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The effect of ions doping on the rheological properties of ferrite ferrofluids

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A series of ferrite nanoparticles were synthesized via ion doping and then were coated by surfactant and dispersed in perfluorinated polyether oil (PFPE-oil), and the various ferrite ferrofluids were obtained. The scanning electron microscope was used to characterize the morphology of particles and the dispersed state of ferrofluid, energy-dispersive spectroscopy was used to study the chemical composition of particles, fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis were used to study the coated effect of PFPE-acids on particles, vibrating sample magnetometer was used to research the magnetization curves of ferrite particles, and the rheological property of the ferrite ferrofluids was studied by a rheometer. The results show that Zn^{2+}, Mn^{2+}/Zn^{2+}, and Dy^{3+} ions were doped in the ferrite nanoparticles with a size less than 50 nm. The four kinds of ferrite nanoparticles have the characteristics of super-paramagnetic materials, and the *M-T* curves decrease with increasing temperature, while their decline rates are notably different. The ferrite particles are coated with PFPE acids chemically, and the ferrofluids have well dispersion stability. The rheological properties of the ferrite ferrofluids change with the variation of ion doping, magnetic field strength, temperature, etc. The magnetism and viscosity of ferrite ferrofluids are regularly affected by ion doping, and the results will have a great significance on basic research and related applications.

KEYWORDS

ion doping, ferrite nanoparticle, magnetization curve, viscosity, ferrofluid

1 Introduction

Ferrofluid is a kind of colloidal solution, which is synthesized through dispersing magnetic nanoparticles coated by surfactant in base liquid (Chen et al., 2018). Under a magnetic field, ferrofluids have both magnetism and mobility and thus are widely used in sealing, biomedicine, aerospace, military, etc. (Cunha et al., 2018; Chen et al., 2019; Nilankush et al., 2019; Chen et al., 2021a; Taghizadeh et al., 2021; Li et al., 2022). The rheological properties of ferrofluid, such as viscosity-temperature, magneto-viscous, and viscosity-shear rate, greatly affect the related application effects, such as sealing, biomedicine, and damping (Hong et al., 1997; Hosseini et al., 2012; Wang et al., 2012; Chand et al., 2013; Chen et al., 2021b). The relationship between the rheological property and magnetic field strength was researched by Odenbach, and the experimental law was confirmed to be consistent with the simulated law (Odenbach and Raj, 2000). Stork revealed that the viscosity of ferrofluid under a magnetic field was larger and bigger than that of the fluids under no magnetic field (Odenbach and Stork, 1998). It was reported by Ruoyu Hong that the viscosity of water-based ferrofluids was related to nanoparticle concentration and shear rate (Hong et al., 2007). The relationship between the rheological property of the ferrofluid, shear rate, and magnetic field strength was revealed by Cuifeng Jiang (Jiang et al., 2008). Ly H. V. found that when the nanoparticle concentration of paraffin-based ferrofluids was too high, the peak value of the viscosity curve appeared, which was caused by the phase separation (Ly et al., 1999). Saeid Atashrouz revealed that the relationship between the rheological property of alcohol-water-based ferrofluid and temperature was consistent with the theoretical prediction (Atashrouz et al., 2016). It follows that the rheological properties of ferrofluids are related to particle nanoparticles, base liquid type, shear rate, magnetic field strength, and temperature.

What is more? Hongchao Cui revealed that the viscosity-temperature of PFPE oil-based ferrofluid was affected by base liquid type (Cui and Li, 2019). Bai Le showed that the silicon oil-based ferrofluid had superior stability of viscosity-temperature (Bai et al., 2017). J. R. Cantow (Cantow et al., 1987) and Z. K. Li (Li et al., 2017) reported that the viscosity of PFPE oil-based ferrofluid decreased gradually with increasing temperature, attributing to the viscosity-temperature characteristics of base liquid. In addition, the rheological property of ferrofluids was related to the magnetism temperature of magnetic nanoparticles, which was reported by S. Odenbach (Odenbach and Stork, 1998).

