

Alternating field magneto-optical response of hard magnetic nanoplate dispersions

Artem A. Eliseev^{1,a,b}, Zitian Xia^{2,c}, Alexey V. Lukashin^{2,3,d}, Pavel E. Kazin^{1,e}, Andrei A. Eliseev^{2,f}

¹Department of Chemistry, Lomonosov Moscow State University, Leninskie Gory, 1b3, Moscow 119991, Russia

²Department of Materials Science, Lomonosov Moscow State University, Leninskie Gory, 1b73, Moscow 119991, Russia

³Kurnakov Institute of General and Inorganic Chemistry of the Russian Academy of Sciences, Moscow, 119991 Russia

^aartem.a.eliseev@gmail.com, ^baeliseev@inorg.chem.msu.ru, ^cxiazitian1999@gmail.com,

^dlukashinav@my.msu.ru, ^ekazin@inorg.chem.msu.ru, ^feliseev@inorg.chem.msu.ru

Corresponding author: Artem A. Eliseev: artem.a.eliseev@gmail.com, aeliseev@inorg.chem.msu.ru;
Andrei A. Eliseev: eliseev@inorg.chem.msu.ru

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ABSTRACT Here we provide an analysis of alternating field magneto-optical (AFMO) response for concentrated dispersions of hard magnetic nanoplates. Dynamics of hexaferrite 50×5 nm nanoplates in 0.3–30 g/L magnetic fluids was traced with amplitude and phase shift of transmission AFMO response in the frequency range of 1 Hz – 10 kHz and magnetic fields up to 50 Oe. AFMO signal was used for reconstructing fast magnetization susceptibility dependence on the applied field depending on concentration of nanoplates. The two modes of platelets reorientation were revealed with AC field frequency and amplitude variation. High-frequency mode, characterized by magnetization phase lag, increasing with the excitation field frequency, corresponds to incomplete orientation of platelets after the field. Contrary, the low-frequency mode with magnetization phase lag, increasing reciprocally to the excitation field frequency, reveals AC coercivity of ferrofluid. The last is addressed to the self-stabilization of particles orientations by magnetic momenta of constituent particles.

KEYWORDS Hexaferrite, ferrofluid, magneto-optics, magnetization dynamics.

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1. Introduction

In the recent years, magnetic fluids of hard magnetic nanoparticles with anisotropic shape have attracted considerable attention [1]. Compared to conventional magnetic fluids, built from soft magnetic nanospheres nanoparticles, ferrofluids comprising of hard magnetic wires or platelets enables both stabilization of magnetic momenta within the particles with magnetocrystalline anisotropy and stabilization of particles orientation within the liquid with drag momenta [2]. These properties make these ferrofluids extremely attractive for many technological applications, including reconfigurable magnets, bistable solenoids, tunable friction pairs and bearings, magnetorheological systems, magnetic lubrication, viscous dampers, triboelectric generators etc [3–5].

Nanoscale dynamics in ferrofluids is considered a most important factor, affecting their technological applicability. The dynamic behavior of magnetic particles in a viscous medium is determined by the interplay of Brownian motion, viscous drag, external magnetic field strength and interparticle interactions [2, 6]. In common magnetic fluids, the relaxation of the magnetization of the system proceeds by two distinct mechanisms: Néel and Brownian relaxation [7, 8]. Soft superparamagnetic particles predominantly exhibit Néel relaxation, whereas hard magnetic particles are dominated by Brownian mechanism. In hard magnetic materials magnetocrystalline anisotropy barrier strongly suppresses internal moment rotation and demagnetization proceeds by physical rotation of particles [1, 9, 10]. The dynamics of this process is governed by solvent viscosity and particle shape [9]. While spherical particles possess lowest drag momenta for rotational movement, dominated by viscous friction, nanorods and nanoplates experience additional drag due to the necessity of fluid displacement [11]. The largest drag momentum is attained for disc-shaped particles, which makes them most attractive for hydrodynamic and magnetorheological applications.

