Investigation of Commercial Iron Oxide Nanoparticles: Structural and Magnetic Property Characterization

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ABSTRACT: Magnetic nanoparticles (MNPs) have been extensively used as tiny heating sources in magnetic hyperthermia therapy, contrast agents in magnetic resonance imaging, tracers in magnetic particle imaging, carriers for drug/gene delivery, etc. There have emerged many MNP/microbead suppliers since the past decade, such as Ocean NanoTech, Nanoprobes, US Research Nanomaterials, Miltenyi Biotec, micromod Partikeltechnologie GmbH, nanoComposix, and so forth. In this paper, we report the physical and magnetic characterizations on iron oxide nanoparticle products from Ocean NanoTech. Standard characterization tools such as vibrating-sample magnetometry, X-ray diffraction, dynamic light scattering, transmission electron microscopy, and zeta potential analysis are used to provide MNP customers and researchers with an overview of these iron oxide nanoparticle products. In addition, the dynamic magnetic responses of these iron oxide nanoparticles in aqueous solutions are investigated under low- and high-frequency alternating magnetic fields, giving a standardized operating procedure for characterizing the MNPs from Ocean NanoTech, thereby yielding the best of MNPs for different applications.

1. INTRODUCTION

Magnetic nanoparticles (MNPs) are nanomaterials with sizes between 1 and 100 nm. Due to their large surface-to-volume ratio and tunable magnetic properties, MNPs have emerged as one of the most important nanomaterials in magnetic, chemical, and biomedical applications. The surface of the MNPs can be functionalized with various coatings from inorganic coatings such as silica1 and carbon2 to organic coatings such as polyethylene glycol3 and dopamine.4 Compared to non-magnetic particles, MNPs can be manipulated by an external magnetic field without any physical contact, which leads to various applications such as drug delivery5 as well as the separation and concentration of certain molecules.6 Under an alternating magnetic field, MNPs can induce a localized temperature increase at the target spot, which makes them promising candidates for hyperthermia applications.7 Under an external magnetic field, MNPs can generate stray fields. By integrating with various magnetic sensors such as magnetoresistance sensors,8,9 hall sensors,10,11 nuclear magnetic resonance (NMR) sensors,11 magnetic resonance imaging (MRI),12 and magnetic particle spectroscopy (MPS),13 MNPs can also serve as magnetic markers in diagnostic applications.

To date, MNPs with various sizes and surface coatings have been successfully commercialized and are available in many companies such as Ocean NanoTech (San Diego, USA), Nanoprobes (New York City, USA), US Research Nanomaterials (Houston, USA), Miltenyi Biotec (Bergisch Gladbach, Germany), micromod Partikeltechnologie GmbH (Rostock, Germany), nanoComposix (San Diego, USA), and so forth.

For these aforementioned applications, the quest for a high magnetic moment, uniform size distribution, and colloidal stability of MNPs has pushed the development of various nanoparticle manufacturers. In this paper, we first characterized the magnetic and physical properties of single-core, differently sized iron oxide nanoparticle products from Ocean NanoTech using vibrating-sample magnetometry (VSM), X-ray diffraction (XRD), dynamic light scattering (DLS), transmission electron microscopy (TEM), and zeta potential analysis (summarized in Table 1). In addition, we give application-oriented assessments on these MNP products using a home-built MPS system. Practical suggestions on the applications of these iron oxide nanoparticles with varying core sizes are given at the end of this paper to maximize the use of them.

2. RESULTS AND DISCUSSION

2.1. Magnetic Properties of SHA Series MNPs. The hysteresis curves of SHA series MNPs are recorded by VSM under field ranges of −5000 to 5000, −500 to 500, and −200 to 200 Oe. The magnetic moment per volume of MNP suspension is averaged over 25 μL of the MNP sample and plotted in Figure 1a−c, with SHA-5, SHA-10, SHA-15, and...
SHA-20 being superparamagnetic. We also observed coercivities from SHA-25 and SHA-30. Due to the varying particle concentrations in SHA series MNP products (listed in Table 1), the magnetic moment per volume is not comprehensive to represent the magnetic property of each MNP. In addition, the magnetic moment per particle is also summarized in Figure 1d–f, with SHA-30 showing the highest magnetic moment per particle, followed by SHA-25, SHA-20, SHA-15, SHA-10, and SHA-5 showing the lowest magnetic moment per particle. Under a field strength of 5000 Oe, the specific magnetizations (calculated from the magnetic moment per gram of Fe, unit: emu/g) from the highest to the lowest are SHA-15 > SHA-20 > SHA-5 > SHA-25 > SHA-30 > SHA-10, as shown in Figure 1h,i. Note that the magnetization of SHA-5 is not saturated at 5000 Oe, while the remaining SHA series MNPs are saturated. Thus, we use specific magnetization instead of

