



Preparation and Property Research of Perfluoropolyether Oil-Based Ferrofluid

Hongchao Cui¹ · Decai Li¹

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Abstract

In this study, perfluoropolyether oil-based ferrofluid was prepared to satisfy special demands in applications which are too harsh to conventional ferrofluids. Fe₃O₄ magnetic nanoparticles were synthesized by co-precipitation and modified by perfluoropolyether carboxylic acid surfactant in the atmosphere without protective gas. Perfluoropolyether oil-based ferrofluid was prepared with modified nanoparticles and perfluoropolyether by high-energy ball milling. Properties of ferrofluid fabricated were verified through a series of experiments. Ferrofluid stability was tested by interval point-density method; the average density change is merely 0.0077 g/ml, and change rate is 0.37% after 500 days. Saturation magnetization is 582.46 Gs, much larger than the other four ferrofluids. The average volatility rate is 7.4985×10^{-5} g/h/cm² at 80 °C after 260 h. Viscosity increases to 1825.7 mPa·s under −20 °C and still have mobility at −40 °C. The ferrofluid can resist air blast for 8 h under 200 °C without oxidation or decomposition. Perfluoropolyether oil-based ferrofluid cannot be damaged by water or organic solvents. In addition, the ferrofluid can survive in pH = 1 acid or pH = 14 alkali at least 279 days without corrosion.

Keywords Ferrofluid · Magnetic fluid · Perfluoropolyether · Fe₃O₄ · Acids and alkali resistance

1 Introduction

Ferrofluids are called smart functional nanomaterials because of their exceptional characteristics, establishing concurrent magnetic and fluid properties. As magnetically controllable and tunable fluids, ferrofluids have high academic value and wide applications. After decades of research, ferrofluid such as water-based ferrofluid [1–5], kerosene oil-based ferrofluid [6, 7], ester-based ferrofluid [8–10], diester-based ferrofluid [11, 12], silicon oil-based ferrofluid [13], etc., have reached a maturity practical stage; the applications from machinery [14–17], electronics [18, 19], biomedicine [20–27], optics [28–31], chemicals [32],

and energy [33–35] extended to military [36] and other fields [37, 38]. With application expansion, ferrofluid working conditions and environment became more and more complex, such as vapor compression distillation device in space station, station cantilever windows, motor shaft of aircraft carrier motor, key equipment of nuclear explosion, large-scale wind tunnel, and other key projects in sealing requirements. Conventional ferrofluids are strongly temperature dependent, generally used from 20~90 °C. They cannot withstand extreme temperatures like < −40 °C or > 300 °C and cannot seal acids, alkali, or oxidation because they are prone to react with sealing medium in contact. In addition, they cannot resist high pressure and radiation; stability is poor under exceptional working conditions, so their applications are restricted. In our early research, Fe₃O₄ magnetic nanoparticles were successfully modified by perfluoropolyether carboxylic acid surfactant to get over the disadvantage above, and the performance has been greatly improved. The work has been published in Mater Lett [39]. In this study, we further synthesize perfluoropolyether oil-based ferrofluid basis on a preliminary work. The prepared

✉ Hongchao Cui
09116323@bjtu.edu.cn

¹ State Key Laboratory of Tribology, Department of Mechanical Engineering, Tsinghua University, Beijing 100084, People's Republic of China

ferrofluid is truly stable to acids, alkali, oxidation, and reduction even at high temperatures, and other unique properties are expected to satisfy applications. Their outstanding chemical and thermal stabilities permit perfluoropolyether oil-based ferrofluid to be applied in more extensive applications such as space station, aerospace systems, and nuclear burst, where it is too severe to survive for conventional ferrofluids.

2 Experimental

2.1 Chemicals

Ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and ferric trichloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) are purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. HCl, NaOH, absolute ethyl alcohol, diethyl ether, acetone, and ammonium hydroxide (25%) are procured from Beijing Chemical Industry Group Co., Ltd. Perfluoropolyether carboxylic acid surfactant and perfluoropolyether were obtained from Dupont. The chemicals were used as received without further purification. Water used in all experiment processes is deionized water, which is refined and filtered by an ultrapure water machine (PSDK2-20-C) in the lab.

2.2 Procedure

Preparation process of perfluoropolyether oil-based ferrofluid is divided into three parts: bare Fe_3O_4 magnetic nanoparticles (MNPs), modified Fe_3O_4 MNPs, and ferrofluid. Bare Fe_3O_4 MNPs were prepared by coprecipitation method according to $\text{Fe}^{2+} + 2\text{Fe}^{3+} + 8\text{OH}^- = \text{Fe}_3\text{O}_4 + 4\text{H}_2\text{O}$ in nonprotective atmosphere. Considering that Fe^{2+} would be partially oxidized into Fe^{3+} , the molar ratio of $\text{Fe}^{2+}/\text{Fe}^{3+}$ was adjusted to 1:1.75. Twenty-five percent ammonia serving as precipitant was added into the mixed solution of Fe^{2+} and Fe^{3+} at 60 °C with intensive agitation; solution color immediately turned from red brown to black. The mixture was stirred for additional 10 min at 60 °C then heated up to 80 °C. Perfluoropolyether carboxylic acid surfactant was added to the solution and continuously stirred for 2 h at 80°. Modified Fe_3O_4 MNPs were obtained after collecting, rinsing, filtering, and drying. Modified Fe_3O_4 MNPs and perfluoropolyether were adequately ground in certain proportions with high-energy ball milling, strictly controlling grind time and speed. Excess surfactant was added for further coating, if necessary. In order to prevent Fe_3O_4 MNP oxidation when temperature rise up in ball-milling process, circulation cooling was adopted. Crude product was obtained after 6 to 15 h of cumulative ball milling. By placing the crude product on a cylindrical magnet, larger-size particle precipitated down to

the bottom; the upper bright and shiny black liquid is the perfluoropolyether oil-based ferrofluid.