Among the few hard magnetic materials capable of forming stable colloidal solutions of anisotropic particles, M-type hexaferrites stand out due to their high thermodynamic and chemical stability, unique magnetic structure and strong structural anisotropy [12–15]. These materials exhibit a pronounced uniaxial magnetocrystalline anisotropy with a magnetic easy axis aligned along the crystallographic *c*-axis. Owing to the superexchange interactions of Fe^{3+} ions occupying five inequivalent lattice sites, a ferrimagnetic order is established, with the moment rigidly fixed perpendicular to the basal plane of nanoplates [14, 16]. Consequently, even at extremely small dimensions, these particles possess a substantial intrinsic magnetic moment and high coercivity. The combination of high magnetocrystalline anisotropy and platelet morphology enables complex rotational dynamics of hexaferrite hard magnetic nanoplates under an alternating magnetic field. Furthermore, due to the significant difference in the depolarization factors of nanoplates and dielectric ellipsoid components of hexaferrite phase along *a* and *c* crystallographic axis [9, 17], the colloidal solutions of such particles exhibit strong magneto-optical response upon particle reorientation [14]. These effects form the physical basis for the magneto-optical response of hexaferrite ferrofluids and allow optical tracking of the rotational dynamics of the nanoplates.

Based on the above principles, our group recently used strontium hexaferrite nanoplates as nanorotor viscosity sensors [18]. Through a combination of experiments and theoretical modeling, the rotational dynamics of the nanoplates under an alternating magnetic field was systematically investigated. A relation of drag force, phase lag tangent and the viscosity of the medium was established, enabling fast, in-situ evaluation of liquid viscosity over a range of 0.5–1000 mPa·s. Furthermore, a spatial resolution down to 4 μm was demonstrated, allowing viscosity visualization in microchannels with micrometer resolution.

The detailed studies of dynamic behavior of colloid nanoparticles under rotating magnetic field showed the presence of two distinct regimes of particles precession: synchronous and asynchronous modes [19, 20]. Romodina et al. [19] showed that Brownian torque induces the flattening of the breakdown transition between the synchronous and asynchronous modes. The results of Brownian dynamics simulations agree with the experimental data, confirming the essential role of thermal fluctuations in field-driven rotation. Honecker et al. have demonstrated [20] that, as the frequency of a rotating magnetic field increases, the motion of hematite nanospindle (length 374 nm, diameter 51.5 nm) evolves from coherent precession involving rotation about the minor axis to synchronized spinning about the principal axis with spindles tending to collinear alignment perpendicular to the rotation field plane. In contrast to rotating magnetic field, an alternating magnetic field causes periodic reversal of the magnetic torque [18], leading to the observation of three distinct regimes of particle rotation with increasing frequency: synchronous rotation, synchronous oscillation, and asynchronous response.

Despite of rather a significant progress made in the recent years in the field, the information on particles dynamics in ferrofluid remains rather scarce. To the best of our knowledge no information on the collective effects on nanoplates dynamics in ferrofluids has been reported yet. Thus, in the present study we provide a comprehensive investigation of the dispersions of hexaferrite hard magnetic nanoplates over a concentration range of 0.3–30 g/L with earlier developed alternating field magneto-optical (AFMO) method [18]. This study enabled to reveal the dynamics of reorientation of platelets in the dispersion depending on concentration. Using the AFMO signal, we reconstructed the dependence of the magnetic susceptibility on the applied magnetic field for magnetic fluids at different concentrations. Additional information on the structure of dispersions is gained with the analysis of AC coercivity on excitation frequency, enabling to propose self-stabilization of particles orientations by the magnetic moment of the platelets.

2. Experimental

Plate-like strontium hexaferrite nanoparticles were synthesized via the glass-ceramic route [14, 21]. The precursor glass with the nominal composition $4\text{Na}_2\text{O} \times 9\text{SrO} \times 5.5\text{Fe}_2\text{O}_3 \times 4.5\text{Al}_2\text{O}_3 \times 4\text{B}_2\text{O}_3$ was prepared by rapid melt quenching. To induce crystallization, the glass was subjected to isothermal heat treatment at 700°C for 2 hours, followed by air quenching. The resulting glass-ceramic was treated with 3% aqueous hydrochloric acid to dissolve borate matrix. The residual particles were washed with deionized water and redispersed in acidic aqueous media with pH = 4, obtaining a stable colloidal solution with concentration of ~ 0.3 g/L. The concentrated dispersions of approximately 30 g/L were obtained by centrifugation at 10,000 rpm (11,000 g) for 30 minutes followed by 1 day separation in gradient magnetic field. Condensed phase was extracted from the bottom of the vial.

Magnetic measurements of field-frozen colloid particles were performed using a Faraday balance type magnetometer with magnetic fields of up to 18 kOe at 77 K. The magnetic moment measurement accuracy was 3%, and the applied magnetic field accuracy was ± 100 Oe. Magnetic properties of colloidal solution were measured at room temperature (298 K) using a Quantum Design SQUID MPMS-7XL magnetometer. Particle sizes and shape were analyzed using transmission electron microscopy (Zeiss Libra 200 and Jeol JEM-2100).

