Study on the Fluidity Experiments of Engine Oil-Based Magnetic Fluid with Fe₃O₄/Ag Nanoparticles

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Abstract. In this research, a new engine oil-based Fe₃O₄/Ag magnetic fluid is prepared applying the method of modified chemical co-precipitation. This preparation method is green without toxic gases being released. The nanoparticles are characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM) and vibration sample magnetometer (VSM). These characterizations demonstrate that the Fe₃O₄/Ag nanoparticles modified by oleic acid are successfully synthesized and uniformly dispersed in the engine oil. The hysteresis loop of this new magnetic fluid shows that it has no remnant magnetism and maintains superparamagnetic properties. Shear stress and viscosity of both Fe₃O₄ magnetic fluid and Fe₃O₄/Ag magnetic fluid are measured by the rotational rheometer. The shear stress increases with the increasing shear rate while the viscosity decreases with the increasing shear rate. What’s more, the viscosity of Fe₃O₄/Ag magnetic fluid significantly increases compared with the traditional magnetic fluid.

1. Introduction
Magnetic fluid is a kind of colloidal solution formed by magnetic (Fe₃O₄ or β–Fe₂O₃) nanoparticles uniformly dispersing in a carrier liquid. Combining the properties of solid and liquid materials, magnetic fluid maintains the properties of both fluidity and magnetic properties[1, 2]. As a functional material, magnetic fluid has aroused considerable interest for its unique properties which have been used in a wide range of applications, such as sealing[3], lubrication[4], heat transfer enhancement[5], magnetic separation[6], biomedicine[7] and so on. Besides, magnetic fluid is also a symmetrical system under zero external magnetic fields. However, when it is placed in a magnetic field, aggregations of Fe₃O₄ or β–Fe₂O₃ nanoparticles elongated along the field direction due to the strong inter-particle interactions which changed the magnetic fluid into an anisotropic system[8, 9]. In this research, Fe₃O₄ is selected as magnetic nanoparticles due to the superparamagnetic property at room temperature. When the size falls down to nanoscale, the thermal energy will overcome the anisotropy energy barrier of a single domain.

In recent years, Fe₃O₄/metallic nanocomposites have attracted increasing interest in the field of magnetic fluid due to their combined plasmonics, magneto-optical, physicochemical and anti-bacterial properties in catalyst[10], biotechnology[11] and biomedicine by offering merits to overtake the limits of conventional magnetic nanoparticles[12]. Meanwhile, Fe₃O₄/metallic nanoparticles can alter the viscosity of magnetic fluid due to the change of the microstructure of magnetic nanoparticles. The
improved viscosity property of magnetic fluid is important for sealing in mechanical engineering, especially under high-speed rotation conditions. In this condition, the temperature of magnetic fluid will increase with the high-speed rotating shaft. When the temperature exceeds a certain value, the sealing performance of magnetic fluid will be greatly reduced. The common method to solve this problem is to adding cooling water circulation devices in the system. However, cooling water needs to be replaced frequently, which is troublesome for engineers. Another method is to prepare Fe3O4/metallic (such as silver or copper) magnetic fluid which has good electrical and thermal conductivity that can increased the reliability and service life in sealing[13].

Some research has been reported about the preparation and properties of noble metallic directly coating on Fe3O4/metallic nanoparticles using sol-gel synthesis[14], co-precipitation[15], sonochemical reactions[16] and so on. Among these methods, co-precipitation is the simplest and most efficient approach to obtain a large amount of aqueous dispersions nanoparticles[17]. One of the most efficient method is the sequential growth of metallic components onto the surface of the Fe3O4 core. However, due to the difference of the two surfaces and lattice, stable nanoparticles are difficult to obtain[18]. Another common method to prepare Fe3O4/metallic nanoparticles include multi-step methods such as the seed-mediated method, resulting in aggregated core-shell and multilayers or hybrid Fe3O4/metallic structures[19]. However, the synthesis leads to a low products yield because of the complex and time-consuming process is the bottleneck for this route[18].