2.3 Apparatus

Perfluoropolyether oil-based ferrofluid was prepared in high-energy ball milling (YF01). Magnetic property was carried out by VSM (Lakeshore 7307), applied magnetic field was from $-10,000$ to $10,000$ Oe. 20 ml perfluoropolyether oil-based ferrofluid and 20 ml water-based ferrofluid were continuously heated at 80 °C under atmospheric pressure. Volatilization rate was tested in a vacuum drying oven (DZF-6050AB). The evaporation dish area was $7.54 \times 10^{-3} \text{ m}^2$. NXE-1B cone-plate viscometer was used to test viscosity-temperature performance of perfluoropolyether oil-based ferrofluid. The heating and cooling processes were both tested at shear rate $D = 3.84n$ (1/S) between -20 and 100 °C in B system. Thermal oxygen performance was tested in high-low-temperature test chamber (WD6003, $-60 \sim 300$ °C). 20 ml perfluoropolyether oil-based ferrofluid and ester-based ferrofluids were continuously heated from 25 to 200 °C with air blast (electrothermal blowing dry box, DGF2503) for 8 h.

3 Results and Discussion

3.1 Stability in Magnetic Field

Many methods can be used to characterize stability of ferrofluids, including L-C inductance vibration circuit method, fixed-point saturation magnetization method, redox method, interval point-density method, and so on. Interval point-density method was adopted due to being an accurate, simple, and easy operation. Taking a 30 ml graduated test tube, 30 ml graduation marks as the first sample point, and every 5 ml decrease for the rest sample point is named 1, 2, 3, ... 6. 30 ml perfluoropolyether oil-based ferrofluid was injected into the graduated test tube, then placed in 5000 Gs applied magnetic field for 7, 15, 30, 60, 90, 150, 200 and 500 days, and density values of the six sample points were tested, as shown in Table 1.

The data in Table 1 is plotted with ORIGIN, as shown in Fig. 1. From density changes, we can get parts of the magnetic particles settled down under the action of magnetic force, density increases at lower sample point, while the upper decreases. However, after 500 days, the average density change is only 0.0077 g/ml, change rate is 0.37%, and the sediment can be ignored. Such small density change is permitted in ferrofluid engineering application. This indicates that the prepared perfluoropolyether oil-based ferrofluid is stable enough in magnetic field.

Table 1 Density at different sample points in magnetic field g/ml

| Days | Sample point 1 | Sample point 2 | Sample point 3 | Sample point 4 | Sample point 5 | Sample point 6 |
|-----------|----------------|----------------|----------------|----------------|----------------|----------------|
| 7 | 2.059 | 2.059 | 2.06 | 2.062 | 2.064 | 2.068 |
| 15 | 2.059 | 2.059 | 2.06 | 2.061 | 2.064 | 2.069 |
| 30 | 2.059 | 2.058 | 2.06 | 2.061 | 2.065 | 2.069 |
| 60 | 2.058 | 2.058 | 2.059 | 2.06 | 2.066 | 2.07 |
| 90 | 2.057 | 2.057 | 2.058 | 2.059 | 2.066 | 2.071 |
| 150 | 2.056 | 2.056 | 2.058 | 2.058 | 2.067 | 2.072 |
| 200 | 2.054 | 2.053 | 2.056 | 2.057 | 2.068 | 2.074 |
| 500 | 2.051 | 2.05 | 2.053 | 2.054 | 2.069 | 2.077 |
| variation | 0.008 | 0.009 | 0.007 | 0.008 | 0.005 | 0.009 |

3.2 Characterization of Magnetic Property

Ferrofluids can respond to applied magnetic field and show magnetic peaks or thorns (Fig. 2). That is because magnetic particles rearrange along the direction of magnetic field lines, causing interface instability. When under the same magnetic field or at the same position, higher magnetic peak shows larger magnetization intensity. The longer time magnetic peak can keep, the more stable ferrofluid is. Magnetic peaks are qualitative judgment of ferrofluid stability.

Water based ferrofluid, engine oil-based ferrofluid, kerosene-based ferrofluid, ester-based ferrofluid and perfluoropolyether oil-based ferrofluid were tested at room temperature, listed in Table 2, and plotted in Fig. 3. Saturation magnetization is calculated based on $M_s=4\pi\rho\sigma_s$.