The AC magneto-optical (AFMO) measurements were performed in a originally developed setup (Fig. 2a) similar to previously reported in [18]. Perpendicular geometry of laser beam (532 nm) and magnetic field was used to maximize the magneto-optical response. To avoid polarization induced artifacts, the laser beam first passed through a polarizer, then through the cuvette (standard 10 mm quartz cuvette for diluted dispersion and 50 μm thick microcuvette for concentrated solution) containing the investigated dispersion. Fast BPW21R photodiode (Vishay Semiconductors) was used for light intensity detection. Microfocus experiments were conducted using Renishaw Invia system. The magnetic field in the micro-cuvette was generated by specially designed Helmholtz coils. A sinusoidal alternating current with a frequency

range from 2 Hz to 5 kHz was supplied by a digital oscilloscope with a built-in signal generator (Velleman Instruments PCSGU250). The signal was amplified using CREST AUDIO VS1500 to provide AC field magnitude of 2–50 Oe. Synchronization of the optical signal with the magnetic field was achieved by simultaneous registration of the Hall sensor (rigidly fixed on the coils) response.

AFMO signal analysis and hysteresis-like loop reconstruction was performed according to the following routine. To extract the positions of maxima and minima of AFMO signal, the experimental field-time dependency was converted into half-period fraction T values (varying in $-\pi/2$ to $\pi/2$ range) by maximum field normalization as momentary field was directly evaluated from Hall sensor response:

$$T = \arcsin\left(\frac{H(t)}{H_{\max}}\right), \quad (1)$$

where $H_{\max}$ is maximum applied field and $H(t)$ is momentary field value at time t . This allowed one to build AFMO- T curve which was fitted with 5-th order polynomial function around extreme values in the range of 0.15π of oscillating field half-period. The corresponding key point time lag and AFMO amplitude values were extracted from the fit curves. Demagnetization and magnetization phase lag (normalized to 0–1 range) values were evaluated as:

$$\varphi_{\text{dem}} = 2 \cdot \text{abs}(T_{\min})/\pi, \quad (2)$$

$$\varphi_{\text{mag}} = 2 \cdot (1 - \text{abs}(T_{\max}))/\pi, \quad (3)$$

where $T_{\min}$ represents the period lag to minimal AFMO signal within the half-period and $T_{\max}$ represents the period lag to maximal AFMO signal within the half-period as extracted from experimental data. Phase lag data were averaged over at least 5 sampling periods.

As AFMO minima positions correspond to the most disordered state, minimal AFMO voltage was considered zero magnetization level. AFMO minima were used as reference key points for inverting AFMO signal and obtaining hysteresis-like form of AFMO magnetic susceptibility. This hysteresis-like form was further used to extract full AFMO amplitude, AC coercive fields and visualize the positions of the maximum field response.

3. Results and discussion

Prior to spectroscopic investigation, the morphology and phase composition of hexaferrite nanoplates was examined with common solid-state methods including XRD, transmission electron microscopy and magnetic measurements (Fig. 1). The plates represent hexaferrite phase $\text{SrFe}_{11.5}\text{Al}_{0.5}\text{O}_{19}$ having average lateral size of 50 ± 20 nm and the thickness of 5 ± 1 nm. The concentration of particles in the dilute colloid as determined by both gravimetry and magnetometry equals to 0.3 ± 0.1 g/L. Combined centrifugation and magnetic separation concentration processes allows one to increase the dispersed phase concentration to 30 ± 5 g/L. Magnetic properties of ferrofluids were analyzed for both field-frozen (at ~ 18 kOe) concentrated dispersion of nanoplates and liquid ferrofluid. While oriented frozen particles reveal high coercivity of ~ 5 kOe (Fig. 1b), the liquid colloidal solution demonstrates soft magnetic behavior with nearly undetectable coercive force (Fig. 1c). Importantly, conventional magnetometry equipment (especially SQUID) have very long measurement duration, which makes impossible investigations of fast particles dynamics. On contrary fast alternating field magneto-optical response allows to follow reorientation of nanoplates enabling development of alternative powerful and fast approach (Fig. 2).

