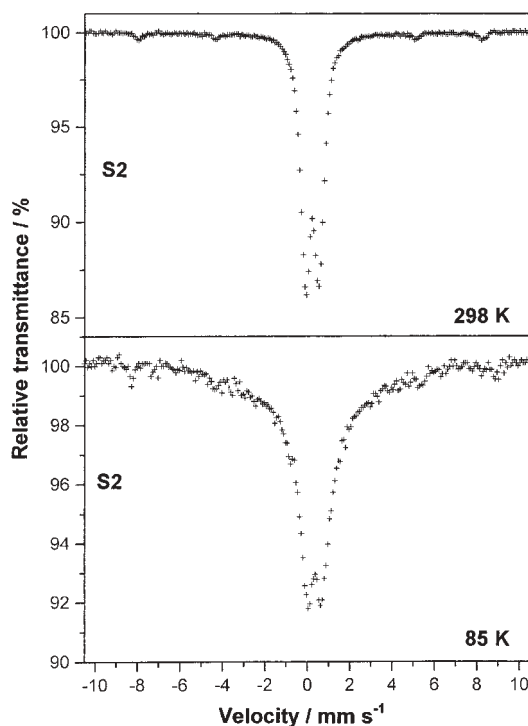Figure 1. Mössbauer spectra of sample S1, recorded at 298 and 80 K.

Figure 2. Mössbauer spectra of sample S2, recorded at 298 and 85 K.

with very broadened and poorly resolved spectral lines. No central quadrupole doublet is observed at 80 K; however, the sextet lines are broadened. These spectra correspond to goethite. Well-crystallized goethite generally exhibits a typical six-line hyperfine magnetic spectrum at RT with peak intensity ratios close to 3:2:1:1:2:3. Goethite precipitated from aqueous solutions shows, as a rule, significant differences in its Mössbauer spectrum.[19] These differences are influenced by the very small particle size, degree of crystallinity and/or different content of structurally bonded water. The superparamagnetic behavior of goethite particles can be used to determine their size. However, due to the combined effects (not only the effect of particle size), precaution is needed in the use of values of the hyperfine magnetic field to measure the particle size of goethite. At room temperature, the Mössbauer spectrum of precipitated goethite can vary from a well-defined sextet up to a quadrupole doublet.

In the present work, Mössbauer spectra were used to monitor the microstructural changes in the iron oxide powders (samples S2 to S6) obtained by the aging of freshly precipitated $Fe(OH)_3$ in a highly alkaline medium at 333 K. Figure 2 shows the Mössbauer spectra of sample S2, recorded at 298

and 85 K. At 298 K, the spectrum is characterized by a central quadrupole doublet (Δ = 0.64 mm s[-1]) and a very weak sextet. The positions of hyperfine magnetic lines correspond to hematite. A central quadrupole doublet is also present at 85 K; however, the presence of the collapsing sextet is also visible in the same spectrum. Mössbauer spectrum of sample S3 (Figure 3) showed significant changes in relation to sample S2 (Figure 2). At 298 K, superposition of the central quadrupole doublet and two sextets was recorded. The outer sextet corresponds to α-Fe$_2$O$_3$, while the inner sextet corresponds to α-FeO(OH). A central quadrupole doublet with broadened spectral lines is also present at 85 K. After 21 days of aging the freshly precipitated Fe(OH)$_3$ in a highly alkaline medium at 333 K, the mixture of α-Fe$_2$O$_3$ (outer sextet) and α-FeO(OH) (inner sextet) was obtained (Figure 4).