From Fig. 3, the five magnetization curves are all S-shaped, which means that the five kinds of ferrofluids are all superparamagnetism. The initial magnetization curves of these five ferrofluids are all approximate straight lines. Initial magnetic susceptibility can be calculated from $\chi =$

M/H . Initial magnetic susceptibility above 0 is also a characteristic of paramagnetism. Magnetization of the five ferrofluids all gradually increased with applied magnetic field until reaching a certain value, and basically no longer grow even up to 10,000 Oe. This means that magnetization has reached saturation substantially. Saturation magnetization of perfluoropolyether oil-based ferrofluid is 582.46 Gs, much larger than the other four, indicating that dosage of perfluoropolyether oil-based ferrofluid is least but efficiency is highest in ferrofluid applications among the five, which can realize lightweight sealing structure in engineering component design. Coercivity and remanence of perfluoropolyether oil-based ferrofluid is 2.3252 Gs and 0.26474 emu/g, respectively, slightly larger than the other four, but they can be ignored in application. Initial magnetic susceptibility of perfluoropolyether oil-based ferrofluid is 3.35831, which is the maximum of the five. Magnetic susceptibility is a physical quantity, indicating magnetization degree in applied magnetic field. The higher the initial magnetic susceptibility, the more easily magnetized, and the more sensitive to magnetic field, the shorter response time. This is particularly important in ferrofluid sealing and sensor application.

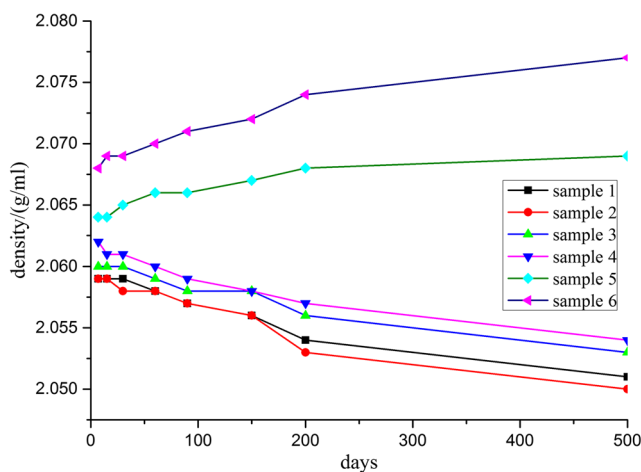


Fig. 1 Density changes at different sample points in magnetic field



Fig. 2 Peak pattern of perfluoropolyether oil-based ferrofluid in applied magnetic field

Table 2 Magnetic properties of five kinds of ferrofluids

| Kinds | H_c (G) | Br (emu/g) | σ_s (emu/g) | χ (M/H) | ρ (g/cm ³) | M_s (Gs) |
|---|-----------|------------|--------------------|--------------|-----------------------------|------------|
| Water-based ferrofluid | 0.60767 | 0.03113 | 17.602 | 1.17129 | 1.272 | 281.22 |
| Kerosene-based ferrofluid | 0.61177 | 0.01878 | 15.541 | 0.49654 | 1.054 | 205.74 |
| Engine oil-based ferrofluid | 0.58041 | 0.02400 | 23.384 | 1.06166 | 1.248 | 366.54 |
| Ester-based ferrofluid | 0.61963 | 0.01149 | 12.846 | 0.30971 | 1.241 | 200.23 |
| Perfluoropolyether oil-based ferrofluid | 2.3252 | 0.26474 | 22.833 | 3.35831 | 2.031 | 582.46 |

3.3 Volatilization

20 ml perfluoropolyether oil-based ferrofluid and 20 ml water-based ferrofluid were continuously heated at 80 °C under atmospheric pressure, and weight tests are list in Table 3. Figure 4 shows the two samples taken out 260 h later. Perfluoropolyether oil-based ferrofluid is still black and bright, and magnetization intensity did not reduce obviously either. But water-based ferrofluid has changed into solid, which was completely damaged.

As shown in Table 3, volatility of perfluoropolyether oil-based ferrofluid is only 0.34 g after 260 h at 80 °C. In the first 17 h, volatility is 0.17 g, which is half the total. Although heat continued, volatility did not increase over time. Perfluoropolyether is a mixture of different chain lengths, some low molecular weight organic volatilize at first but volatility do not increase because experiment condition is not strong enough to break the molecular structure of the long chain. Density of perfluoropolyether did not change at 533 K under 275 MPa [40], which also proves that the experimental condition cannot cause a chemical reaction. The maximum volatility rate calculated of perfluoropolyether oil-based ferrofluid is 13.263×10^{-5} g/h/cm², the minimum

is 1.734×10^{-5} g/h/cm², and average is 7.4985×10^{-5} g/h/cm². While water-based ferrofluid has been dried into solid completely when heated at 80° for 4.2 h, it is no longer ferrofluid at all. The volatility rate of water-based ferrofluid is 3924.976×10^{-5} g/h/cm², 295 times that of the maximum value of perfluoropolyether oil-based ferrofluid.

Ferrofluid is composed of magnetic nanoparticles, surfactant, and based liquid. Magnetic particles are solid and do not volatilize; surfactant ratio is small, volatilization is negligible; therefore, volatilization of ferrofluid is mainly determined by based liquid. Based liquid as organics, volatilization can be theoretically calculated by Mazak (B.T.M) formula. Mazak formula is suitable for organic material volatilization in open storage.

$$G = (5.38 + 4.1 u) P_v * F (M)^{0.5}$$

where G is the volatilization of organics (g/h), u is air speed (m/s), P_v is saturated vapor pressure of organic at room temperature (Pa), F is open area of organics (m²), and M is molecular weight of organics.

At different temperatures, P_v can also be calculated according to the following equation:

$$\log \left(\frac{P_v}{133.32} \right) = \frac{-0.05223A}{T} + B$$

where T is temperature and A and B are constants.

Volatilization of ferrofluid prepared by the same based liquid is a function of temperature; the higher the temperature, the larger the volatilization. For different based liquids, volatilization is mainly determined by saturated vapor pressure and molecular weight of based liquid in addition to temperature. Volatilization of ferrofluid can be approximately calculated according to them.