Table 1. Physical and Magnetic Properties of SHA Series MNPs

| sample | average size (nm) ± SD | zeta potential (mV) ± SD | specific magnetization (emu/g Fe) | specific magnetization (emu/g) | magnetic moment per particle (emu/particle) | material          |
|--------|------------------------|--------------------------|----------------------------------|--------------------------------|---------------------------------------------|-------------------|
| SHA-5  | 10.46 ± 3.88           | −0.03 ± 0.005            | 63.84                            | −44.69                         | 1.54 × 10⁻¹⁷                             | γ-Fe₂O₃, Fe₃O₄   |
| SHA-10 | 18.07 ± 4.72           | 5.03 ± 0.07              | 42.64                            | −29.85                         | 8.23 × 10⁻¹⁷                             | γ-Fe₂O₃, Fe₃O₄   |
| SHA-15 | 20.69 ± 6.31           | 7.66 ± 0.05              | 83.44                            | −58.41                         | 5.13 × 10⁻¹⁶                             | γ-Fe₂O₃, Fe₃O₄   |
| SHA-20 | 27.56 ± 11.29          | −0.41 ± 0.33             | 78.08                            | −54.66                         | 1.18 × 10⁻¹⁵                             | γ-Fe₂O₃, Fe₃O₄   |
| SHA-25 | 28.28 ± 10.38          | 1.15 ± 0.49              | 55.28                            | −38.70                         | 1.58 × 10⁻¹⁵                             | γ-Fe₂O₃, Fe₃O₄   |
| SHA-30 | 32.60 ± 12.17          | −0.69 ± 0.04             | 51.12                            | −35.78                         | 2.50 × 10⁻¹⁵                             | γ-Fe₂O₃, Fe₃O₄   |

The average hydrodynamic sizes of SHA series MNPs are based on number-weighted DLS distribution. The specific magnetization (emu/g) is calculated under a 5000 Oe field. The specific magnetization (unit: emu/g, emu/g) and magnetic moment per particle (unit: emu/particle) are calculated under a 5000 Oe field based on the nanoparticle concentrations provided by Ocean NanoTech. The concentration of Fe is 5 mg/mL for all SHA series MNPs, while the concentrations of nanoparticles are 34.5, 4.3, 1.35, 0.55, 0.29, and 0.17 nmol/mL for SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30, respectively. The specific magnetization (emu/g), that is, the magnetic moment per gram of iron oxide nanoparticle is calculated by assuming that iron holds 70% of the nanoparticle weight.
saturation magnetization here. Due to the surface spin canting effect (also called the magnetically anomalous shell or magnetically dead layer) of nanoparticles, the specific magnetizations of MNPs are always lower than that of the bulk material. Herein, the specific magnetizations of SHA series MNPs are lower than the ideal values of bulk γ-Fe₂O₃ (60–80 emu/g) and Fe₃O₄ (92–100 emu/g) materials.¹⁵⁻¹⁹

Since the magnetic moment of each particle increases with the cube of its magnetic core diameter (or radius), in the ideal case where these SHA series MNPs show similar specific magnetization, the magnetic moment per particle should show a similar trend to the core diameter. However, we do not see the trend of increasing specific magnetizations (under a 5000 Oe field) in SHA series MNPs as the magnetic core size increases, which might be caused by the insufficient oxygenation for larger magnetic core sizes. Thus, as the insufficient oxygenation effect dominates, the SHA-15 MNPs show the highest specific magnetization, and it decreases with increasing core size as seen in Table 1.

The crystal structure of SHA series MNPs is characterized via XRD (Bruker D8 Discover 2D), as shown in Figure 2. It is observed that Fe₂O₄ and γ-Fe₂O₃ are the main phases in SHA series MNPs. There are also several diffraction peaks from the solution denoted by the blue dashed lines in Figure 2. The sharp diffraction peaks (labeled by black diamonds) come from the chemicals in the MNP buffer (NaCl, KCl, Na₂HPO₄, KH₂PO₄, etc), and the peaks labeled by black rounds come from the Si/SiO₂ substrate. The full width at half-maximum of the diffraction peaks is wider for the MNPs compared to their bulk counterparts. The broadening effects are due to the decreased grain size for nanoparticles.