After neutralization of 90% of 1 M Fe(NO$_3$)$_3$ solution with NaOH and aging of the precipitation system at 398 K for between 6 h and 1 day (samples S7 and S8), as single phase α-Fe$_2$O$_3$ was produced. The pH of the liquid phase, after one day of aging, was in the acidic region (pH 2.24). The Mössbauer spectrum of sample S7 at 298 K, corresponding to α-Fe$_2$O$_3$, is
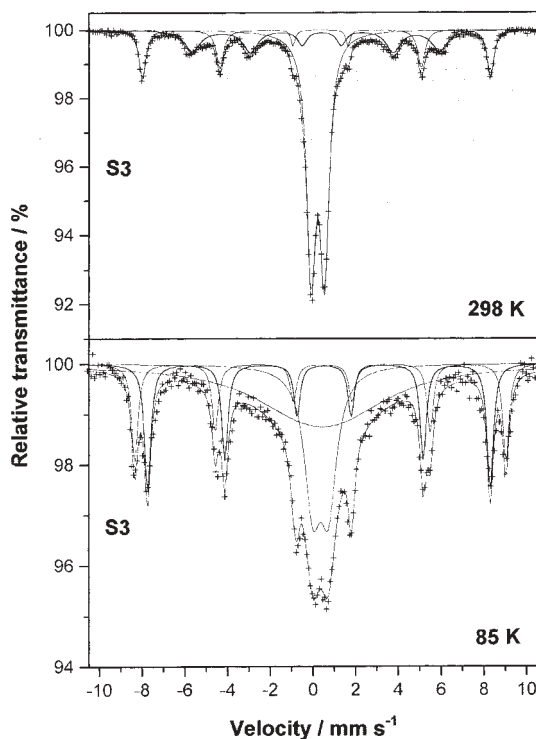

Figure 3. Mössbauer spectra of sample S3, recorded at 298 and 85 K.
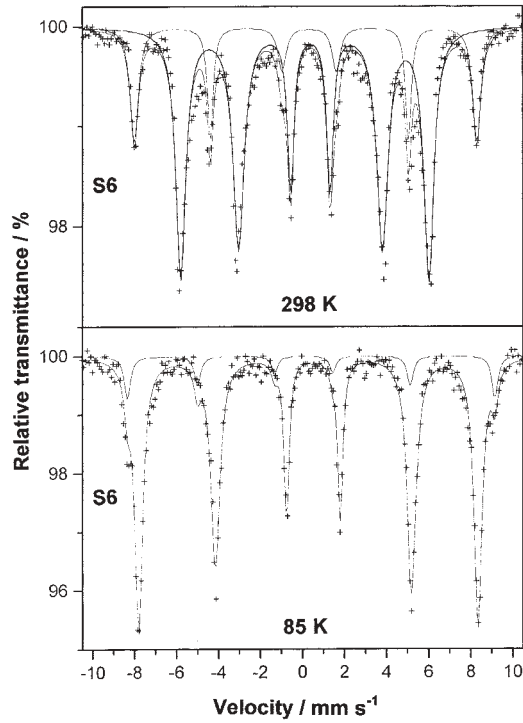
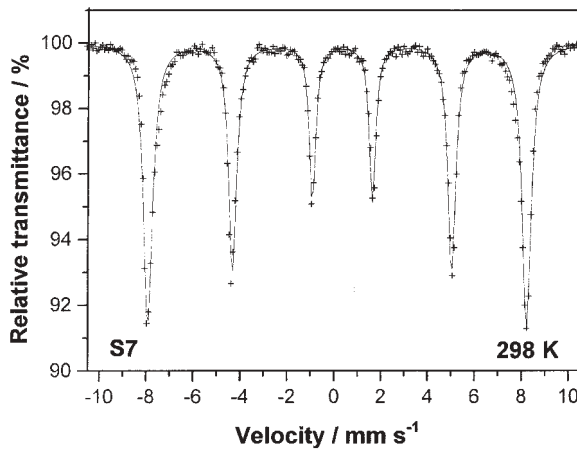Figure 4. Mössbauer spectra of sample S6, recorded at 298 and 85 K.

Figure 5. Mössbauer spectrum of sample S7, recorded at 298 K.

shown in Figure 5. Table II shows ^{57}Fe Mössbauer parameters of selected samples, obtained by the fitting of the recorded spectra.