3.4 Low-Temperature Experiment

Ferrofluid should be selected depending on the specific application conditions. Space station, tanks, radar, and other

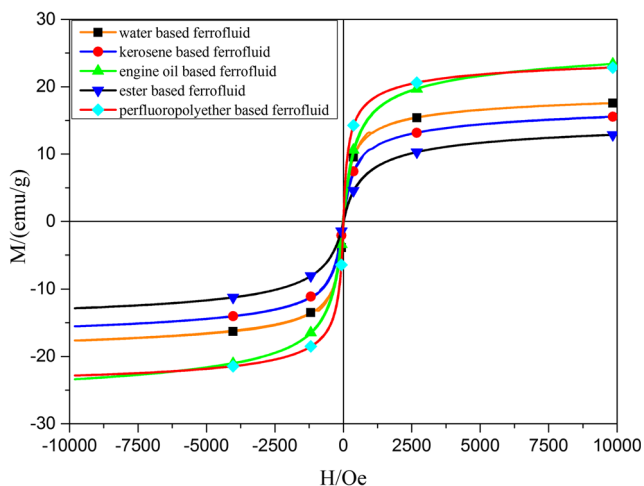
**Fig. 3** Magnetization curves of five kinds of ferrofluids

Table 3 Volatilization of perfluoropolyether oil- and water-based ferrofluids at 80 °C in atmospheric pressure

| Time (h) | Perfluoropolyether oil-based ferrofluid | | Water-based ferrofluid | |
|---------------|---|--|------------------------|--|
| | Residual mass (g) | Volatile quantity (g/h/cm ²) | Residual mass (g) | Volatile quantity (g/h/cm ²) |
| Initial value | 91.24 | – | 75.06 | 4.2 h evaporate to dryness |
| 17 | 91.07 | 13.263*10 ⁻⁵ | 62.63 | 3924.976*10 ⁻⁵ |
| 42 | 91.01 | 7.263*10 ⁻⁵ | 62.62 | – |
| 66 | 90.99 | 5.023*10 ⁻⁵ | 62.61 | – |
| 114 | 90.96 | 3.257*10 ⁻⁵ | 62.59 | – |
| 143 | 90.93 | 2.875*10 ⁻⁵ | 62.58 | – |
| 243 | 90.91 | 1.801*10 ⁻⁵ | 62.52 | – |
| 260 | 90.90 | 1.734*10 ⁻⁵ | 62.50 | – |

military conditions are extremely harsh; the application temperature range demand is wide, such as $-40\sim 200$ °C. Low-temperature fluidity, hightemperature volatilization, and oxidation rate is essential to ferrofluid service life. Based liquid is the main component of ferrofluid; physical and chemical properties are largely determined or affect ferrofluid properties. Pour and flash points of five based liquids are listed in Table 4.

It is possible to roughly speculate the application temperature of ferrofluid prepared by corresponding based liquids from Table 4. Water-based ferrofluid volatility and oxidization increased with temperature and pressure and boiling at 100 °C. When based liquid is lost, ferrofluid turns into Fe₂O₃ solid finally. Water-based ferrofluid freezes and changes into magnetic solid below 0 °C, so water-based ferrofluid is usually used between 0 and 90 °C. Kerosene-based ferrofluid has the narrowest service temperature ranging from -30 to 40 °C, usually used in low temperature. Kerosene volatilizes and mixes with air to form an explosive mixture, and explosive limit is 2–3%. Engine oil-based ferrofluid is usually used between -5 and 150 °C. Special attention should be paid to kerosene-based ferrofluid and engine oil-based ferrofluid to avoid fire or explosion causing personal and instrument damage due to their based liquids



Fig. 4 Phenomena of perfluoropolyether oil-based ferrofluid and water-based ferrofluids after volatilization at 80 °C in atmospheric pressure

belonging to industrial fuel. Ester-based ferrofluid can be used in a wider temperature range, from -45 to 210 °C. When above 248 °C, ester begin to boil and ferrofluid is damaged. Perfluoropolyether oil-based ferrofluid can serve from -60 to 380 °C, especially suited for harsh condition. This ferrofluid can work even thousands of hours above 200 °C without acid or alkaline attack, and it will not be dissolved by any solvent (except chlorofluorocarbon or low viscosity perfluoropolyethers). Perfluoropolyether almost cannot be oxidized and is absolutely not flammable even at high temperature, which itself can be used as a flame retardant leaving no residue; the performance is safe and reliable.

Water-based ferrofluid, ester-based ferrofluid and perfluoropolyether oil-based ferrofluid were subjected to low temperature at -40 °C shown in Fig. 5. From Fig. 5, water-based ferrofluid has fully frozen with white ice on the ferrofluid surface. Ester-based ferrofluid has become a viscous semisolid and hard to flow down at tilted state. Perfluoropolyether oil-based ferrofluid is still black and bright.

Viscosity of perfluoropolyether oil-based ferrofluid was tested at -20 °C in normal pressure. From Fig. 6, we can see that the sample tank has frozen, while ferrofluid is still black and bright. Viscosity rise up from 178.2 mPa·s at room temperature to 1825.7 mPa·s at -20 °C. Fluidity and magnetic properties are not affected, while ester-based ferrofluid no longer flows. There are so many data due to sampling interval being 1 °C, wherein viscosity of -20 , 25, and 100 °C is 1825.7, 178.2, and 13.37 mPa·s; the rest of the dates are no longer listed. The viscosity data are plotted and fitted as shown in Fig. 7.