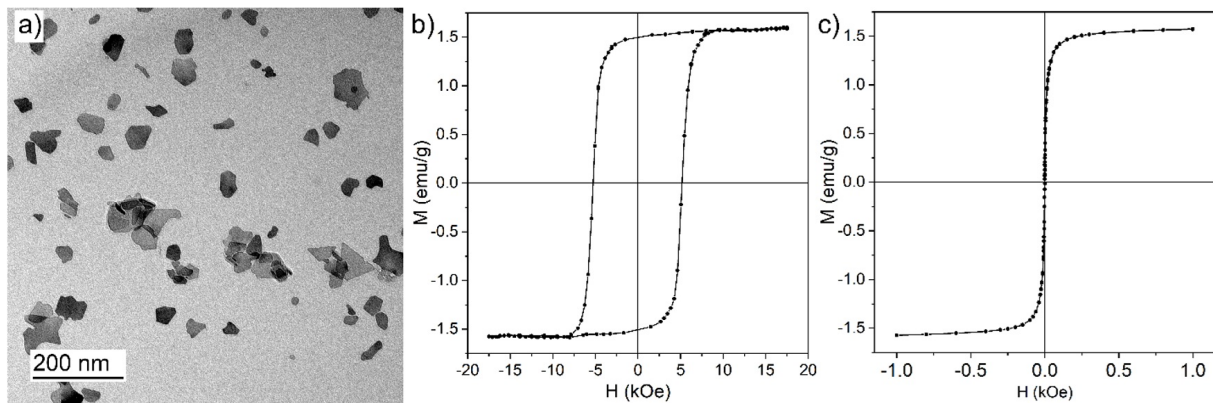


FIG. 1. Transmission electron micrograph of hexaferrite 50×5 nm nanoplates (a) and magnetic hysteresis loops for field-frozen dispersion of nanoplates at 77 K (b) and liquid ferrofluid at 298 K (c)

The optical properties of hexaferrite nanoplates dispersions are determined by both the anisotropic scattering and the absorbance of as reported in previous study [18]. Scattering anisotropy effect improves slightly with diminishing the wavelength all over the range of 800–300 nm (Fig. 2b), while absorbance anisotropy has a wide maximum around

700 nm [18]. As a result, the most pronounced optical effect is achieved for the transmission geometry at the low wavelengths. However as hexaferrite is a semiconductor with a band gap of ~ 2 eV with optical absorption increasing strongly below 500 nm, further magneto-optical measurements were conducted at the wavelength of 532 nm.