In this research, silver is selected as composite particles because of its low reactivity with Fe3O4 nanoparticles. However, the hydrophobic surfaces of Fe3O4 nanoparticles makes them tend to agglomerate to clusters and their strong magnetic dipole-dipole interaction will degrade the stability and physical behavior of magnetic fluid[20]. What’s more, preparing Fe3O4/Ag magnetic nanoparticles is difficult to realize due to the stability of noble metals. Despite these difficulties, Fe3O4/Ag nanoparticles and engine oil are combined to prepare a new magnetic fluid in this research. A modified method of co-precipitation is applied to synthesize this new magnetic fluid by reducing silver nitrate solution using formaldehyde solution. The obtained Fe3O4/Ag nanoparticle structure can be modified with different charges, functional groups or moieties on the surface to improve stability and compatibility. In order to improve the stability and compatibility, oleic acid isimpolyed in the surface treating process to increase the dispersibility of nanoparticles in the engine oil. Oleic acid retains the polar active and molecular chains which have a good compatibility with engine oil. The polar activity can be attached to the surface of the nanoparticles and the molecular chains of the surfactant have good compatibility with the engine oil.

In addition, the viscosity of magnetic fluid plays an important role in different application fields. Therefore, more and more researchers pay attention to this property of the magnetic fluid. In this research, the viscosity of this new magnetic fluid is also measured by the rotational rheometer. The results show that the shear stress increases with the increasing shear rate while the viscosity decreases with the increasing shear rate. What’s more, the viscosity of Fe3O4/Ag magnetic fluidsignificantly increases compared with the traditional magnetic fluid.

2. Materials and experiments

2.1. Materials
Ferric chloride hexahydrate (FeCl₃·6H₂O), ferrous chloride tetrahydrate (FeCl₂·4H₂O), silver nitrate (AgNO₃), ammonia solution (25%), formaldehyde solution (CH₂O), oleic acid and engine oil are purchased in Beijing Chemical Reagents Company (China). All chemical reagents used in the experiments are analytical reagent grade without further purification. Ultrapure water prepared by PSDK1-20-C is used throughout the experiments.

2.2. Experiments
A common method to prepare magnetic fluid is chemical co-precipitation. In this method, a mixture of salts suspended in an aqueous alkaline medium is prepared. Subsequently, different procedures such as
decantation, magnetic separation, centrifugation and dilution are applied to the suspension[21, 22]. In this research, a modified method of co-precipitation was adopted to prepare the Fe₃O₄/Ag engine oil-based magnetic fluid. Figure 1 illustrates the flow chart of Fe₃O₄/Ag magnetic fluid preparation process. Firstly, 27.05g ferric chloride hexahydrate and 9.95g ferrous chloride tetrahydrate were dissolved in 300ml ultrapure water under mechanical stirring at the temperature of 60°C ~ 70°C. Then 75 ml of ammonia solution was added rapidly to the mixture solution with mechanical stirring and reaction for 15minutes. Ammonia solution was added to 100 ml 0.1mol/l silver nitrate solution by drop. After stirring, the mixed silver ammonia solution were added into the mixture solution along with formaldehyde solution. The mixture solution was heated to 60°C. As indicated in Figure 1, to further modify the particles, oleic acid used as the surfactant was added to the mixture solution and stirred for 1h. Black precipitates were obtained by centrifugation or magnetic sedimentation through a permanent magnet. The precipitates were washed with ultrapure water until the PH decreased to 7 and dried in a vacuum for 36h at 60°C. Then, 2g modified Fe₃O₄/Ag nanoparticles were dispersed in 20ml engine oil by ball milling or ultrasonic dispersion for 12h at room temperature to get raw product. Finally, Fe₃O₄/Ag magnetic fluid was obtained through centrifugation or magnetic sedimentation.

Figure 1. Flow chart of Fe₃O₄/Ag magnetic fluid preparation process.
2.3. Characterization

The morphology was characterized with Hi-Tech 7700 Transmission Electron Microscope (TEM) and Hitachi SU-8010 Scanning Electron Microscope (SEM). X-ray Diffraction (XRD) characterization was measured by a D8 Advance Bruker AXS diffractometer at 40 kV, 100mA using a Cu-target tube and a graphite monochromator. Scans were made in the 2θ range of 20-80° with a step size of 0.2° and a count time of 2s per step. The qualitative analysis of the XRD patterns were performed using the PDF-2 reference database from the International Center for Diffraction Data database. Magnetic properties were measured by a Vibration Sample Magnetometer (VSM, Lakeshore 7307) at room temperature. Shear stress and viscosity experiments were carried out employed the Anton Paar MCR 302 rotational rheometer.

3. Results and discussion

3.1. XRD analysis

One of the most widely used application of X-ray diffraction is to qualitatively analyze the phase of the sample to be tested. The qualitative analysis of the phase is based on the structure of the crystal. Crystallized substance has its specific structural parameters, including the number of atoms (ions or molecules) in the unit cell and their position, unit cell size, lattice type and so on. These parameters can be reflected in the X-ray diffraction pattern. Therefore, characteristics of the substance can be determined according to the position, number, and intensity of the polycrystalline diffraction lines. If there are several mixture substances, the result is a superposition of the diffraction lines of each individual phase. According to this principle, it is possible to retrieve each individual phase one by one from the diffraction pattern of the mixture substances.