3.5 Thermal Oxidative Stability

Ester-based ferrofluid is better in heat resistant than other ferrofluids. Perfluoropolyether oil-based ferrofluid and ester-based ferrofluid were taken 20 ml each and continuously heated from 25 to 200 °C with air blast for 8 h. Samples taken out are shown in Fig. 8.

Table 4 Pour point and flash point of five kinds of based liquid

| Parameter | Water | Kerosene | Engine oil | | Ester | Perfluoropolyether |
|------------------|---------------|----------|-----------------|--------------------|----------------------|--------------------|
| | | | 10 [#] | 20~30 [#] | | |
| Molecular weight | 18 | 200~250 | 300~370 | | 426.67 | 2500 |
| Pour point (°C) | 0 | - 30 | - 5 | - 5 | - 45 | - 60 |
| Flash point (°C) | 100 (boiling) | 40 | 130 | 150 | 210 (boiling at 248) | 380 (nonflammable) |

Fig. 5 Apparent state of water based ferrofluid, ester-based ferrofluid and perfluoropolyether oil-based ferrofluid at -40 °C



Fig. 6 Apparent state of perfluoropolyether oil-based ferrofluid at -20 °C

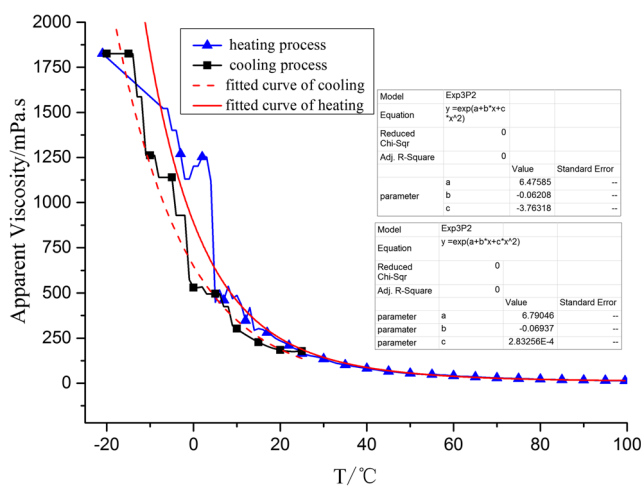


Fig. 7 Viscosity of perfluoropolyether oil-based ferrofluid changed with temperature

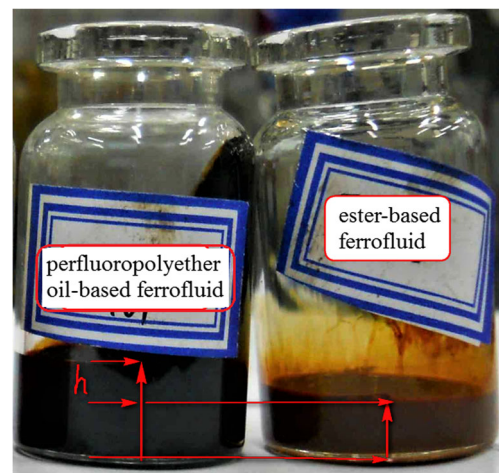


Fig. 8 Volatilization of perfluoropolyether oil-based ferrofluid and esterr-based ferrofluid at 200 °C after 8 h air blast

Table 5 Critical decomposition temperatures of several based liquids

| Kind | Hydrocarbon | Silane | Fluorosilane | Perfluoroether |
|------------------------------|-------------|--------|--------------|----------------|
| Degradation temperature (°C) | 79 | 58 | 82 | 142 |

The color of perfluoropolyether oil-based ferrofluid is still bright black, while ester-based ferrofluid has changed from bright black to reddish brown, which indicates that Fe₃O₄ particles in ester-based ferrofluid have mostly oxidated to Fe₂O₃. As Fe₂O₃ is nonmagnetic, the magnetic property of ester ferrofluid decreased largely, while perfluoropolyether oil-based ferrofluid have not obviously declined. From Table 4, ester-based liquid flash and boiling points are 210 and 248 °C respectively, while based liquid of perfluoropolyether begins to decompose above 380 °C. After 8 h of air blasting and heating, there is a “h” liquid level difference between ester-based ferrofluid and perfluoropolyether oil-based ferrofluid, indicating that volatilization of ester-based ferrofluid is higher than perfluoropolyether oil-based ferrofluid.

The thermal oxidative stability difference between ester-based ferrofluid and perfluoropolyether oil-based ferrofluids can be explained from based liquid and surfactant. Critical decomposition temperatures of some representative-based liquids in the presence of Lewis acid are shown in Table 5. Based liquid of ester-based ferrofluid is hydrocarbon. Hydrocarbon begins to decompose at 79 °C in the presence of Lewis acid. While the initial decomposition temperature of perfluoropolyether is 142 °C from Table 5, we can speculate that ester-based ferrofluid begins to decompose or oxidize at much lower temperature than perfluoropolyether oil-based ferrofluid under the same conditions, which is consistent with the liquid surface difference shown in Fig. 8.

