Research Article

Facile one-pot synthesis of water-soluble fcc FePt₃ alloy nanostructures

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Abstract

Processible FePt₃ alloy nanoparticles with sizes smaller than 50 nm open the avenue to novel magnetic sensor, catalytic and biomedical applications. Our research objective was to establish a highly scalable synthesis technique for production of single-crystalline FePt₃ alloy nanoparticles. We have elaborated a one-pot thermal decomposition technique for the synthesis of superparamagnetic FePt₃ nanoparticles (FePt₃ NPs) with mean sizes of 10 nm. Subsequent tiron coating provided water solubility of the FePt₃ NPs and further processibility as bidentate ligands enable binding to catalyst surfaces, smart substrates or biosensors. The chemical composition, structure, morphology, magnetic, optical and crystallographic properties of the FePt₃ NPs were examined using high resolution transmission electron microscopy, high-angle annular dark field-scanning transmission electron microscopy, scanning transmission electron microscopy-energy-dispersive X-ray spectroscopy mapping, Fourier transform infrared-attenuated total reflection, X-ray powder diffraction, X-ray photoelectron spectroscopy, vibrating sample magnetometry and UV–Vis absorption spectroscopy.

Keywords Iron-platinum alloy · FePt₃ nanoparticles · XPS · XRD analysis · Thermal decomposition route

1 Introduction

In recent years, bimetallic nanocrystals have received significant attention for nanoengineering, nanoelectronics and nanomedicine because of their particularly physical and chemical properties. Iron platinum alloy nanostructures in the L1₀, L1₁ and L1₂ phases were engineered for applications as being magnetic sensors, catalysts, drug delivery tools, and biomedical imaging [6, 14, 21, 27, 32, 39]. Highly ordered FePt alloy nanocomposites exhibit an encouraging potential for ultrahigh density magnetic

recording media and high-performance permanent magnets due to their uniaxial magnetocrystalline anisotropy, whereas chemically disordered FePt alloy nanoparticles promise applications in medical diagnostics (e.g. MPI, CT and MRI) [4, 14, 23]. FePt and FePt₃ alloys may crystallize in three different crystal structures: FePt₃ (L1₂, AuCu₃-type cubic), FePt (L1₀, AuCu-type tetragonal) and Fe₃Pt (L1₂, AuCu₃-type cubic), the magnetic properties of which depend on the synthesis procedure [2, 8, 9, 14]. On the other hand, in the respective chemically disordered high-temperature FePt phase, the Fe and Pt atoms are randomly

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distributed over the lattice sites and thus, exhibit isotropic magnetic behavior [27, 35]. The most common synthesis routes for preparing FePt, Fe₃Pt or FePt₃ nanoparticles are the thermal decomposition technique and the polyol synthesis [21, 23]. The face centered cubic (fcc) FePt phase can be transformed into the face centered tetragonal (fct) phase by high temperature annealing. Unfortunately, thermal annealing gives rise to agglomeration of FePt alloy nanocrystals which is accompanied by the emergence of undesired shapes and broad size distributions [13, 21]. The formation of FePt alloy nanocrystals depend on the growth from two different metals (Fe and Pt) at distinct reaction temperatures [21]. Sun et al. synthesized FePt alloy nanocrystals by thermally decomposing Fe(CO)₅ and Pt(acac)₂ in the presence of the reducing agent oleyl amine and the surfactant oleic acid. Cytotoxic Fe(CO)₅ can be substituted by iron(III) acetylacetonate or iron(III) oleate. Moreover high-boiling temperature solvents such as di-*n*-octyl ether or 1-octadecene are required for thermal decomposition synthesis routes. Polyol synthesis techniques are based on solvents such as tetraethylene glycol (TEG) and ethylene glycol (EG) which simultaneously serve as solvent and reducing agent for the metallic precursor [23, 33]. One major drawback of these demanding synthesis techniques is the impossibility to adjust the growth of FePt alloy nanoparticles within a uniform crystal phase (fcc or fct).