![XRD patterns](image)

**Figure 2.** XRD patterns of Fe3O4 nanoparticles and Fe3O4/Ag nanoparticles.

In this research, both Fe3O4 nanoparticles and Fe3O4/Ag nanoparticles are investigated by XRD patterns to explain the crystalline nature. XRD patterns of Fe3O4 nanoparticles and Fe3O4/Ag nanoparticles are illustrated in Figure 2. As shown in Figure 2, peaks at 2θ=30.1°, 35.5°, 43.1° and 56.9° can be indexed to (220), (311), (400) and (511) crystalline planes respectively which are in good agreement with the structure of Fe3O4. The pattern of Fe3O4/Ag nanoparticles shows peaks at 20
values of 38.2° and 44.6° corresponding to the reflections of the (111) and (200) crystalline planes of Ag. Oleic acid has no effect on the crystal structure of Fe₃O₄/Ag nanoparticles because oleic acid is a kind of amorphous polymer materials which cannot be reflected in the diffraction pattern. The intensity of the diffraction line of each phase increases with the increasing content of the phase. Thus, the intensity of Fe₃O₄ in the Fe₃O₄/Ag nanoparticles decreases possibly because of the existence of silver.

3.2. TEM & SEM images
Morphology of Fe₃O₄/Ag nanoparticles is characterized by TEM and SEM. The particles were dispersed in ultrapure water under ultrasonic treatment for 30 minutes. Then a drop of the colloidal suspensions was dripped on a carbon-coated Cu grid and allowed to dry before observation. Images are shown in Figure 3 that the small Ag particles adhered to the surface of Fe₃O₄. There is a slight aggregation among the particles, which are due to the small size and high surface energy of the nanoparticles. What’s more, it is observed that the surface of Fe₃O₄/Ag nanoparticles was not smooth which can increase the viscosity of magnetic fluid. This conclusion is consistent with the results in the following experiments.

![Figure 3. (a)TEM and (b)SEM image of Fe₃O₄/Ag nanoparticles in the magnetic fluid.](image)

![Figure 4. Hysteresis loops of (a) Fe₃O₄ nanoparticles and (b) Fe₃O₄/Ag nanoparticles.](image)
3.3. Magnetic properties
Saturation magnetization ($M_s$) is one of the most important parameters of magnetic fluids measured by vibration sample magnetometer (VSM). The VSM is based on the principle of electromagnetic induction. One of the commonly used methods is to make a small amplitude vibration between the detecting coils by using only a small amount of the sample. The sample can be regarded as a magnetic dipole and the detecting coils sense the change value of the magnetic dipole field caused by the vibration of the sample. Thus, the induced electromotive force of the coils can be derived proportional to the magnetization of the sample.

Hysteresis loops of $Fe_3O_4$ nanoparticles and $Fe_3O_4/Ag$ nanoparticles measured by VSM at 300 K are shown in Figure 4. $M_s$ of $Fe_3O_4/Ag$ nanoparticles reduced slightly. They still have no remanent magnetism properties yet. The reduction in $M_s$ may be due to the decrease in magnetic particle density because units for magnetization are reported per gram of materials. The energy of magnetic materials in an external magnetic field is proportional to the number of magnetic molecules in a single magnetic domain. This decrease reflects a smaller percentage of net magnetic material per gram of overall sample. The large surface-to-volume ratio of $Fe_3O_4/Ag$ nanoparticles are possibly another factor that leads to the decrease in $M_s$.

3.4. Relationship between shear stress and shear rate
Viscosity is an important property of magnetic fluid. Many applications of magnetic fluid, especially in the sealing field, are closely related to the viscosity. In the current study, the viscosity of magnetic liquid are mainly measured by rheometers. There are mainly two types of rheometers: the commercial rheometer equipped with magnetic field module and self-designed magnetic field rheometer. The self-designed rheometer can adjust the structure of the rheometer according to the shape of the magnetic field generated by the coil. The uniformity of the magnetic field in the self-designed rheometers is higher than the commercial rheometer. However, it is reduced in the control and measurement accuracy compared with the commercial rheometer. The commercial rheometer equipped with the magnetic field module can optimize the uniformity of the magnetic field under the condition of a certain magnetic field strength, which has become a common choice for studying the viscosity of magnetic fluid.