Then analyze thermal oxidative stability of ferrofluid from surfactant. Surfactant of ester-based ferrofluid is oleic acid, and its molecular structure is CH₃(CH₂)₇CH=CH(CH₂)₇COOH. Flash and boiling points of oleic acid are 189 and 350~360 °C. Owing to continuous air blowing at 200 °C, oleic acid oxidized and lost its modification to Fe₃O₄ and Fe₃O₄ oxidized into red brown Fe₂O₃. Oleic acid is flammable and incompatible with strong oxidants. Resins containing 28, 40, and 65% oleic acid respectively were heated, and decomposition temperature decreased when oleic acid increases [41]. This also shows ester-based ferrofluid being damaged under high temperature, resulting from oleic acid decomposition. Perfluoropolyether carboxylic acid surfactant do not decompose or oxidize at 200 °C and modification is not affected, so thermal stability of perfluoropolyether oil-based ferrofluid is much stronger than ester-based ferrofluid.

Table 6 Phenomenon of five ferrofluids blended with solvents

| Ferrofluids | Water | Alcohol | Diethyl ether | Acetone |
|---|---|---|--|---|
| Water-based ferrofluid | Miscible, ferrofluid was weakened to disappear | Miscible, ferrofluid was weakened to disappear | Layered, subnatant was at slurry state, ferrofluid was damaged | Basically miscible, ferrofluid was diluted, magnetism was weakened to disappear |
| Kerosene-based ferrofluid | Layered, clear interface, ferrofluid was not affected | Magnetic particles precipitated | Magnetic particles precipitated | Magnetic particles precipitated |
| Engine oil-based ferrofluid | Layered, clear interface, ferrofluid was not affected | Magnetic particles precipitated | Magnetic particles precipitated | Magnetic particles precipitated |
| Ester-based ferrofluid | Layered, clear interface, ferrofluid was not affected | Layered, subnatant was slurry state, ferrofluid was destroyed | Layered, subnatant was slurry state, ferrofluid was destroyed | Layered, subnatant was slurry state, ferrofluid was destroyed |
| Perfluoropolyether oil-based ferrofluid | Layered, clear interface, ferrofluid was not affected | Layered, clear interface, ferrofluid was not affected | Layered, clear interface, ferrofluid was not affected | Layered, clear interface, ferrofluid was not affected |

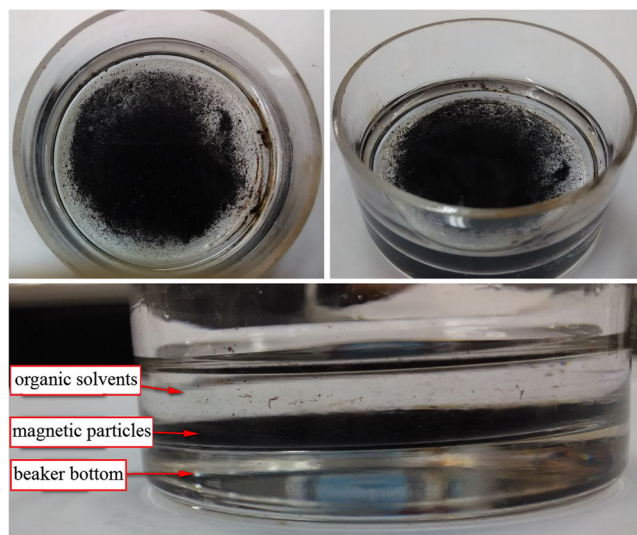


Fig. 9 Phenomenon of kerosene-based ferrofluid and engine oil-based ferrofluids blended with solvents

3.6 Solvent Destruction Experiment

Take 5 ml water-based ferrofluid, kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid and perfluoropolyether oil-based ferrofluid each and mix with water, ethanol, diethyl ether, and acetone, respectively, then observe the phenomenon after 10 min, as listed in Table 6.

Water-based ferrofluid mix with water is equal to dilution with magnetism becoming smaller even disappearing when

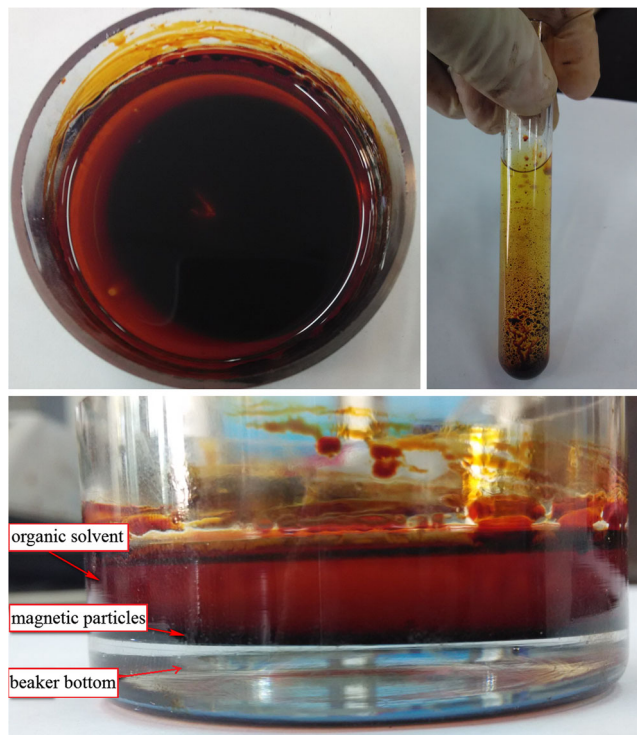


Fig. 10 Phenomenon of ester-based ferrofluid blended with solvents

more water is added, while the other four ferrofluids are immiscible with water, and there is a clear interface, so they are stable with water. Kerosene-based ferrofluid, engine oil-based ferrofluid and ester-based ferrofluid all can be damaged by organic solvents such as ethanol, diethyl ether, and acetone inordinately. The following will be explained separately.