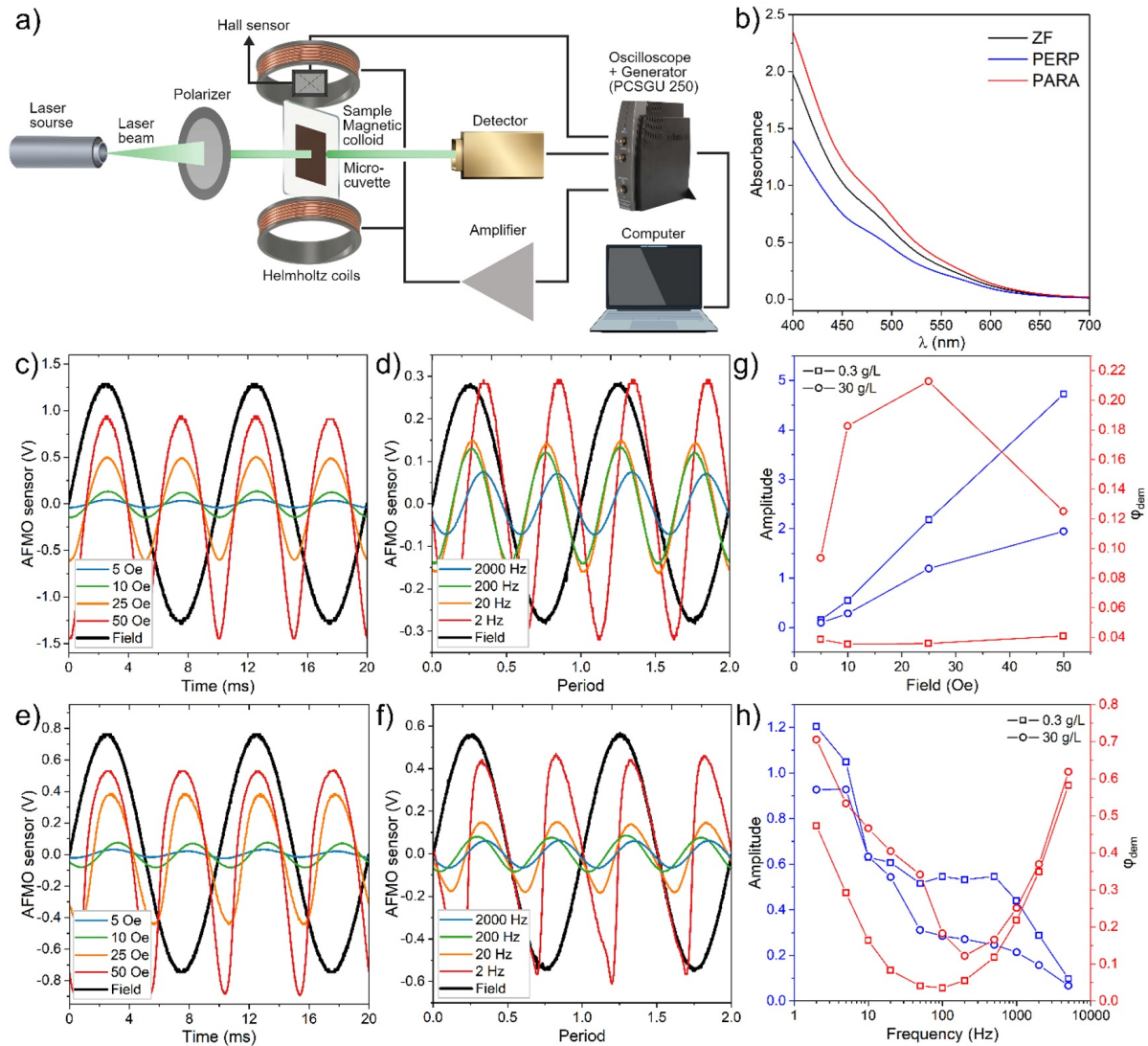


FIG. 2. Optical scheme for evaluation of alternating field magneto-optical (t-AFMO) response (a). Transmission spectra of nanoplate dispersions aligned along and perpendicular to the field direction (b). Example AFMO response for diluted (0.3 g/L, c–d) and concentrated (30 g/L, e–f) hexaferrite nanoplate dispersions depending on field amplitude at 100 Hz (c, e) and frequency at 10 Oe (d, f). Field (g) and frequency (h) amplitude and phase angle dependencies for diluted and concentrated colloid. Note the 200x difference in the optical path for diluted and concentrated dispersions during data acquisition.

The alternating field magneto-optical (AFMO) response was registered with an optical scheme depicted in Fig. 2a in a wide range of frequencies from 2 Hz to 5 kHz and magnetic field amplitudes up to 50 Oe (RMS). As detector voltage is linearly proportional to illumination intensity, AFMO should be considered as transmission-like response with minima, corresponding to the disordered state of dispersion, and maxima, reflecting maximum orientational order in ferrofluid.

Generally, the amplitude of magneto-optical response grows proportionally with AC field amplitude and starts to saturate for concentrated ferrofluid at the fields above 50 Oe (Fig. 2g). AFMO amplitude also generally decreases with increasing excitation field frequency (Fig. 2h). Contrary, both field and frequency effect on phase lag is far from being monotonic (Fig. 2d, f, h). That is addressed to nonlinear variation of a phase angle between the excitation field and magneto-optical response. According to experimental results, the phase angle diminishes at low frequencies but grows at high frequencies with the frequency of excitation field (Fig. 2h). For better understanding of underlying processes complete field-frequency maps for AFMO signal amplitude and phase shifts were assembled for further analysis (Fig. 3).

Importantly, one can notice an asymmetry in AFMO waves at high fields and low frequencies (Fig. 2e,f). Such signal form obviously arises from the difference in the dynamics of magnetization and relaxation processes. According to earlier

study, magnetization is driven by magnetic and Brownian torque (thus strongly dependent on field strength), while the surrounding medium slows down particle rotation with drag forces [18]. On the contrary, demagnetization relies only on Brownian relaxation, being also affected by the medium drag. These processes are described with magnetization and demagnetization relaxation times defined with:

$$\tau_{r,magn} = \frac{3\eta\pi^3 R^3}{2\mu_0\mu H + ok_B T}, \quad (4)$$

$$\tau_{r,demagn} = \frac{3\eta\pi^2 R^3}{k_B T}, \quad (5)$$

where η is media viscosity, R is the particle radius, μ_0 is the vacuum magnetic permeability, μ is the magnetic moment of the particle, H is the applied field, coefficient o depicting orientational distribution (averaged number of available degrees of freedom), k_B is the Boltzmann constant and T is the temperature.