TABLE II

[57]Fe Mössbasuer parameters of selected samples

Sample	Temp. (K)	Spectral component	δ (mm s^{-1})	Δ or E_q (mm s^{-1})	HMF (T)	A (%)
S2	298	Q	0.22	0.64		
	298	S				sextet of very small intensity
	85	Q	0.36	−0.00[a]		
	85	S				sextet of very small intensity
S3	298	Q	0.22	0.69		59
	298	S$_1$	0.25	−0.19	50.6	19
	298	S$_2$	0.26	−0.28	35.9	22
	85	Q$_1$	0.35	0.74		24
	85	Q$_2$	0.42	−1.82[b]		42
	85	S$_1$	0.39	−0.10	53.9	15
	85	S$_2$	0.39		49.7	19
S6	298	S$_1$	0.24	−0.18	50.7	24
	298	S$_2$	0.24	−0.27	36.7	76
	85	S$_1$	0.23		54.3	10
	85	S$_2$	0.40	−0.25	50.1	90
S7	298	S	0.24	−0.21	50.0	100
S8	298	S	0.25	−0.21	50.1	100

Key: δ = isomer shift relative to α-Fe, Δ = quadrupole splitting of doublet, E_q = quadrupole splitting of sextet, HMF = hyperfine magnetic field, A = area under the peaks, a = input parameter 0.846 mm s^{-1}, b = input parameter 0.64 mm s^{-1} given for the fitting procedure.

FT-IR Spectroscopy

Figure 6 shows the FT-IR spectrum of sample S1. The IR bands at 890 and 802 cm^{-1}, typical of α-FeO(OH), are visible. The presence of a band at 1635 cm^{-1} is due to molecular water. An intensive band at 1385 cm^{-1} is also visible, due to residual nitrates that have not been completely washed out from the precipitate. Peptization of this precipitate was pronounced during its washing. This is not surprising because the dimensions of the particles of sample S1 are very small, as shown by transmission electron microscopy.
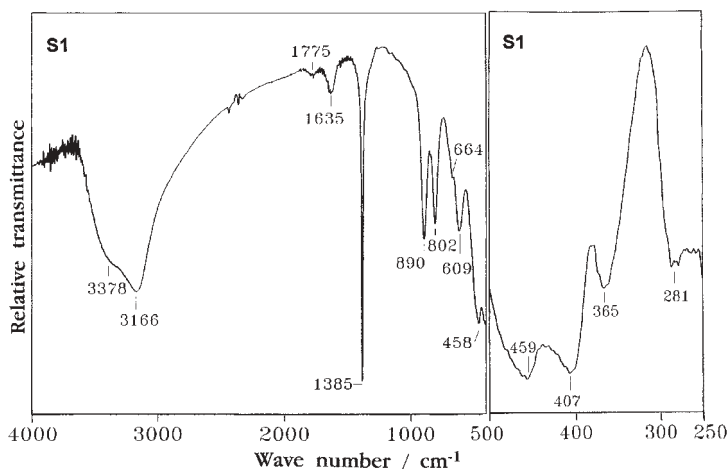
Figure 6. FT-IR spectrum of sample S1, recorded at room temperature.
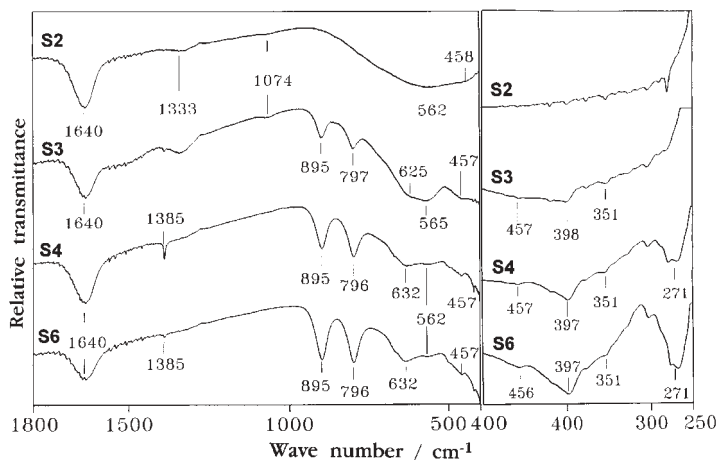

Figure 7. FT-IR spectra of samples S2, S3, S4 and S6, recorded at room temperature.