Magnetic particles precipitate directly from kerosene-based ferrofluid and engine oil-based ferrofluids, because their surfactant and based liquid are both hydrocarbon compounds. Based liquids are miscible with organic solvent. Surfactants also can be gradually stripped from Fe_3O_4 surface by organic solvent. When surfactant modification is lost, magnetic nanoparticles can no longer be suspended in based liquid by swinging of surfactant long chain, so magnetic particles precipitated from based liquid at last, as shown in Fig. 9.

There is no obvious magnetic particles precipitating in ester-based ferrofluid. But ester-based ferrofluid is slurry like and no longer uniform because organic solvent permeated into it. Viscosity of ester-based ferrofluid is higher than kerosene-based ferrofluid and engine oil-based ferrofluids, so magnetic particles precipitate more slowly and with more difficulty. Organic solvent molecules mix with the aggregation of magnetic particles as shown in Fig. 10.

Perfluoropolyether oil-based ferrofluid is immiscible with water and organic solvents. There is a clear interface between the two phases, shown in Fig. 11. The stability and immiscibility of perfluoropolyether oil-based ferrofluid make it possible for ferrofluid to seal other liquid medium, which is not solved by other ferrofluids before. It is worth noting that perfluoropolyether oil-based ferrofluid can be dissolved by fluorine solvent or chlorine solvent such as FC_77 and CH_3CCl_3 . The phenomenon is the same as water-based ferrofluid is mixed with water. It is necessary to avoid direct contact with fluorine solvent or chlorine solvent in ferrofluid sealing.

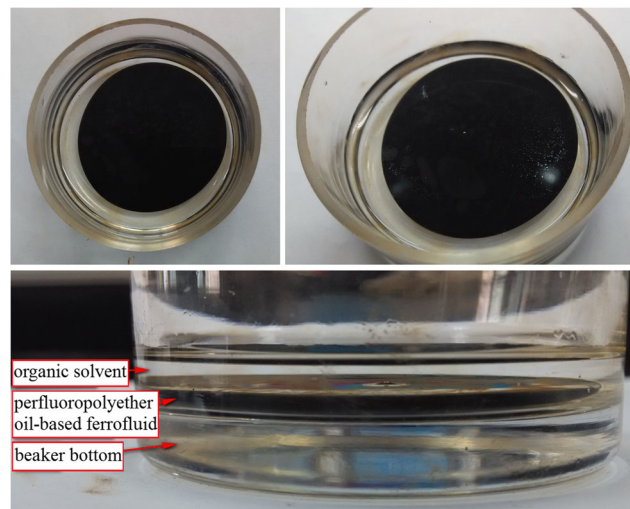


Fig. 11 Phenomenon of perfluoropolyether oil-based ferrofluid blended with solvents