According to (4),(5), synchronization of the magnetic field and AFMO signal occurs at equality of field-induced and drag momenta of platelets rotation, while the torque values for magnetization and relaxation processes can appear to be quite different, especially at high fields. Moreover field-driven structural transformations in ferrofluid can also significantly affect relaxation times. Thus, extreme magnetization and demagnetization phase shifts were extracted separately from AFMO data. Maximum AFMO signal was ascribed to a magnetized state and characterized with φ_{mag} magnetization process phase shift term, while φ_{dem} term (minimal AFMO signal) was used for disordered state phase shift characterization.

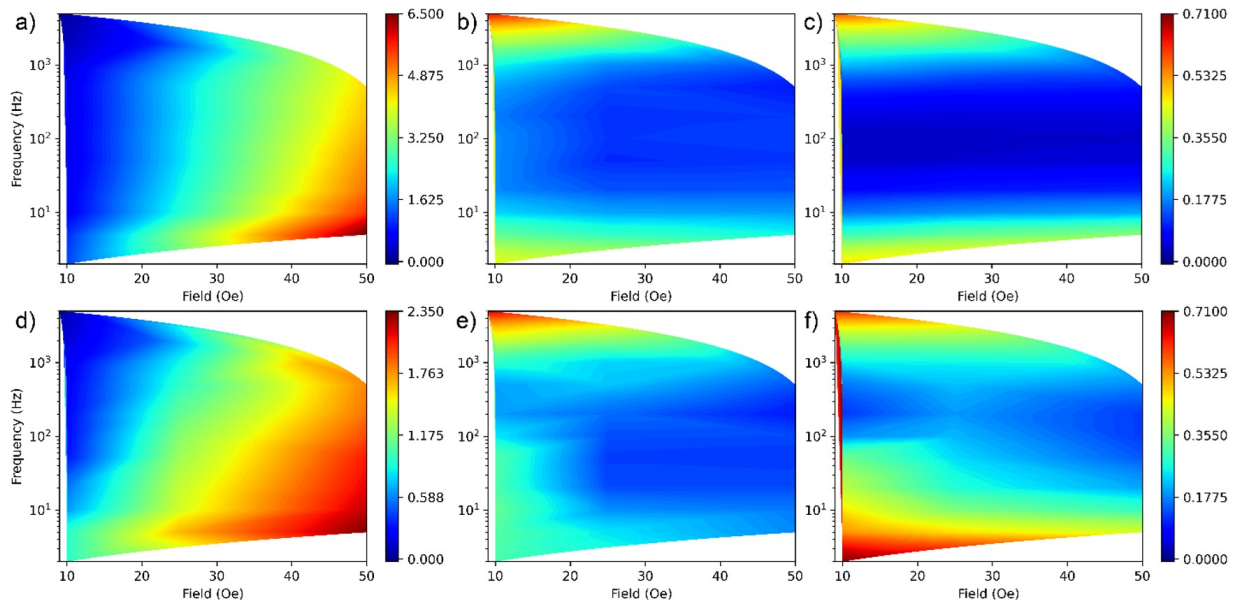


FIG. 3. Transmission AFMO maps for signal amplitude and phases for diluted (0.3 g/L, a–c) and concentrated (30 g/L, d–f) hexaferrite nanoplate dispersions. Due to asymmetry in magnetization and demagnetization processes, the phase maps are provided for AFMO maxima φ_{mag} (corresponding to utmost alignment of nanoplates, b, e) and AFMO minima φ_{dem} (corresponding to the disordered state, c, f)

In high frequency region (above 100 Hz) phase angle comes in agreement with previously reported AC magnetometry data for both diluted and concentrated colloids as magneto-optical response in this case arises from single particle reorientation (Fig. 3b,c,e,f). Fig. 3a, d illustrate decline in AFMO amplitude with frequency and its rise with field intensity as indented by “paramagnetic gas” model. Thus, one can conclude on domination of individual particle response and uncorrelated rotation of platelets in either diluted or concentrated ferrofluids at high frequencies.