Figure 7 shows the FT-IR spectra of samples S2, S3, S4 and S6. The FT-IR spectrum of sample S2 is characterized by a very broad band showing two poorly resolved peaks at 562 and 458 cm^{-1}. These IR bands could be ascribed to α-Fe$_2$O$_3$; however, the characteristic band of α-Fe$_2$O$_3$ at 350–380 cm^{-1} is not visible in the far IR region. Taking into account the results of Mössbauer spectroscopy, it can be concluded that sample S1 contains a significant amount of superparamagnetic and/or amorphous oxide particles. After three days of aging the precipitate (sample S3), the IR bands at 895 and 797 cm^{-1}, typical of α-FeO(OH), and the bands at 565 and 457 cm^{-1},
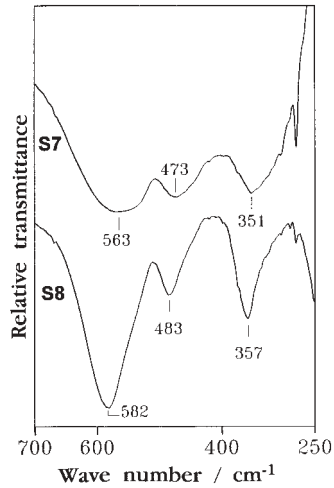
Figure 8. FT-IR spectra of samples S7 and S8, recorded at room temperature.

typical of α-Fe_2O_3, are visible. With further aging, there is an increase of the relative intensity of the IR bands corresponding to α-FeO(OH) and a decrease of the relative intensity of the IR bands corresponding to α-Fe_2O_3.

Figure 8 shows the FT-IR spectra of samples S7 and S8. The spectrum of sample S7 is characterized by bands at 563, 473 and 351 cm^{-1}, while the spectrum of sample S8 is characterized by bands at 582, 483 and 357 cm^{-1}. These bands can be ascribed to α-Fe_2O_3 in accordance with the Mössbauer results.
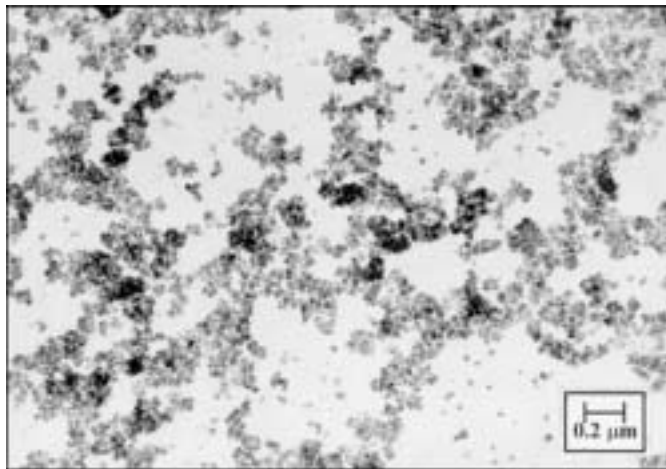

Figure 9. TEM microphotograph of sample S1.

Transmission electron microscopy

Transmission electron microscopic observations of the samples showed that the particle size and morphology of the oxide phases are very dependent on the experimental parameters. Figure 9 shows a TEM microphotograph of sample S1. Very small particles of α-FeO(OH) were obtained; the average size of these particles varies from 30 to 40 nm. Their phase composition was proved by Mössbauer and FT-IR spectroscopies. Figure 10 shows TEM microphotographs of samples (a) S2 and (b) S3. Sample S2 consists of big particles (agglomerates) of irregular shape, while sample S3 also shows sixpoint stars in addition to big particles. Needles and sixpoint stars are clearly visible for sample S6, as shown in Figure 11. Wang *et al.*[20,21] ob-
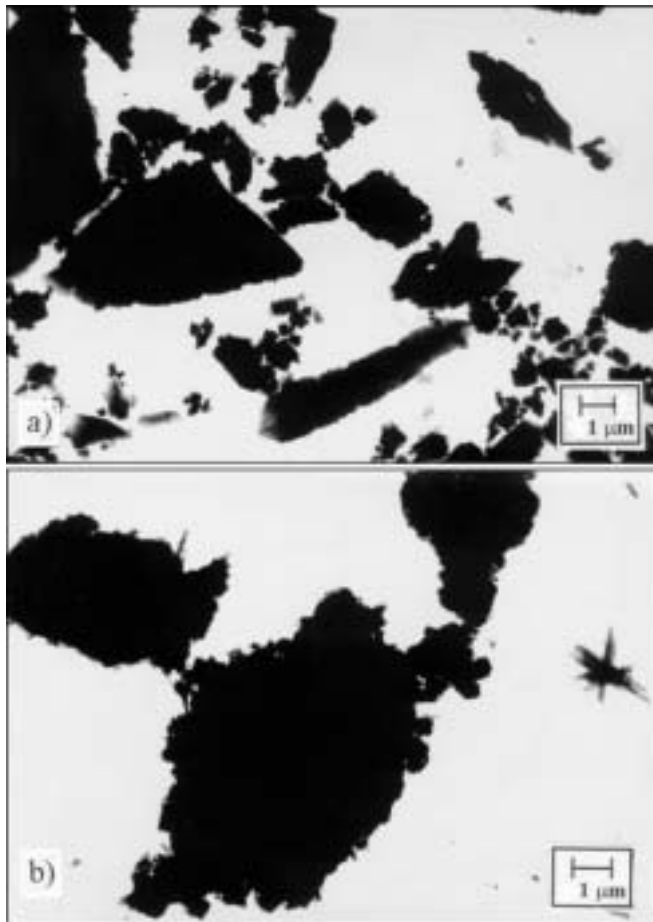