In the present work, we have elaborated a straightforward synthesis route for chemically disordered fcc FePt₃ nanocrystals. This one-pot synthesis is highly scalable for the preparation of superparamagnetic FePt₃ nanoparticles (FePt₃ NPs) for future electronic and biomedical applications. The one-pot synthesis technique comprises the preparation of a Pt(II) precursor solution and subsequent formation of FePt₃ NPs. Subsequent ligand exchange with tiron provided dispersion stability and water solubility of the FePt₃ NPs and moreover, facilitates their processing to thin magnetic films and their application as MRT/CT contrast agents in biomedical imaging. The tiron-coated FePt₃ NPs were characterized by determining the average size, size distribution, morphology, crystallographic properties, colloidal stability, magnetic and optical properties. High resolution transmission electron microscopy (HRTEM), high-angle annular dark field-scanning transmission electron microscopy (HAADF-STEM), scanning transmission electron microscopy-energy-dispersive X-ray spectroscopy (STEM-EDX) mapping, Fourier transform infrared-attenuated total reflection (FTIR-ATR), X-ray powder diffraction (XRD), X-ray photoelectron spectroscopy (XPS), UV-Vis absorption spectroscopies were employed as microscopy and spectroscopy characterization techniques. The magnetic properties were examined using vibrating sample magnetometry.

2 Experimental

2.1 Materials

Dihydrogenhexachloroplatinate hexahydrate (H₂Cl₆Pt × 6H₂O, ≥ 99.9%, Alfa Aesar), ethanol (≥ 99.8%, Carl-Roth), oleic acid (90%, Sigma-Aldrich), 1-octadecene (90%, Sigma-Aldrich), tert-butylamine borane (TBAB, 97%, Sigma-Aldrich), chloroform (≥ 99.9%, Sigma-Aldrich), isopropanol (≥ 98%, Sigma-Aldrich), tiron (97%, Sigma-Aldrich), oleylamine (80–90%, Acros Organics), *n*-hexane (95%, VWR), iron(III) acetylacetonate (Fe(acac)₃, 97%, Fluka) were used for the synthesis of the FePt₃ NPs. All chemicals are commercially available and were used without further purification. In all syntheses, ultrapure milli-Q water (18 MΩ) was used.

2.2 Preparation of the Pt(II) precursor solution

40.0 mg H₂Cl₆Pt × 6 H₂O (0.077 mmol) were dissolved in 4.00 mL chloroform and 4.00 mL oleylamine (12.2 mmol) were added. The precursor solution was stirred for 10 min under ambient conditions. 70.0 mg TBAB (0.804 mmol) and 1000 µL oleylamine (1.22 mmol) in 1000 µL chloroform were mixed by sonication and added to the precursor solution. The mixture was stirred for 1 h at room temperature.

2.3 Synthesis of FePt₃ NPs

Under argon atmosphere, the Pt(II) precursor solution in chloroform was added to a solution containing 15.0 mL 1-octadecene, 1.00 mL oleic acid (3.15 mmol) and 1.00 mL oleylamine (3.04 mmol). The reaction mixture was stirred for 30 min at 120 °C. Then 90.0 mg Fe(acac)₃ (0.255 mmol) dissolved in 5.00 mL 1-octadecene were injected into the hot solution. The reaction mixture was heated to 320 °C and boiled under reflux over 2 h. After cooling down to room temperature the reaction mixture was exposed to air for 1 h. The FePt₃ NPs were precipitated by the addition of 60 mL isopropanol and collected by centrifugation (11,000 rpm, 10 min). Afterwards the FePt₃ NPs were washed with ethanol and centrifugation and redispersed in 10 mL *n*-hexane for storage.

2.4 Tiron-coating of the FePt₃ NPs

100 mg tiron (0.302 mmol) in 1.50 mL milli-Q water were added to a dispersion of 10 mg FePt₃ NPs in 2.50 mL *n*-hexane. This biphasic *n*-hexane/water NP mixture was sonicated for 1 h. Then *n*-hexane was removed using a

rotary evaporator. The tiron-coated FePt₃ NP dispersion formed a stable aqueous solution.