![Figure 2. XRD patterns of SHA series MNPs. The powder diffraction files of FeO, Fe₂O₃, α-Fe₂O₃, and γ-Fe₂O₃ are added for references.](https://example.com/xrd.png)

2.2. Hydrodynamic Size and Morphological Characterizations on SHA Series MNPs. Figure 3a–f shows the hydrodynamic size distributions of samples SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30 with mean values of 10.46, 18.07, 20.69, 27.56, 28.28, and 32.6 nm, respectively. The average hydrodynamic sizes of SHA series MNPs are summarized in Table 1. The SHA series MNPs have two organic coating layers: one monolayer of oleic acid and another monolayer of the amphiphilic polymer. The total thickness of the organic layer coating is about 4 nm. This causes the hydrodynamic size of the nanoparticles to be about 8–10 nm larger than their inorganic core size measured by TEM (see Figure 4).

The magnetic core morphologies of SHA series MNPs are shown in Figure 4. Some MNPs are agglomerated during the evaporation process of the MNP suspensions. For the MNPs with smaller sizes such as samples SHA-5 and SHA-10, the magnetic core shapes are not strictly spherical, which might cause higher shape anisotropies as well as higher effective magnetic anisotropies in these MNPs. However, larger MNPs show spherical magnetic cores. The contrast of different MNPs from one TEM image is due to the different crystal orientations. When the crystal zone axis is close to the incident electron beam, the MNPs show a darker color.

2.3. Zeta Potential of SHA Series MNPs. The SHA series MNPs have a neutral to slightly alkaline pH between 7.2 and 7.6. The measured zeta potential values for SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30 are −0.03, +5.03, +7.66, −0.41, +1.15, and −0.69 mV, respectively.

2.4. Dynamic Magnetic Responses of SHA Series MNPs under a Low-Frequency Driving Field. The dynamic magnetic responses of SHA series MNPs under a mono-frequency driving field are investigated. The driving field frequency is varied from 50 to 2850 Hz, and the field amplitude is set at 170 Oe (Gauss).²⁰,²⁰ Each plastic vial containing 200 μL of SHA series MNPs in 10 mM PBS and 0.03% NaN₃ is placed under the alternating magnetic field for MPS measurements. For MNPs suspended in liquid solution under an external magnetic field, they undergo two distinct relaxation mechanisms by which the magnetic moments rotate in response to the field: the Néel relaxation is the flipping of the magnetic moment between easy axes inside a stationary MNP, and on the other hand, the Brownian relaxation is the physical rotation of the entire MNP along with its magnetic moment. In principle, both relaxation mechanisms play important roles in determining the dynamic magnetic responses of MNPs in suspension when subjected to the alternating magnetic field. Depending on the magnetic properties (such as effective anisotropy constant and saturation magnetization),²⁸,²⁹ the physical properties (magnetic core size and the hydrodynamic size including the polymer coatings and anchored biological compounds such as proteins, peptides, cells, and so forth) of MNPs, the nanoparticle volume fraction of the suspension (i.e., dipolar interactions),¹⁵,³⁶–³⁸ and the physical properties of the suspension (temperature and viscosity),²²,²³,²⁵,²⁷–³⁵ MNP concentrations could undergo either Néel or Brownian process-dominated relaxation. It has been reported that for a system of non-interacting iron oxide nanoparticles with negligible polymer coatings, the magnetic dynamics will be dominated by the Brownian process when the core size is above 15 nm and the Néel process dominates when the core size is below 15 nm.⁹,³⁸–⁴⁹ (see S7 from the Supporting Information).

Under a low-frequency driving field (f < 500 Hz), magnetic moments of SHA series MNPs with diameters from 5 to 30 nm are able to follow the time-varying magnetic field. As shown in...
S8 from the Supporting Information, all the six SHA series MNPs show similar phase angles to the driving field \( (f < 500 \text{ Hz}) \), and as the field frequency increases, the differences in the phase angles between six samples increase. Larger MNPs with a larger effective relaxation time show a larger phase lag to the driving field.