Figure 10. TEM microphotographs of samples (a) S2 and (b) S3.

Figure 11. TEM microphotograph of sample S6.

served α-FeO(OH) stars, the hydrolytic product of diluted $Fe(ClO_4)_3$ solution, at 343 K, and these authors suggested that α-FeO(OH) stars served as nuclei for the formation of α-Fe_2O_3. On the other hand, Wefers[22] suggested that α-Fe_2O_3 could act as nuclei for the formation of α-FeO(OH), but no evidence was given for this hypothesis. Evidently, the role of sixpoint stars in the formation of α-FeO(OH) and α-Fe_2O_3 will be the subject of dicussion in the future. Figure 12 shows TEM microphotographs of samples (a) S7 and (b) S8. The lemon-shape morphology of α-Fe_2O_3 particles is visible for both samples.

CONCLUSION

In a highly alkaline medium, at room temperature, the single phase of α-FeO(OH) precipitated from highly condensed $Fe(OH)_3$ gel. α-FeO(OH) particles were very small (average size of 30 to 40 nm), as shown by TEM, and this particle size influenced the shape of the corresponding Mössbauer spectra. For a short aging time (1 day) at 333 K, a small fraction of α-Fe_2O_3 and the dominant fraction of superparamagnetic and/or amorphous particles, probably based on the α-Fe_2O_3 structure, were obtained. FT-IR spectroscopy did not show any presence of α-FeO(OH) particles in the sample obtained after 1 day of aging at 333 K, thus suggesting that crystallization of α-Fe_2O_3 is favoured at the beginning of the precipitation process. However, once α-FeO(OH) is formed, its crystallization is accelerated. It was proposed in the literature (Refs. 23, 24) that two competitive mechanisms are present in a highly alkaline medium: (a) formation of goethite by disso-
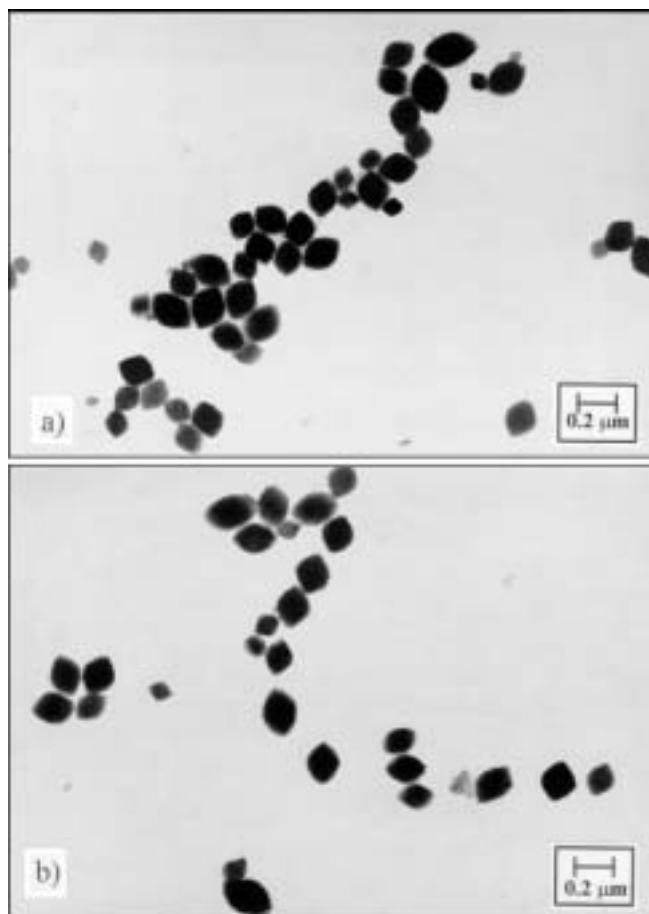
Figure 12. TEM microphotographs of samples (a) S7 and (b) S8.