2.5 Microscopic and spectroscopic characterization

HRTEM images were obtained using a Titan3 Themis 300 (FEI Company, Netherlands) equipped with a high-brightness field-emission gun (X-FEG), a monochromator system and two Cs-correctors (probe and image side) (CEOS GmbH, Germany). The microscope operates at an acceleration voltage of 200 kV. The morphology and elemental composition of the FePt₃ NPs were examined by means HAADF-STEM using the Z-contrast. The STEM-EDX mappings were obtained using a Super-X detector for EDX spectroscopy. The HRTEM images were recorded using a FEI Ceta 16 M 4 K CMOS (complementary metal-oxide semiconductor) camera. HRTEM samples were prepared by dropping the FePt₃ NP dispersions on Cu grids coated with an ultrathin carbon film layer.

XRD patterns of FePt₃ NPs powder samples were measured at room temperature using a Philips/Panalytical X-Pert Pro equipped with a copper radiation, ($K_{\alpha 1} = 0.154056$ nm and $K_{\alpha 2} = 0.154439$ nm). The XRD measurements were repeated 10 times. In addition, the Rietveld profile fitting of the XRD pattern of the FePt₃ NP sample was recorded by mounting in a silicon wafer to the XRD instrument at room temperature. Rietveld fitting was obtained by means of *MAUD* (Materials Analysis Using Diffraction) [15–18].

FTIR transmission spectra of the FePt₃ NPs were recorded on an IR Prestige-21 spectrometer (Shimadzu, Japan) equipped with an attenuated ATR setup with a diamond/ZnSe crystal plate (Miracle ATR, Pike Technologies, U.S.A.).

XPS was conducted on a Quantera II microprobe (Physical Electronics Inc., Chanhassen, MN, USA) under ultrahigh vacuum conditions. The powder samples were mounted on a double-sided sticky tape to the sample holder. Monochromated Al K α x-rays (1486.6 eV, source at 15 kV and 50 W) were focused on 200 $\times$ 200 μm^2 homogeneously covered spots of the powder samples. The mostly non-conducting samples were neutralized by Ar⁺ ions and electrons. The pure Pt and pure Fe reference samples were presputtered to clean the surface. Photoelectrons were detected at an analyzer pass energy of 280 eV and 140 eV and the number of sweeps for signal averaging were 3 and 25 for survey and high-resolution spectra, respectively. A peak at 284.8 eV associated with C 1s core-level was taken as a reference for the binding energy scale of the FePt₃ NP samples.

The magnetic properties of the FePt₃ NPs were analyzed using a Quantum Design VSM-MPMS SQUID

magnetometer at room temperature under an external magnetic field varied between -5T and $+5\text{T}$.

3 Results and discussions

3.1 HRTEM/HAADF-STEM/STEM-EDX analysis

According to the HRTEM image in Fig. 1a the FePt₃ NPs are single crystalline and flower-like nanostructures with a mean size of 10 nm. Furthermore, the HRTEM image (Fig. 1a, insert) exhibits lattice fringe spacings with 0.194 nm which identifies the (200) lattice plane of the face centered cubic (fcc) phase. Figure 1b shows the HAADF-STEM image of the FePt₃ NPs. The STEM-EDX mapping images visualize the homogeneous elemental distribution of Pt (green) and Fe (red) in the FePt₃ NPs, and thereupon revealing their quasi-alloy FePt₃ composition (Fig. 1c–e).

3.2 XRD analysis

The crystal structure of the FePt₃ NPs was elucidated by performing a powder XRD analysis. The XRD pattern in Fig. 2 displays prominent reflection peaks at 40.25, 46.75, 68.41, and 82.34 which are assigned to the (111), (200), (220), (311) planes of the disordered fcc FePt₃ phase with the space group $Fm\bar{3}m$ (ICDD Ref: 001-089-2050) [29]. The Rietveld fit of the XRD data (Fig. 3) was obtained for the fcc phase of FePt₃ and yielded the lattice constant $a = 0.3852$ nm [15–18]. The superlattice reflections (001) and (110) indicate a partially $L1_2$ -ordered phase of FePt₃. The average NP diameter was calculated to be 10 nm using the Debye–Scherrer equation [11], which agrees with the HRTEM analysis.