Low frequency region demonstrates odd behavior for dilute and concentrated dispersions. The major difference appears in both magnetization and demagnetization phase maps at low field/low frequency region. Obviously, it should be ascribed to the difference in the collective behavior of nanoplates. It is known, that the internal structure of concentrated ferrofluid (30 g/L) contains large thread-like dynamic aggregates [22, 23]. Field-driven assembly and relaxation of these threads should occur at much higher relaxation times, as it requires movement of large volumes in the dispersion. It gives rise to magnetization and relaxation phase lags at low frequencies in concentrated nanoplate dispersions (Fig. 3e,f). Notably, a strong enhancement of φ_{dem} compared to φ_{mag} in concentrated ferrofluid suggests stabilization of its magnetized state. We consider this effect to arise due to internal field provided by the oriented constituent particles. With

disorientation process slowed down by both the internal field and drag forces, one can expect an appearance of remanent magnetization and AC coercive force, rising at lower frequencies due to larger threads formation.

In order to give one common representation for this statement, we have reconstructed hysteresis-like field dependencies of vectorized orientational order of nanoplates from time-resolved AFMO data (Fig. 4). Those can be considered a direct analog to fast magnetic susceptibility measurements.

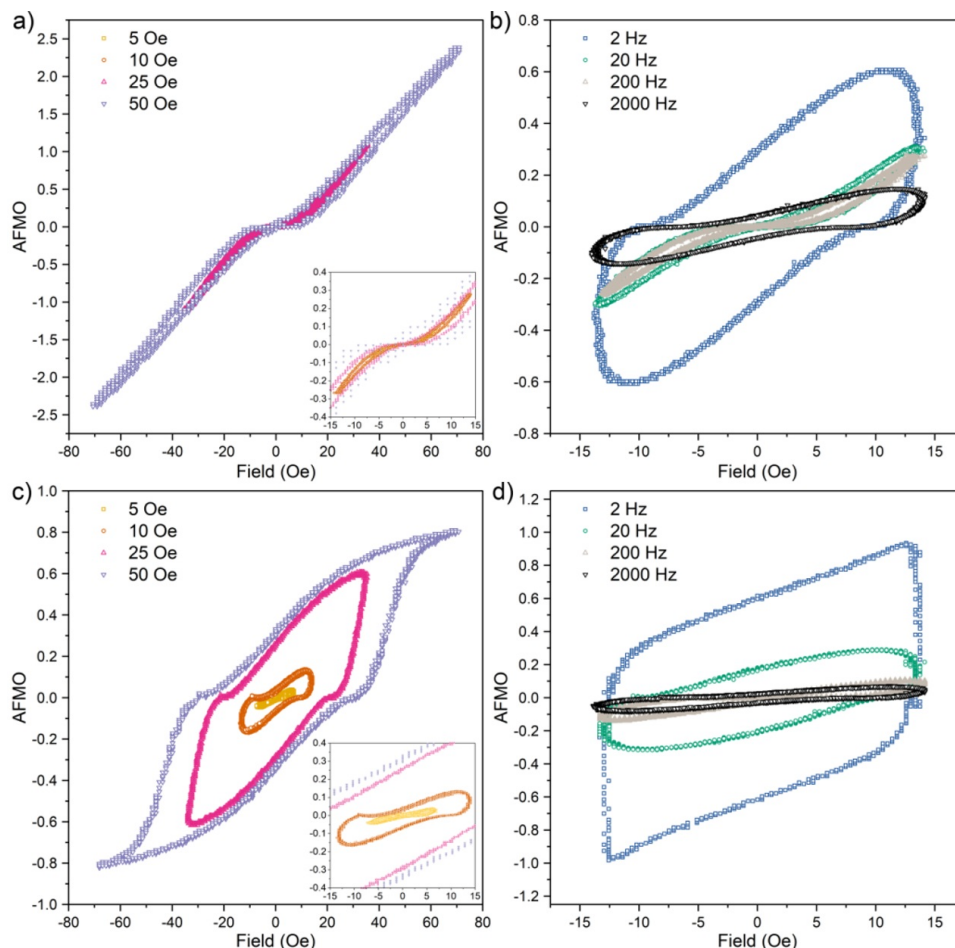


FIG. 4. Sample magnetic hysteresis loops reconstructed with t-AFMO signal for diluted (0.3 g/L, a,b) and concentrated (30 g/L, c,d) hexaferrite nanoplate dispersions depending on AC field amplitude (a, c) and frequency (b, d)

For the diluted colloid at high and intermediate frequency region of > 100 Hz, AFMO magnetization shows closely a linear field dependence without any significant remanence, as expected for freely rotating magnetic particles in the colloid (Fig. 4a). However decreasing field frequency induces hysteresis like response (Fig. 4b). While high frequency irreversibility can be ascribed to particle rotation phase lag due to domination of drag momentum, appearance of hysteresis at low frequency remains of unknown nature. Analysis of concentrated phase AFMO curves can provide necessary insight to unravel reasons for such behavior.