**Figure 3.** DLS number-weighted distributions of the hydrodynamic size of MNPs from samples (a) SHA-5, (b) SHA-10, (c) SHA-15, (d) SHA-20, (e) SHA-25, and (f) SHA-30 as characterized by DLS. In each figure, the solid green lines are the fitted log-normal distribution curves and the solid red lines are the cumulative distribution curves. The \( \mu \) values represent the statistical mean of the hydrodynamic sizes of the samples. The standard deviation and \( R^2 \) values are represented by \( \sigma \) and \( R^2 \), respectively, for each case.

**Figure 4.** TEM images of SHA series MNPs. (a–f) SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30, respectively. Scale bars represent 20 nm. TEM images of SHA series MNPs under different magnifications are given in S5 from the Supporting Information.
As summarized in Figure 5, under a low-frequency driving field, the dynamic magnetic responses of six SHA series MNPs from the strongest to the weakest are SHA-30 > SHA-20 > SHA-15 > SHA-25 > SHA-10 > SHA-5. Figure 5a–c summarizes the amplitudes measured at the third, fifth, and seventh harmonics, respectively. Figure 5d–f highlights the corresponding harmonic amplitudes under driving field frequencies of 350, 650, 1250, and 1850 Hz.

Figure 6 summarizes the real-time voltage signal obtained from pickup coils at driving field frequencies of 350, 950, and 1850 Hz. The extracted harmonics are plotted along with the total signal in real time. MNPs with stronger dynamic magnetic responses to the driving field generate larger harmonic signals and are thus able to cause the distortions in the total signal (the highlighted dark areas in Figure 6). It is observed that SHA-30 and SHA-20 show the strongest dynamic magnetic responses to the low-frequency driving field, followed by SHA-15 and SHA-25. SHA-5 and SHA-10 show negligible dynamic magnetic responses compared to the former SHA series MNPs, which are mainly due to the low magnetic moments and linear magnetization curves, as shown in Figure 1.

2.5. Dynamic Magnetic Responses of SHA Series MNPs under a High-Frequency Driving Field. In this section, we report the dynamic magnetic responses of SHA series MNPs under high-frequency driving fields. A dual-frequency method is used herein; one excitation field is set at 10 Hz and a magnitude of 170 Oe, and the other high-frequency driving field is set at varying frequencies (from 1 to 20 kHz) and a magnitude of 17 Oe.

Under a high-frequency driving field, larger MNPs (i.e., SHA-30) are unable to rotate their magnetic moments to the fast-switching magnetic field; thus, their dynamic magnetic responses are weakened. As shown in S10 from the Supporting Information, there is a constant harmonic phase difference of 50° between SHA-10 and SHA-30 MNPs. As a result, under a high-frequency driving field, the dynamic magnetic responses of six SHA series MNPs from the strongest to the weakest are SHA-15 > SHA-20 > SHA-30 > SHA-25 > SHA-10 > SHA-5 (as shown in Figure 7).

Although recent in origin, MNPs of different core sizes have found their applications in various fields of science. This section of the paper is dedicated to identifying the utility of the different-sized and surface-functionalized MNPs in realistic applications. The SHA series particles are amine-functionalized MNPs. As the amine groups are less selective and less specific for antibodies and proteins, they capture a varied range of bacterial pathogens and allow purification of water, food, and urine samples. The VSM characterization of the SHA series in Figure 1a–f shows that SHA-5, SHA-10, SHA-15, and SHA-20 are superparamagnetic. Although SHA-25 and SHA-30 show higher magnetic moments, they show remanent magnetizations. For magnetic biosensing, higher-moment particles are preferred in order to generate a higher magnetic signal per particle. However, practical limitations such as colloidal stability (no clustering) should also be considered. For the
SHA series, SHA-25 exhibits the second highest magnetic moment/particle with a remanent magnetization of 1.28% $M$, where $M$ is the specific magnetization under 5000 Oe. Although SHA-30 has a higher magnetic moment/particle compared to SHA-25, a much larger remanence magnetization of 10.93% $M$ is observed from SHA-30. Taking both magnetic moments and remanent magnetizations into consideration, SHA-25 is the optimum candidate from SHA series for biosensing applications. On a different note, for cell separation and sorting and drug/gene delivery, as the property of superparamagnetism is not essential and a higher magnetic moment ensures a larger magnetic torque (force), the highest-magnetic-moment MNP, SHA-30, is probably a better candidate.