Ion doping has a great effect on the magnetism temperature of ferrite nanoparticles and their ferrofluids. The $Mn_{1-x}Zn_xFe_2O_4$ ferrofluids had temperature-sensitive characteristics, and their saturation magnetization was reduced with rising temperature (Zhuang et al., 2012). The $Mn_{1-x}Zn_xFe_2O_4$ ferrofluid with temperature-sensitive characteristics was obtained by Z. H. Liao, and its curie temperature was reduced with increasing ion doping (Liao et al., 2011). Yibiao Chen revealed that the saturation magnetization of ferrofluids reduced with increasing temperature (Chen, 2019).

Nevertheless, the effect of ion doping on the rheological properties of ferrite ferrofluids has not been reported. Therefore, in the paper, a series of ferrite nanoparticles are synthesized by ion doping, the micro-structures and magnetism of the particles are characterized, and the influence of magnetism temperature of the ferrite particles on the rheological properties is revealed.

2 Experimental

2.1 Preparation of ferrite nanoparticles and ferrofluids

Briefly, ferric chloride ($FeCl_3{\cdot}6H_2O$), ferrous sulfate ($FeSO_4{\cdot}7H_2O$), zinc chloride ($ZnCl_2$), manganese chloride ($MnCl_2{\cdot}4H_2O$), dysprosium chloride ($DyCl_3$), sodium hydroxide (NaOH), and hydrochloric acid (HCl) were purchased from Alfa Aesar Chemical Reagent Co., Ltd. (Shanghai, China). PFPE oil (F-$(C_3F_6O)_n$-C_2F_5, M_w = 4600g/moL) and PFPE acids (C_3F_7O-$(C_3F_6O)_n$-$C_3F_4O_2H$, M_w = 3800g/moL) were purchased from Shanghai ICAN Chemical S&T Co., Ltd. (Shanghai, China). All chemicals were of analytical grade and were used without any further purification.

TABLE 1 The ferrite nanoparticles.

Sample	Ferrite nanoparticles	Doping ratio *x*
1	Fe_3O_4	—
2	$Zn_xFe_{3-x}O_4$	0.2
3	$Mn_{1-x}Zn_xFe_2O_4$	0.2
4	$Dy_xFe_{3-x}O_4$	0.05

In a typical procedure, the $Zn_xFe_{3-x}O_4$ (x = 0.2) ferrite nanoparticles were synthesized via a chemical co-precipitation method by the following steps: firstly, $FeCl_3{\cdot}6H_2O$ (0.032 M, 8.66 g, 99%), $FeSO_4{\cdot}7H_2O$ (0.013 M, 3.56 g, 99%), and $ZnCl_2$ (0.003 M, 0.43 g, 99.99%) were dissolved in ultrapure water (600 mL) and stirred at 300r/min and 70°C. Secondly, NaOH (0.200 M, 8.00 g, and 99.99%) was added to the solution until the pH reached 12.0; meanwhile, mechanical agitation was carried out. After 30 min, the pH of the solution was adjusted to be ~6.5 by adding HCl solution, and subsequently, PFPE acids (0.001 M, 3.00 g, 99%) were added to coat particles. One hour later, the particles were cleaned with ultra-pure water until the pH of the solution reached ~7.0, and the particles were separated by a magnet. Next, particles were dried in an oven for 10 h and were ground to a powder-like state using a mortar. Finally, the particles were dispersed by mechanical agitation at 500 r/min for 10 h, and the ferrofluids were synthesized, respectively. Fe_3O_4, $Mn_{1-x}Zn_xFe_2O_4$, and $Dy_xFe_{3-x}O_4$ ferrite nanoparticles and their ferrofluids could be prepared in the same way as described above, and the doping ratio x is shown in Table 1. A similar method has been reported by the author (Chen et al., 2018).