lution/reprecipitation mechanism, and (b) nucleation and crystallization of hematite inside the amorphous $Fe(OH)_3$ gel. Under strong alkaline conditions, negatively charged complexes, $Fe(OH)_3^{(n-3)-}$, $n > 3$, may work as feeding material for the growth of α-Fe_2O_3. Since the surface charge of α-Fe_2O_3 is highly negative, it is difficult to overcome repulsion in the double layer under mild temperature conditions for these complexes. Also, diffusion of these complexes under static conditions is limited. For this reason, with a prolonged time of aging the α-$FeO(OH)$ fraction increased relatively to the α-Fe_2O_3 fraction, and the crystallization of α-Fe_2O_3 was not completed at 333 K, even after 21 days of aging. On the other hand, only α-Fe_2O_3 was produced at low pH, at 398 K after 6 hours. The forced hydrolysis of acidic $Fe(NO_3)_3$ solutions produced α-$FeO(OH)$ particles at the beginning of the

precipitation process, which transformed to α-Fe_2O_3 with prolonged hydrolysis by the dissolution/reprecipitation mechanism.[6] In the present case, the excess of $Fe(NO_3)_3$ solution, after neutralization of 90% of 1 M $Fe(NO_3)_3$ solution, increased the rate of α-Fe_2O_3 formation. Lemon-shaped α-Fe_2O_3 particles of relatively high uniformity were obtained.

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SAŽETAK

Primjena ^{57}Fe – Mössbauerove spektroskopije, FT-IR spektroskopije i transmisijske elektronske mikroskopije u proučavanju oksidnih faza istaloženih iz koncentriranih otopina Fe(NO$_3$)$_3$

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Kisele koncentrirane otopine Fe(NO$_3$)$_3$ bile su potpuno ili djelomično neutralizirane dodatkom koncentrirane otopine NaOH, a zatim su precipitacijski sustavi stareni u ovisnosti o temperaturi i vremenu. Oksidne faze proučavane su primjenom Mössbauerove spektroskopije, FT-IR spektroskopije i transmisijske elektronske mikroskopije. U jako alkalnom mediju, starenjem suspenzije Fe(OH)$_3$ pri sobnoj temperaturi kroz 135 dana dobiven je α-FeOOH kao jedina faza u talogu. Mössbauerov spektar tog uzorka pri 298 K pokazao je preklapanje centralnog kvadrupolnog dubleta i seksteta u nestajanju, dok je pri 80 K snimljen sekset čiji parametri odgovaraju α-FeOOH. U jako alkalnom mediju, nakon zagrijavanja suspenzije Fe(OH)$_3$ pri 333 K kroz 1 dan, dobiven je amorfni materijal, a spektroskopska ispitivanja ukazala su na moguću prisutnost α-Fe$_2$O$_3$ u amorfnom materijalu. S produženjem vremena zagrijavanja opaženo je nastajanje α-FeOOH pri 333 K, s tendencijom povećanju udjela spomenute faze u talogu. Na osnovi prikazanih rezultata zaključeno je da u jako alkalnom mediju dolazi do nukleacije i kristalizacije α-Fe$_2$O$_3$ unutar strukture amorfnog gela, dok α-FeO(OH) kristalizira mehanizmom otapanja/reprecipitacije. Pošto je neutralizirano 90% 1 M otopine Fe(NO$_3$)$_3$, starenjem precipitacijskog sustava pri 398 K između 6 i 24 sata dobivene su čestice α-Fe$_2$O$_3$ koje su pokazivale morfologiju limuna. U radu su dane interpretacije spektroskopskih mjerenja i elektronsko-mikroskopskih prikaza.