3.3 FTIR-ATR spectra

Oleic acid and oleylamine were used as stabilizing agents for the high-temperature synthesis of the FePt₃ NPs. In the FTIR-ATR spectrum of the FePt₃ NPs (Fig. 4, black solid line) the peaks at 2853 cm^{-1} and 2925 cm^{-1} were assigned to the symmetric and asymmetric CH₂ stretching modes of oleic acid and oleylamine [5]. Water solubility and further processability of the FePt₃ NPs were achieved by tiron coating through ligand exchange. The binding modus of tiron was elucidated by analyzing the FTIR-ATR spectrum of tiron-coated FePt₃ NPs (Fig. 4, red solid line). This FTIR-ATR spectrum exhibits the characteristic vibrational bands of tiron being bound via the catechol group at the FePt₃ NP surface (Fig. 5). The broad band in the region of 3100–3600 cm^{-1} arose from the symmetric O–H stretching vibrations of water. The vibrational peak at 1250 cm^{-1} and 1190 cm^{-1} are assigned to

Fig. 1 **a** HRTEM image of the FePt₃ NPs, **b** HAADF-STEM image of FePt₃ NPs, **c** STEM-EDX elemental mapping images showing the distribution of Pt (green) (Pt K-edge), **d** STEM-EDX elemental mapping images showing the distribution of Fe (red) (Fe K-edge), **e** merged image of the FePt₃ NPs of the elemental distribution of Fe (red) and Pt (green)

Fig. 2 XRD pattern of the FePt₃ NPs

the asymmetric S=O stretching vibration, and the band at 1031 cm⁻¹ is ascribed to the symmetric O=S=O stretch of tiron [28]. The peak at 630 cm⁻¹ identifies the C–S stretching vibration. The peaks at 1596 cm⁻¹, 1459 cm⁻¹ and 1426 cm⁻¹ refer to the carbon–carbon stretching vibrations in the aromatic ring of the tiron [28].

3.4 XPS analysis

Figure 6 exhibits the Fe 2*p* spectral region, namely the Fe 2*p*_{3/2} and Fe 2*p*_{1/2} multiplet for the FePt₃ NPs and a pure metal Fe. The Fe 2*p* spectral region consists of two peaks at approximately 712.8 eV and 726.8 eV. The higher binding energy of the multiplet as compared to the metal reference (707.0 eV and 720.0 eV) and relatively high full width at half maximum of approximately 7 eV for the peak at lower binding energy is often assigned to the presence of a multitude of different iron species in positive oxidation states such as iron oxides [19, 22, 25, 26, 36, 37]. Qualitatively, the low binding energy tail of the NPs Fe 2*p*_{3/2} component overlaps with the respective signal of the pure metal Fe at 707.0 eV. This effect is assigned to the presence of iron species that show only slightly higher binding energy as compared to metal Fe like for example Fe–Pt alloys [20, 30].

Figure 7 shows the Pt 4*f* region comprising the Pt 4*f*_{7/2} and Pt 4*f*_{5/2} multiplet. The Pt 4*f*_{7/2} peak for the NPs is evidenced at 0.2 eV towards lower binding energies as compared to the pure metal Pt at 71.2 eV. The effect,

Fig. 3 The Rietveld profile fitting to the XRD pattern of the FePt₃ NPs

Fig. 4 FTIR-ATR transmission spectra of the as-synthesized FePt₃ NPs (black solid line) and tiron-coated FePt₃ NPs (red solid line)

which is just of the order of the uncertainty from the peak referencing of the spectrum, points to the formation of an interfacial Fe–Pt alloy and electron transfer at the interface towards the more electronegative Pt [20,

30]. The peak at higher binding energies (75–78 eV) is assigned to the presence of precursor ions in the spectrum of the FePt₃ NPs.