2.2 Characterization methods

The morphology of particles and the dispersed state of ferrofluids were characterized by scanning electron microscopy (SEM, Phenom Pharos G2); the energy-dispersive spectroscopy (EDS) was united to analyze the chemical composition of particles. Fourier Transform Infrared Spectrometer (FTIR, Nicolet iS10) spectra were used over the wavenumber range of 400-4000 cm^{-1} to investigate the interactions between surfactant and particles. The coated mass of PFPE acid on the particles was performed using a thermogravimetric analysis analyzer (TGA, TG209F1) from RT to 600°C with a heating rate of 10 C/min in an argon atmosphere. A vibrating sample magnetometer (VSM, Lake Shore 7410) was used to test the magnetic characterization of nanoparticles at an external magnetic field ranging from $-{\times}2.510^4$ Oe to $+2.5{\times}10^4$ Oe at 25°C. The magnetism temperature (M-T) of ferrite of nanoparticles was measured in the temperature range of 5–400 K, $3.0{\times}10^4$ Oe. The rheological properties of ferrite ferrofluids were investigated using a rotational rheometer (MCR 301, Anton-Paar), which generates an external magnetic field in a direction perpendicular to shear flow. A cone-plate system of non-magnetic metal with a diameter of 20 mm was used, and the gap was set to 0.5 mm.