For homogeneous bioassays that are based on a conjugation-mediated change in Brownian relaxation time, MNPs should be thermally blocked.\textsuperscript{13,44–59} Thus, an in-depth study on the Brownian and Néel relaxation times of these MNPs under different driving fields should be carried out.\textsuperscript{60,61} For magnetic hyperthermia therapy, the dissipated energy or specific absorption rate (SAR) is directly proportional to the imaginary component of AC susceptibility and saturation magnetization ($M_s$) of MNPs, the applied field frequency, and the amplitude squared.\textsuperscript{62–66} Thus, a high $M_s$ of MNPs does not guarantee a high SAR, and practical measurements on the hyperthermia performance are required to find out which SHA series MNPs are better suited for hyperthermia applications.

Furthermore, MRI techniques require the MNPs to be injected into the body fluids which then accumulate in the

Figure 6. Recorded real-time dynamic magnetic responses of SHA series MNPs under a low-frequency driving field. The higher harmonics are extracted and plotted in parallel with the total signal obtained from the pickup coils. [(a,d,g,j,m,p)], [(b,e,h,k,n,q)], and [(c,f,i,l,o,r)] are the real-time total signal and higher harmonics under driving field frequencies of 350, 950, and 1850 Hz for samples SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30, respectively. A zoom-in view of the higher harmonics is plotted in S9 from the Supporting Information.
target tissues. Hence, for MRI applications, it is extremely essential for the MNPs to be small as larger MNPs have greater tendency to block the arteries. In this case, SHA-5 and SHA-10 MNPs will be quite useful.67 The dynamic magnetic responses of SHA series MNPs are compared in this paper using a homebuilt MPS system. The harmonics are induced under different driving magnetic fields, which are a result of the joint effects of relaxation mechanisms and the magnetic moment of each MNP. For magnetic particle imaging (MPI) and MPS-based bioassays, larger dynamic magnetic responses (higher harmonic amplitudes) ensure a higher signal-to-noise ratio and sensitivity. Thus, SHA-30 MNPs are suggested for MPI and MPS-based bioassays where the driving field frequencies are below 2 kHz, while SHA-15 MNPs are suggested for these applications where the driving field frequencies are above 2 kHz.

3. CONCLUSIONS

In this paper, we characterized the magnetic and physical properties of SHA series MNPs from Ocean NanoTech using standard characterization tools. The VSM results show that SHA-5, SHA-10, SHA-15, and SHA-20 MNPs are superparamagnetic and on the other hand, SHA-25 and SHA-30 are not superparamagnetic, with SHA-30 showing the highest magnetic moment per particle, followed by SHA-25, SHA-20, SHA-15, SHA-10, and SHA-5. Thus, SHA series iron oxide nanoparticles with larger core sizes are preferred for magnetic biosensing and drug delivery where high-moment MNPs are desired for higher magnetic signals and higher magnetic torques. However, SHA-25 and SHA-30 show remnant magnetizations upon the removal of the magnetic field (non-superparamagnetic), and thus, they are not applicable for applications where superparamagnetism is required. The XRD results show that all SHA series MNPs are composed of γ-Fe₂O₃ and Fe₃O₄. The dynamic magnetic responses of these iron oxide nanoparticles are investigated using a homebuilt MPS system, where both the responses under low and high driving field frequencies are summarized. It is observed that under low driving field frequencies, the dynamic magnetic responses of SHA series MNPs from the strongest to the weakest are SHA-30 > SHA-20 > SHA-15 > SHA-25 > SHA-10 > SHA-5. However, under high driving field frequencies, due to the larger phase lags of larger MNPs, the dynamic magnetic responses from the strongest to the weakest are modified: SHA-15 > SHA-20 > SHA-30 > SHA-25 > SHA-10 > SHA-5. These results give hints on designing MPI and MPS-based bioassays to maximize the use of different MNPs of different core sizes. At the end of this paper, based on the requirements and goals of MNP-based applications, we suggested different SHA MNPs for each application.

4. MATERIALS AND METHODS

4.1. Materials. The SHA series MNPs are provided by Ocean NanoTech. Six SHA series MNPs with average magnetic core sizes of 5, 10, 15, 20, 25, and 30 nm are characterized in this paper (denoted as SHA-5, SHA-10, SHA-15, SHA-20, SHA-25, and SHA-30, respectively; photographs of SHA series MNPs used in this work can be found in the Supporting Information, S1). The SHA series MNPs are a group of water-soluble iron oxide nanoparticles coated with the
amphiphilic polymer and functionalized amine reactive groups. They are very stable in most buffers in the pH range of 4–10 and can be readily conjugated to proteins, peptides, and other carboxylic acid-containing molecules.