In this research, the shear stress and the viscosity of magnetic fluid is measured by the Anton PaarPhysica MCR 302 high-precision rotational rheometer equipped with MRD170 magnetic field module. MCR 302 belongs to CMT type rheometer and is driven by synchronous electronic rectification (EC) motor. There is a linear correlation between the output torque of synchronous electronic rectification motor and the input current of stator coil. This is beneficial to accurately measure and control the output torque of the motor. The rotating parts are fixed and supported by high-precision air bearings in the axial and radial directions, respectively. The rheometer has a minimum controllable torque of 1nN·m and a minimum take-up time of 1ms, which can ensure the accurate measurement of the experiments.

For the accuracy of the experiment, both the $Fe_3O_4$ engine oil-based magnetic fluid and the $Fe_3O_4/Ag$ engine oil-based magnetic fluid have been pre-sheared for about 30s at a shear rate of 50 1/s. Data are not recorded in this pretreatment process. Then, the relationship between shear stress and shear rate of both the $Fe_3O_4$ engine oil-based magnetic fluid and the $Fe_3O_4/Ag$ engine oil-based magnetic fluid are measured at 20$^\circ$C with the shear rate varying from 1 1/s to 100 1/s. Figure 5 and 6 illustrate the relationship between the shear stress and the shear rate of both $Fe_3O_4$ engine oil-based magnetic fluid and $Fe_3O_4/Ag$ engine oil-based magnetic fluid under different magnetic fields. Figure 5 shows the fitting curves of the shear stress and the shear rate of (a) $Fe_3O_4$ engine oil-based magnetic fluid and (b) $Fe_3O_4/Ag$ engine oil-based magnetic fluid at 0.25 A ($\approx$500 Gs) magnetic field strength and 0.5 A ($\approx$1000 Gs) magnetic field strength and figure 6 is the fitting curves of the shear stress and the shear rate of $Fe_3O_4$ engine oil-based magnetic fluid and $Fe_3O_4/Ag$ engine oil-based magnetic fluid at (a)0.25 A ($\approx$500 Gs) magnetic field strength and (b)0.5 A ($\approx$1000 Gs) magnetic field strength. The trend of the two magnetic fluids is consistent. The shear stress rises with the increasing shear rate.
When the magnetic field strength varies from 0.25A to 0.5A, the shear stress of Fe₃O₄ engine oil-based magnetic fluid rises from 4.59 Pa to 9.22 Pa and the shear stress of Fe₃O₄/Ag engine oil-based magnetic fluid rises from 6.34 Pa to 10.83 Pa at the highest speed of 100 1/s. The change value of these two magnetic fluid is 4.63 Pa and 4.49 Pa, respectively. On the other hand, when the magnetic field strength is 0.25A, the shear stress of Fe₃O₄ engine oil-based magnetic fluid is 4.59 Pa while the shear stress is 6.34 Pa in the Fe₃O₄/Ag engine oil-based magnetic fluid. When the shear rate reaches to the highest speed of 100 1/s, the shear stress of this two magnetic fluids becomes 9.22 Pa and 10.83 Pa, respectively. This can be explained by the unique microstructure of magnetic fluid. The chain/column structure formed by the magnetic nanoparticles in the magnetic field under the external magnetic field becomes smaller as the shear rate increases. The deformation of the microstructure becomes larger leading to the increasing shear stress. As shown in Figure 3, the surface of Fe₃O₄/Ag nanoparticles are rougher than Fe₃O₄ nanoparticles which will enhance the shear stress.

![Figure 5](image1.png)

**Figure 5.** Fitting curve of the shear stress and the shear rate of (a)Fe₃O₄ engine oil-based magnetic fluid and (b)Fe₃O₄/Ag engine oil-based magnetic fluid at 0.25A(≈500Gs) magnetic field strength and 0.5A(≈1000Gs) magnetic field strength.

![Figure 6](image2.png)

**Figure 6.** Fitting curve of the shear stress and the shear rate of Fe₃O₄ engine oil-based magnetic fluid and Fe₃O₄/Ag engine oil-based magnetic fluid at (a)0.25A(≈500Gs) magnetic field strength and (b)0.5A(≈1000Gs) magnetic field strength.