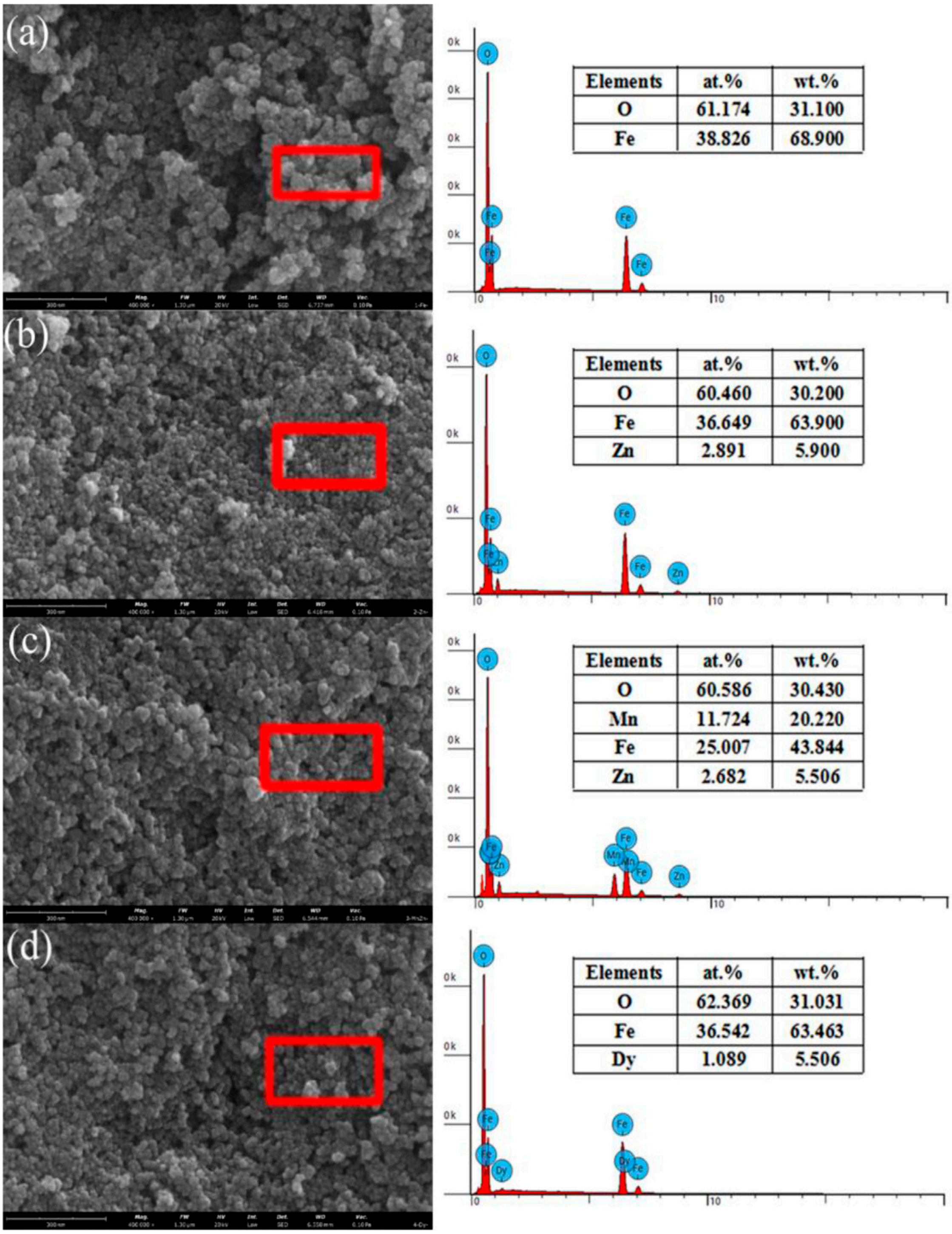

FIGURE 1
The SEM and EDS images of the particles: **(A)** Fe_3O_4, **(B)** $Zn_xFe_{3-x}O_4$, **(C)** $Mn_{1-x}Zn_xFe_2O_4$, and **(D)** $Dy_xFe_{3-x}O_4$.

3 Results and discussion

3.1 The micro-structure and the chemical composition of particles

Figure 1 shows the SEM images and EDS images of particles. In Figure 1, the morphology of the four kinds of particles is nearly spherical, and the size of the particles is less than 50 nm. From the EDS results, it can be seen that Zn^{2+}, Mn^{2+}/Zn^{2+}, and Dy^{3+} have been doped in the ferrite particles. Fe_3O_4 was partly oxidized and the atom ratio between Fe and O was deviated at 3/4. The atom ratio between Zn and Fe in the $Zn_xFe_{3-x}O_4$, the atom ratio between Mn and Zn in the $Mn_{1-x}Zn_xFe_2O_4$, and the atom ration between Dy and Fe in the $Dy_xFe_{3-x}O_4$, respectively, are all close to the raw material ratio, indicating the successful ion doping in particles.

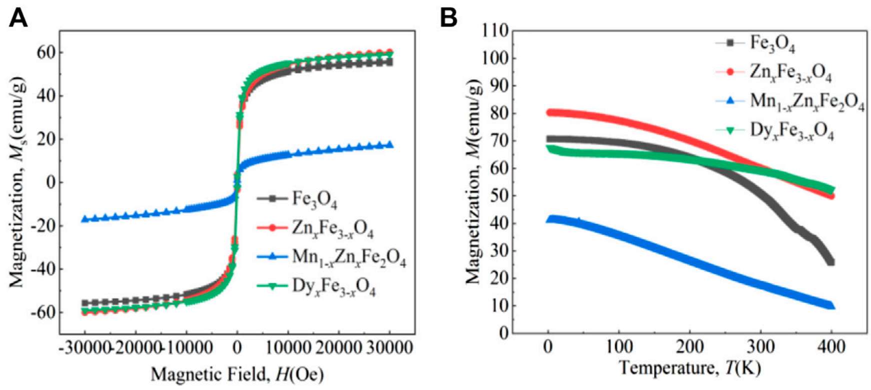

FIGURE 2
The magnetic curves of the ferrite nanoparticles: **(A)** at room temperature and **(B)** at a variable temperature.