4.2. VSM Measurement. 25 μL of the SHA series MNP suspension is pipetted onto a filter paper and air-dried before the VSM measurements. Three independent magnetization curves of each sample are obtained at 20 °C, with the external magnetic field swept from −5000 to +5000 Oe (a field step of 10 Oe and an averaging time of 200 ms), −500 to +500 Oe (a field step of 2 Oe and an averaging time of 200 ms), and −200 to +200 Oe (a field step of 1 Oe and an averaging time of 200 ms).

4.3. XRD Measurement. 50 μL of the SHA series MNP suspension is pipetted onto a Si/SiO2 slide and air-dried before the XRD characterization. A cobalt radiation source (wavelength ≈1.79 Å) is used for the XRD characterization since it has lower fluorescence, especially for magnetite and maghemite. For a convenient comparison, the characterized XRD patterns are converted to copper radiation. The crystal structure of SHA series MNPs is characterized via XRD (Bruker D8 Discover 2D).

4.4. DLS Measurement. The hydrodynamic size distribution of the SHA series MNPs is characterized using a DLS Particle Tracking Analyzer (model: Microtac Nanoflex). 100 μL of the SHA series MNP suspension is diluted in 1.4 mL of deionized (DI) water, reaching a total sample volume of 1.5 mL of the mixture, followed by ultrasonication for 30 min before the DLS characterization.

4.5. TEM Analysis. The morphologies of these SHA series MNPs are characterized using a TEM system (FEI T12 120 kV). Each TEM sample is prepared by putting a droplet (~10 μL) of the MNP suspension onto a TEM grid (copper mesh coated with an amorphous carbon film). These samples are ready for TEM characterization when the solutions are fully evaporated at room temperature in air.

4.6. Zeta Potential Measurement. A zeta potential analyzer (model: Stabino) is used to characterize the particle charge distribution or the zeta potential of the SHA series MNPs in DI water. 100 μL of SHA series MNPs is diluted in 4.9 mL of DI water, reaching a total sample volume of 5 mL, followed by ultrasonication for 30 min, and then used for zeta potential characterization. This particle charge characterization helps to analyze the surface binding capabilities of these SHA series MNPs.

4.7. MPS Measurement. The dynamic magnetic responses of SHA series MNPs are characterized using a homebuilt MPS system (see the schematic view and photographs of the MPS system in S2 and S3 from the Supporting Information). 200 μL of the SHA series MNP sample is sealed in a plastic vial (a maximum capacity of 300 μL). Two sets of copper coils are used to generate sinusoidal magnetic fields with tunable frequencies and magnitudes. One pair of differentially wound pickup coils (600 windings clockwise and 600 windings counter-clockwise) collects the induced voltage signals due to the dynamic magnetic responses of MNPs under driving magnetic fields. A laptop with LabVIEW controls the frequency and magnitude of the driving magnetic field through a data acquisition card (DAQ, NI USB-6289). The analog voltage signals are sent back from pickup coils to DAQ, sampled at 500 kHz, and converted to the frequency domain after discrete Fourier transform. For each MPS measurement, the MPS system runs for 10 s to collect the baseline signal (noise), followed by inserting the vial containing the MNP sample for another 10 s of signal (total) collection. The induced voltage due to dynamic magnetic responses of MNPs is recovered from the total signal by the phasor theory (see S4 from the Supporting Information). The higher harmonics specific to dynamic magnetic responses of MNPs are extracted for analysis (see S6 from the Supporting Information).

## ASSOCIATED CONTENT

### Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c05845.

Photographs of SHA series iron oxide nanoparticles; MPS system; MPS system setups; phasor theory; magnetic moment per gram of Fe; TEM images of SHA series MNPs captured at various magnifications; magnetic dynamic responses and higher harmonic models; Brownian and Neel relaxations; phase angles of higher harmonics monitored under a low-frequency driving field for SHA series samples; recorded real-time dynamic magnetic responses of SHA series MNPs under a low-frequency driving field; and phase angles of higher harmonics monitored under a high-frequency driving field for SHA series samples (PDF)

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#### Notes
The authors declare no competing financial interest.
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