3.5 UV–Vis absorption spectroscopy

Figure 8 depicts the UV–Vis absorption spectrum of tiron-coated FePt₃ NPs in water.

The UV–Vis absorption spectrum of tiron-coated FePt₃ NPs displays a broad absorption band around 650 nm (Fig. 8) which is due to electron transfer from Pt to the antibonding π -molecular orbitals of tiron [3, 10, 31, 38]. Valence tautomerization of tiron (OH to =O) of tiron may also contribute to the emergence of these broad absorption band (Fig. 9) [1, 3, 7, 10, 12, 24, 31, 34, 38].

Figure 10 presents the magnetization curve of a powder sample of tiron-coated FePt₃ NPs at room temperature under an external magnetic field varied between –5T and +5T. The lack of any hysteresis loop verifies superparamagnetism of the FePt₃ NPs [29]. The M_s value of the FePt₃ NPs is 35.90 emu/g. The observed superparamagnetism reflects the chemical disorder of the FePt₃ NPs.

Fig. 5 Tiron-coating of the FePt₃ NPs

Fig. 6 Fe 2p XPS spectrum of the FePt₃ NPs

Fig. 8 UV-Vis absorption spectrum of tiron-coated FePt₃ NPs in water

Fig. 7 Pt 4f XPS spectrum of the FePt₃ NPs

3.6 Formation of FePt₃ alloy nanoparticles

The FePt₃ alloy nanoparticles were synthesized via the thermal decomposition route at 320 °C using Fe(acac)₃ and oleylamine stabilized Pt²⁺ cations as Pt source. The formation of the chemically disordered FePt₃ nanoparticles requires the co-reduction of Fe³⁺ and Pt²⁺ cations. Since Pt is a noble metal and Fe is a transition metal, the Pt²⁺ cations are faster reduced to Pt⁰ which sparks Pt nucleation. The Pt nuclei presumably catalyze the oleylamine-induced reduction of Fe³⁺ to Fe⁰ [2]. Subsequent nanoparticle growth occurs via the incorporation of Fe atoms into the Pt rich nuclei. The final size and stabilization are

adjusted by the precursor/oleyl acid (oleylamine) ratio. The final stoichiometric composition and structure of the FePt₃ alloy nanoparticles follow from the reaction temperature as well as from the Fe and Pt precursor concentrations.

4 Conclusions

Our objective was to develop a highly scalable wet-chemistry technique for the synthesis of processible, superparamagnetic FePt₃ NPs for versatile applications in electronics, catalysis and medicine. We elaborated a facile one-pot thermal decomposition procedure for the preparation of 10 nm-sized FePt₃ nanocrystals which exhibit superparamagnetism. A straightforward subsequent ligand exchange procedure yielded watersoluble tiron-coated FePt₃ NPs suited for processing to thin magnetic films or for application as CT/MRI contrast agents.

The stoichiometry and structural properties of the FePt₃ NPs were elucidated using HRTEM, HAADF, STEM-EDX, XPS and XRD. We could unambiguously show that FePt₃ NPs with a mean size of 10 nm crystallize in the chemically disordered fcc phase with the space group Fm $\bar{3}$ m. The chemical disorder substantiated the superparamagnetism of the FePt₃ NPs as being evident from the hysteresis-less magnetization curve. The optical properties and ligand binding modi of tiron-coated FePt₃ NPs were examined using UV-Vis absorption and FTIR transmission spectroscopy. The aqueous FePt₃ NPs solution exhibits a broad absorption band around 650 nm,

Fig. 9 pH dependency of tiron in aqueous solution **Fig. 10** Magnetization curve of tiron-coated FePt₃ NPs

which is ascribed to an electron transfer from the Pt atoms to antibonding π -molecular orbitals of tiron which binds through its catechol moiety to the FePt₃ surface. This particular optical property of tiron-coated FePt₃ NPs demonstrates the high catalytic potential of FePt₃.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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