Figure 2 shows the magnetization curves of the four kinds of ferrite nanoparticles. From the VSM curves tested at RT (Figure 2A), the magnetization of particles becomes saturated when the applied magnetic field magnitude reaches 30,000 Oe. Moreover, the four kinds of particles have the characteristics of super-paramagnetic material; the magnetization curves are "S" shape and their coercivity is very little. The distribution of ions in ferrite particles is generally represented by the formula $(Fe^{3+})_A$ $[Fe^{2+}Fe^{3+}]_BO_4$, where the parentheses and square brackets refer to tetrahedral and octahedral voids, respectively (Srivastava et al., 2018). The four kinds of particles are ordered by the size of their saturation magnetization as $Zn_xFe_{3-x}O_4$, $Dy_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$, which is because the condition of ions occupied in A and B was changed by doped ions, and the net magnetic moment was also changed. What is more? The magnetism value is also affected by the type of doping ions, doping mass, particle size, and particle size distribution (Chen et al., 2021a).

From the *M-T* curves of the four kinds of particles tested at 30,000 Oe (Figure 2B), the saturation magnetism of particles reduces with a heating temperature from 1.8 K to 400 K. Research has shown that the magnetization of particles becomes saturated when the field magnitude reaches 30,000 Oe at RT. In a stronger magnetic field, presumably, we could tell that the magnetization of the ferrite nanoparticles will not change significantly. In a weaker magnetic field, the *M-T* curve will change, and the value of magnetism is probably lower than that of the particle tested under the same temperature. To avoid unknown and complex influences of the field magnitude, we chose to test the *M-T* curves at 30,000 Oe.

Not only that, the rate of decrease $k = \Delta M/\Delta T$ of the M-T curves is obviously different, which is related to the Curie temperature of the ferrite nanoparticles. The Curie temperature was related to the interaction effect of A-A, A-B, and B-B, of which the effect of A-B interaction on the Curie temperature was the strongest (Eltabey et al., 2011). It has been reported that the induction of Gd^{3+} ions into the Fe_3O_4 nanoferrite matrix results in the weakening of Fe^{3+}(A)–O_2−–Gd^{3+}(B) superexchange interactions, which are responsible for the lower Curie temperature (Thorat et al., 2016). What is more? The decline rate of the M-T curve is affected by particle size, morphology of particles, particle size distribution, type of ion doping, doping mass of ions, etc., which is very complicated. For the four different kinds and masses of ions doping, it is very difficult to explain the M-T decline rate of the four different kinds of ferrite nanoparticles. The four kinds of particles are ordered by the size of their saturation magnetization as $Dy_xFe_{3-x}O_4$, $Zn_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$ at ~348 K.

3.2 The FTIR and TGA of magnetic nanoparticles

Figure 3 shows the TGA and FTIR of coated nanoparticles. From Figure 3A, the particles could be chemically coated by surfactant PFPE acids so that the stretching vibrations of C=O from COOH in the PFPE acid shift from 1777cm^{-1} to 1685cm^{-1} in coated particles using PFPE acid, which may be due to the red-shift effect of the covalent bonding with the bare charge on particles (Chen et al., 2018). The stretching vibrations of uncoated ferrite nanoparticles often appear in low peak positions (Mozaffari et al., 2010; Cui et al., 2015). In Figure 3B, ion doping has little effect on the coated mass of particles, and the four kinds of particles are ordered by their coated mass as $Mn_{1-x}Zn_xFe_2O_4$, $Dy_xFe_{3-x}O_4$, Fe_3O_4, and $Zn_xFe_{3-x}O_4$.

3.3 The SEM images of the four kinds of ferrite ferrofluids

Figure 4 shows the SEM images of the four kinds of ferrite nanoparticle ferrofluids. From the images, the four kinds of ferrite particles were relatively uniformly dispersed in the PFPE oil. The particles were arranged in a short chain length, and no obvious clusters occurred. This was because of the magnetic attraction and repulsion by the coated surfactant between particles (Chen et al., 2021a).