Concentrated magnetic fluid demonstrates strong hysteresis in both field and frequency dependencies (Fig. 4c,d) with maximum detectable value of AC coercive force of about 40 Oe at low frequencies (below 100 Hz). Moreover, this coercivity is proportional to the applied field amplitude with appearance of saturation above 60 Oe. which points out ferromagnetic nature of concentrated colloid. Low frequency hysteresis has a quasi-rectangular shape, indicating very slow relaxation of threads orientational order even at high opposite fields. While such stabilization expected for concentrated liquid due to presence of large dynamic aggregates the same behavior for 0.3 g/L suspensions cannot be addressed using a common “paramagnetic gas” interpretation.

It is known from previous experiments that diluted state is prone to condensation under external field [23]. Formation of dynamic aggregates consisting of several platelets under applied field can lead to significant increase of the drag momentum. Therefore, presence of high coercive force in this case indicates slow assembly of similar to concentrated phase dynamic aggregates, which also can reorient under external magnetic field without thread disruption.

Therefore, the magnetic fluids, consisting of hard magnetic nanoplates combine both the properties of classical magnetic fluids (such as fluidity and ability of particles reorientation) and solid-state magnets (like coercivity and slow relaxation dynamics). Importantly, the typical relaxation times of 0.1 s achieved in concentrated dispersions, strongly exceed Brownian relaxation of ~ 0.5 ms for single particles, revealing the principal possibility of attaining stable magnetization in nanoplate ferrofluids. These specific features make this material highly prospective for the development of new magnetorheological systems including tunable friction pairs and bearings, magnetic lubrication, shielding materials, viscous dampers, triboelectric generators etc.

4. Conclusions

The proposed methodology of alternating field transmission magneto-optical (t-AFMO) measurements have been successfully applied for investigation of fast dynamics of hard magnetic hexaferrite 50×5 nm nanoplate dispersions. It provides fast and efficient pathway for investigation of the particles reorientation and field-driven assembly in ferrofluids.

The studies reveal a strong dependence of both AFMO phase shift and amplitude on the external field frequency in concentrated dispersions of nanoplates. The phase shift has non-monotonic dependence on frequency, exposing the peculiarities of magnetic nanoplatelets dynamics. Synchronization of the magnetic field and AC magnetization occurs at equality of field-induced and drag momenta of platelets rotation. At high frequencies, magnetization lags the AC field and the phase lag increases proportionally to the excitation field frequency, revealing an incomplete orientation of platelets after the applied field. Contrary, at low frequencies, the magnetization phase lag increases reciprocally to the excitation field frequency. The effect is addressed to stabilization of platelets orientation with an internal magnetic field, produced by the dispersed particles. This self-stabilizing field leads to an appearance of high AC coercivity in a magnetic fluid, proportional to the applied field amplitude. Described peculiarities of the dynamics in the magnetic fluid, consisting of hard magnetic nanoplates illustrate a pivotal role of interparticle interactions in ferrofluids and their great potential for development of new magnetorheological systems.

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Information about the authors:

Artem A. Eliseev – Department of Chemistry, Lomonosov Moscow State University, Leninskie Gory, 1b3, Moscow 119991, Russia; ORCID 0000-0002-3644-9673; artem.a.eliseev@gmail.com, aeliseev@inorg.chem.msu.ru

Zitian Xia – Department of Materials Science, Lomonosov Moscow State University, Leninskie Gory, 1b73, Moscow 119991, Russia; ORCID 0009-0000-3487-0301; xiazitian1999@gmail.com

Alexey V. Lukashin – Department of Materials Science, Lomonosov Moscow State University, Leninskie Gory, 1b73, Moscow 119991, Russia; Kurnakov Institute of General and Inorganic Chemistry of the Russian Academy of Sciences, Moscow, 119991 Russia; ORCID 0000-0002-2418-0689; lukashinav@my.msu.ru

Pavel E. Kazin – Department of Chemistry, Lomonosov Moscow State University, Leninskie Gory, 1b3, Moscow 119991, Russia; ORCID 0000-0002-1415-2190; kazin@inorg.chem.msu.ru

Andrei A. Eliseev – Department of Materials Science, Lomonosov Moscow State University, Leninskie Gory, 1b73, Moscow 119991, Russia; ORCID 0000-0002-2851-6821; eliseev@inorg.chem.msu.ru

Conflict of interest: the authors declare no conflict of interest.