3.5. **Relationship between viscosity and shear rate**

Experiments of the relationship between the viscosity and the shear rate are also measured when the shear rate ranges from 1 1/s to 100 1/s. Figure 7 and 8 reveal the viscosity of both Fe₃O₄ engine oil-
based magnetic fluid and Fe₃O₄/Ag engine oil-based magnetic fluid changing with the shear rate. Figure 7 is the viscosity of (a)Fe₃O₄ engine oil-based magnetic fluid and (b)Fe₃O₄/Ag engine oil-based magnetic fluid at 0.25A(≈ 500Gs) magnetic field strength and 0.5A(≈ 1000Gs) magnetic field strength. Figure 8 is the viscosity of Fe₃O₄ engine oil-based magnetic fluid and Fe₃O₄/Ag engine oil-based magnetic fluid at (a)0.25A(≈ 500Gs) magnetic field strength and (b)0.5A(≈1000Gs) magnetic field strength. When the magnetic field strength increases from 0.25A to 0.5A, the viscosity of Fe₃O₄ engine oil-based magnetic fluid increases from 45.96 mPa·s to 92.24 mPa·s and the viscosity of Fe₃O₄/Ag engine oil-based magnetic fluid varies from 63.42 mPa·s to 108.34 mPa·s. The change value of these two magnetic fluids is 46.28 mPa·s and 44.92 mPa·s, respectively. The applied magnetic field reveals a strong influence on the viscosity of the two magnetic fluids. The viscosity of the magnetic fluid is not only determined by the viscosity of engine oil but also by the interaction between the magnetic nanoparticles. When there is an external magnetic field, the viscosity of the magnetic fluid increases. The external magnetic field enhances the interaction among the magnetic nanoparticles and the chain/column structure is formed in the magnetic field resulting in the increasing viscosity.

![Figure 7](image1.png)

**Figure 7.** The viscosity of (a)Fe₃O₄ engine oil-based magnetic fluid and (b)Fe₃O₄/Ag engine oil-based magnetic fluid at 0.25A(≈500Gs) magnetic field strength and 0.5A(≈1000Gs) magnetic field strength.

![Figure 8](image2.png)

**Figure 8.** The viscosity of Fe₃O₄ engine oil-based magnetic fluid and Fe₃O₄/Ag engine oil-based magnetic fluid at (a)0.25A(≈500Gs) magnetic field strength and (b)0.5A(≈1000Gs) magnetic field strength.
On the other hand, an increase in viscosity caused by the Fe$_3$O$_4$/Ag nanoparticles is also observed over the test range of shear rate. When the magnetic field strength is constant, the viscosity of Fe$_3$O$_4$/Ag engine oil-based magnetic fluid is larger than that of the Fe$_3$O$_4$ engine oil-based magnetic fluid with the value of 17.46 mPa·s at 0.25A magnetic field strength and 16.10 mPa·s at 0.5A magnetic field strength.

In addition, the viscosity of these two magnetic fluids decreases as the shear rate increases which demonstrates the shear thinning phenomenon in both Fe$_3$O$_4$ engine oil-based magnetic fluid and Fe$_3$O$_4$/Ag engine oil-based magnetic fluid. With the increase of shear rate, shear thinning started to occur. In the shear rate region of 0-20 1/s, the viscosity decreases rapidly. The viscosity still decreases in the shear rate region of 20-100 1/s while the rate of decline is slightly slower than 0-20 1/s.

4. Conclusions
To enhance the viscosity of magnetic fluid, Fe$_3$O$_4$/Ag engine oil-based magnetic fluid is prepared by a modified method of co-precipitation. The experiments between shear stress and shear rate of both Fe$_3$O$_4$ engine oil-based magnetic fluid and Fe$_3$O$_4$/Ag engine oil-based magnetic fluid are measured under different magnetic field strength. The shear stress of Fe$_3$O$_4$ engine oil-based magnetic fluid rises from 4.59 Pa to 9.22 Pa and the shear stress of Fe$_3$O$_4$/Ag engine oil-based magnetic fluid rises from 6.34 Pa to 10.83 Pa at the highest speed of 100 1/s when the magnetic field strength is 0.25A and 0.5A. The change value of these two magnetic fluid is 4.63 Pa and 4.49 Pa, respectively. In addition, when the magnetic field strength increases from 0.25A to 0.5A, the viscosity of Fe$_3$O$_4$ engine oil-based magnetic fluid increases from 45.96 mPa·s to 92.24 mPa·s and the viscosity of Fe$_3$O$_4$/Ag engine oil-based magnetic fluid varies from 63.42 mPa·s to 108.34 mPa·s. The change value of these two magnetic fluids is 46.28 mPa·s and 44.92 mPa·s, respectively. Both these two magnetic fluid exhibit shear thinning. The results illustrate that Fe$_3$O$_4$/Ag nanoparticles not only increase the viscosity of magnetic fluid but also keep the magnetic fluid more stable as the magnetic field strength changes.

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