3.4 The rheological properties of various ferrite nanoparticle ferrofluids

Figure 5 shows the rheological properties of the four kinds of ferrite ferrofluids with and without a magnetic field at RT. In

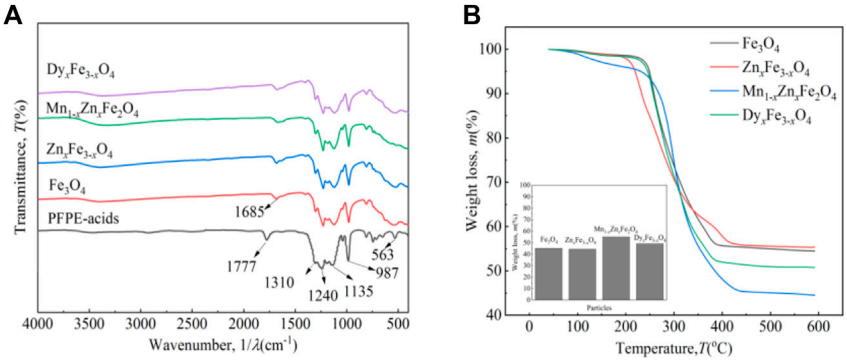

FIGURE. 3
The FTIR **(A)** and TGA **(B)** of the coated ferrite nanoparticles.

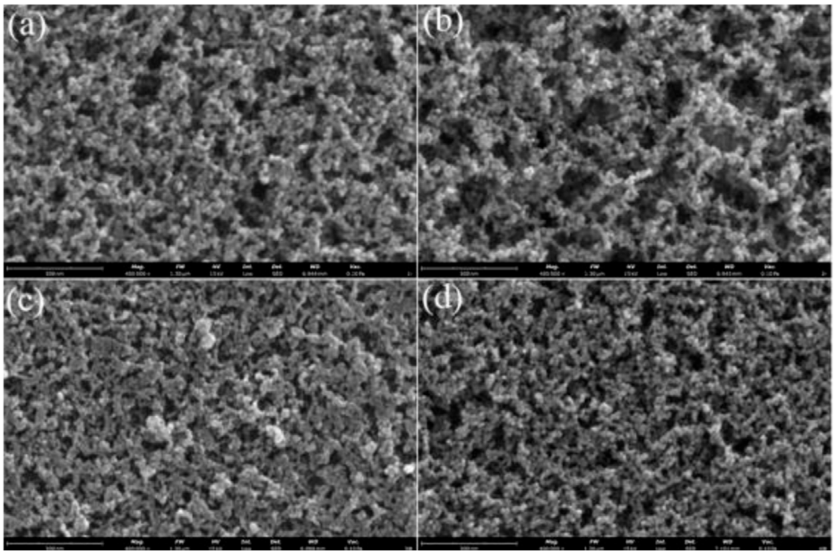

FIGURE 4
The SEM images of the four kinds of ferrite nanoparticles ferrofluids (400000X): **(A)** Fe_3O_4, **(B)** $Zn_xFe_{3-x}O_4$, **(C)** $Mn_{1-x}Zn_xFe_2O_4$, and **(D)** $Dy_xFe_{3-x}O_4$.

Figure 5A, without a magnetic field, by increasing the shear rate, the viscosity of Fe_3O_4, $Zn_xFe_{3-x}O_4$, and $Mn_{1-x}Zn_xFe_2O_4$ ferrofluid is almost unchanged; nevertheless, the viscosity of the $Dy_xFe_{3-x}O_4$ ferrofluid firstly decreases and then becomes flat, and shear thinning appears, which is attributed to the arrangement of nanoparticles and PFPE oil molecules along the shear direction (Chen et al., 2021b). The viscosity of ferrite ferrofluids is ordered by the particle type as $Dy_xFe_{3-x}O_4$, $Zn_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$, which is affected by the particle size, the coated mass of surfactant, and the particle type (Jain et al., 2022).

In Figure 5B, when the applied magnetic field strength is 200 mT, with an increasing shear rate, the viscosity of Fe_3O_4 and $Mn_{1-x}Zn_xFe_2O_4$ ferrofluid is almost unchanged; nevertheless, the viscosity of the $Zn_xFe_{1-x}O_4$ and $Dy_xFe_{3-x}O_4$ ferrofluid firstly decreases and then becomes flat, and shear thinning appears. The viscosity of ferrite ferrofluids is ordered by the particle type as $Zn_xFe_{3-x}O_4$, $Dy_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$. It has been shown that the order of the viscosity size of the four kinds of various ferrofluids is the same as the order of the saturation magnetization of corresponding particles at RT. In other words, the type of the ferrite nanoparticle has an effect on the viscosity of the ferrite ferrofluid. Moreover, the viscosity of ferrofluids under a magnetic field is larger than that of the fluids without a field, attributed to the arrangement of micro-structures which will stop the shear action (Chen et al., 2021b). When the applied magnetic field is 200 mT (2000 Oe), the magnetization of particles and ferrofluids has not reached saturation. It has been reported that when the magnetic field strength is less than 200 mT, the viscosity is mainly affected by the field strength, and when the applied field is big enough, the viscosity is mainly affected by the shear rate (Chen et al., 2021b). When the magnetic field is bigger than 200 mT, the viscosity-shear rate curves will make a difference. It has been shown that viscosity is affected by many effects, such as

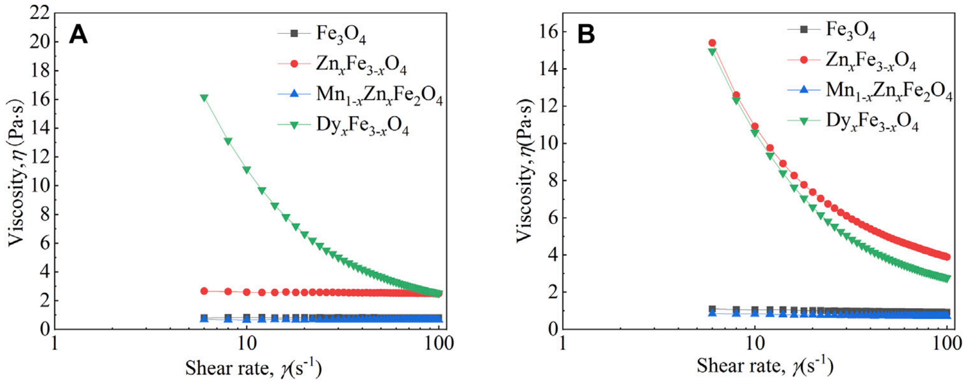

FIGURE 5
The viscosity of the ferrofluids as a function of shear rate:**(A)** 0 mT and **(B)** 200 mT.

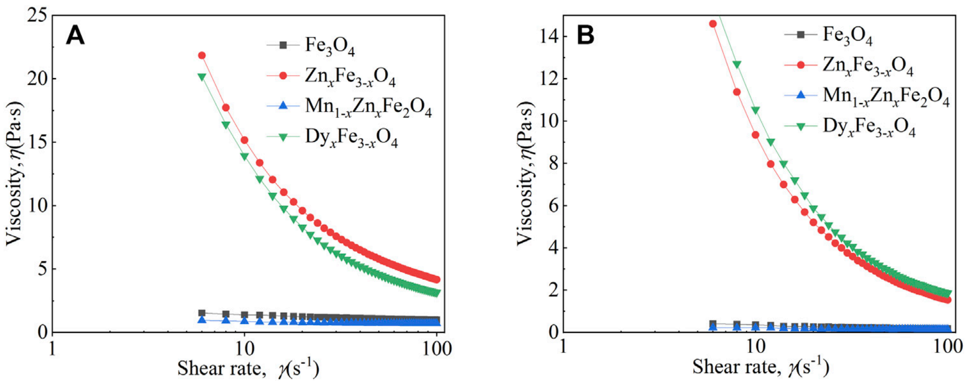

FIGURE 6
The viscosity of the ferrofluids as a function of shear rate: **(A)** 298 K and **(B)** 348 K.

mass of ion doping, particle size, and particle morphology; therefore, it is very difficult to forecast the change of the four kinds of ferrite ferrofluids. Extensive testing of rheological properties is required before the application of ferrofluids.

Figure 6 shows the viscosity of the four kinds of ferrite ferrofluids as a function of shear rate under different temperatures at 400 mT. In Figure 6A, with increasing shear rate, the viscosity of Fe_3O_4 and $Mn_{1-x}Zn_xFe_2O_4$ ferrofluid decreases slightly; however, the viscosity of $Zn_xFe_{1-x}O_4$ and $Dy_xFe_{3-x}O_4$ ferrofluid decreases greatly. It had been reported that the viscosity stability of ferrofluid was required in some applications (Bai et al., 2017). At 298 K, the viscosity of the ferrofluids is higher than that of the fluids at 348 K (as shown in Figure 6B). In addition, the change rate of viscosity is different, which is affected by the particle type. At 298 K, the viscosity size of ferrite ferrofluids is ordered by the particle type as $Zn_xFe_{3-x}O_4$, $Dy_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$. However, at 348 K, the order is changed to be $Dy_xFe_{3-x}O_4$, $Zn_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$, which is the same as that of the saturation magnetization of ferrite nanoparticles. We can infer that the magnetism-temperature of magnetic nanoparticles will affect the viscosity of ferrite ferrofluid.

Research revealed that the force caused by magnetic field strength on magnetic nanoparticles is obtained through the following equation (Odenbach and Storkb, 1998):

$$F_m = \frac{\mu_0 M_0^2 \pi d^6}{24\,(d + 2b)^4}$$

Where M_0 is the magnetization of nanoparticles and it reaches saturation with an increasing magnetic field, d is the average particle size, b is the shell thickness of coated nanoparticles, and μ_0 is the permeability of nanoparticles. According to the equation, the force caused by F_m increases with increasing M_0^2, and meanwhile, the particles arrange to be chain-like structures and even become columns along the magnetic field direction. Research has revealed that the F_m will affect the rheological properties of ferrofluids. According to the equation, the M-T curve of ferrite nanoparticles will affect the force caused by magnetic field strength F_m, and thus the rheological properties are affected by the type of ferrite nanoparticles.

In addition, when the magnetic field is larger than 400 mT, the magnetization of particles will reach saturation. The effect of ion doping in particles on the viscosity-temperature will tend to be a fixed value. When the magnetic field is lower than 400 mT, the effect of ion doping in particles on the viscosity-temperature may be similar so that the magnetization of the particle does not reach saturation. A lot of measurements of rheological properties should be done before the ferrofluids are used in viscosity related applications.

4 Conclusion

Four kinds of ferrite nanoparticles and ferrofluids are obtained through ion doping, and the ferrofluids have high dispersion stability. At 298 K, the size of the saturation magnetization of the four kinds of ferrite particles is ordered by ferrite type as $Dy_xFe_{3-x}O_4$, $Zn_xFe_{3-x}O_4$, Fe_3O_4, and $Mn_{1-x}Zn_xFe_2O_4$. The saturation magnetization of particles reduces with heating temperature from 1.8 K to 400 K. Not only that, the decline rate of $k = \Delta M/\Delta T$ is different and greatly affected by ion doping. The kind and mass of doped ions will change the superexchange interactions of A-B, which are responsible for the lower Curie temperature and decline rate of M-T curves. The type and mass of doped ions will change the magnetism-temperature of ferrite nanoparticles, which further affects the viscosity and its variation of the ferrofluid. What is more? The viscosity is also affected by magnetic field strength, temperature, particle size, etc. It is very difficult to predict the change of the four kinds of ferrite ferrofluids. A lot of measurements should be done before the ferrofluids are used in applications.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding authors.

Author contributions

FC: Writing–original draft. JZ: Writing–review and editing. XL: Supervision, Writing–review and editing. SY: Project administration, Writing–review and editing. WL: Writing–review and editing. ZY: Project administration, Writing–review and editing. ZL: Supervision, Writing–review and editing.

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Conflict of interest

Author ZY was employed by Zigong Zhaoqiang Sealing Products Industrial Co., Ltd.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Publisher's note

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