Fig. 12 Phenomenon of water-based ferrofluid corroded at pH = 1 and 14

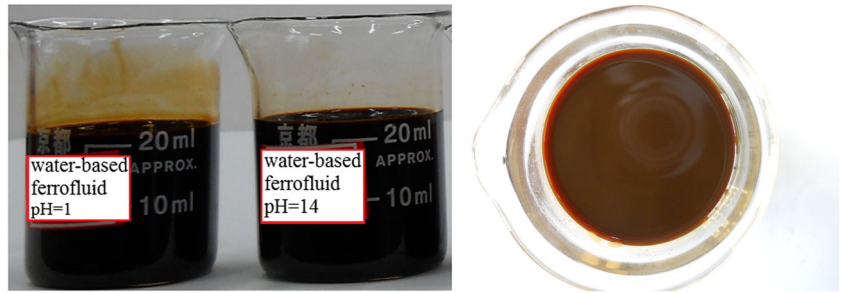


Fig. 13 Phenomenon of five ferrofluids corroded by HCl at pH = 1

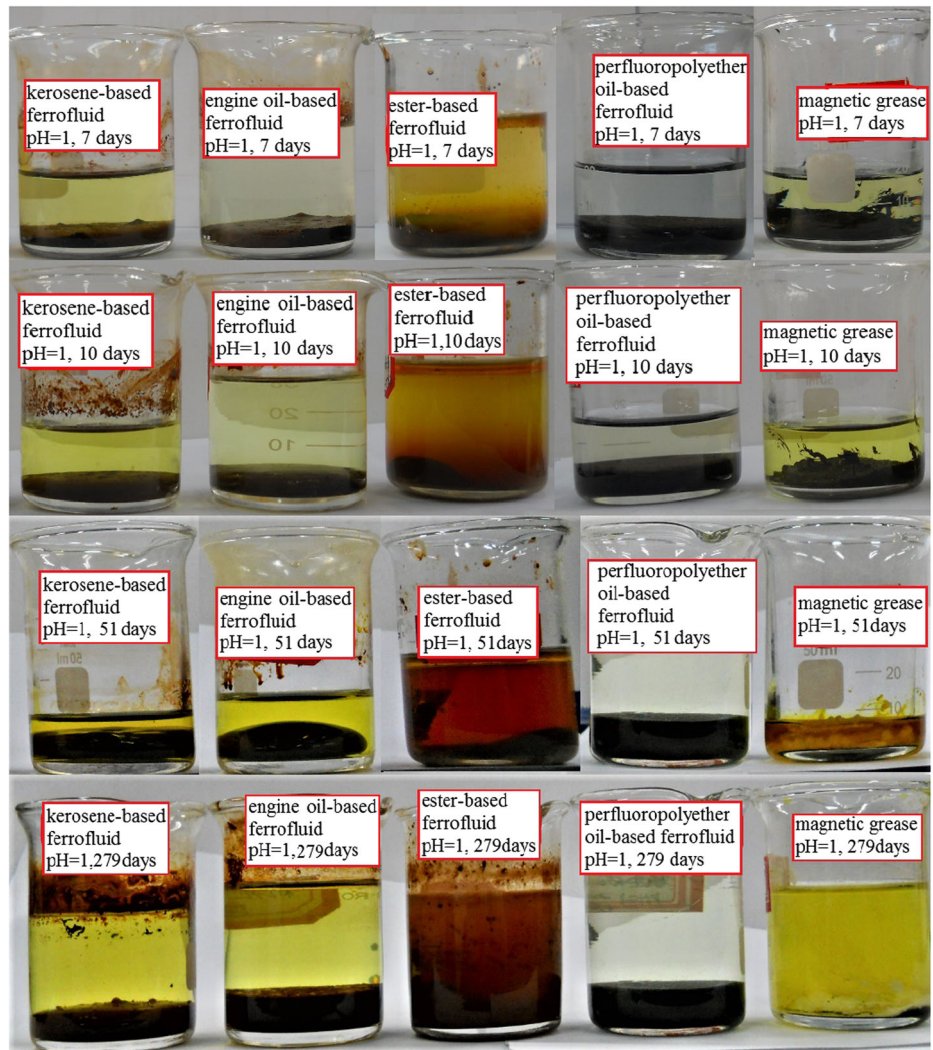


Fig. 14 Kerosene oil-based ferrofluid corroded by HCl at pH = 1 for 7, 51 and 279 days



Fig. 15 Engine oil-based ferrofluid corroded by HCl at pH = 1 for 7, 51 and 279 days

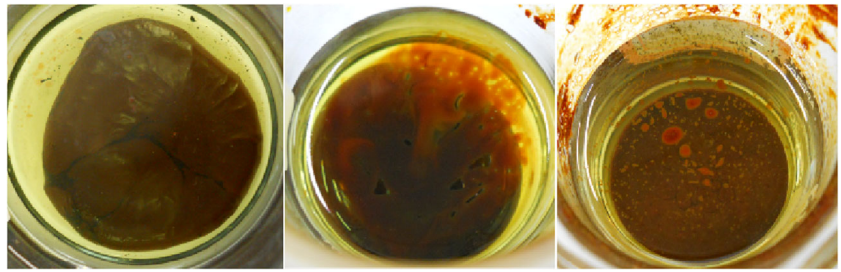


Fig. 16 Ester-based ferrofluid corroded by HCl at pH=1 for 7, 51 and 279 days



Fig. 17 Magnetic grease corroded by HCl at pH = 1 for 7, 51 and 279 days



Fig. 18 Perfluoropolyether oil-based ferrofluid corroded by HCl at pH = 1 for 7, 51 and 279 days



Table 7 Phenomenon of five ferrofluids corroded by HCl at pH = 1

| Ferrofluids | Days | Phenomenon of corrosion at pH=1 |
|-----------------------------|-----------------|--|
| Kerosene-based ferrofluid | Mixed instantly | Immiscible, the upper liquid slowly turned yellowish and transparent, distinct interface, oil-like thin layer on surface, magnetic peak |
| | 7 | Immiscible, light yellow and transparent upper liquid, oil-like thin layer on surface, red brown interface, no magnetic peak but can move with magnetic field |
| | 10 | Immiscible, yellow and transparent upper liquid, oil-like thin layer on surface, red brown interface, no magnetic peak but can move with magnetic field and magnetic decline |
| | 51 | Immiscible, golden yellow and transparent upper liquid, oil-like thin layer on surface, reddish brown gully on interface, no magnetic peak, magnetism decreased obviously but still can move with magnetic field |
| | 279 | Immiscible, dark yellow and transparent upper liquid, oil-like thin layer get together on surface, unclear interface, no magnetic peak, magnetic declined further but still can move with magnetic field |
| Engine oil-based ferrofluid | Mixed instantly | Immiscible, basically colorless and transparent upper liquid, distinct interface, magnetic peaks |
| | 7 | Immiscible, yellowish and transparent upper liquid, oil-like thin layer on surface, distinct interface, no magnetic peak, magnetism declined slightly but can move with magnetic field |
| | 10 | Immiscible, light yellow and transparent upper liquid, oil-like thin layer on surface, red brown soft layer covering on ferrofluid interface, no magnetic peak, magnetism declined but still can move with magnetic field |
| | 51 | Immiscible, bright yellow and transparent upper liquid, oil-like precipitate increased on surface, reddish brown soft interface layer, no magnetic peak, magnetism declined obviously but still can move with magnetic field |
| | 279 | Immiscible, yellow and transparent upper liquid, oil-like precipitate aggregated into bulks on surface, reddish brown interface layer solidification, no magnetic peak, magnetism further declined but still can move with magnetic field, magnetism slightly stronger than kerosene |
| Ester-based ferrofluid | Mixed instantly | Partial miscibility, light yellow and transparent upper liquid, oil-like thin layer on surface, unclear interface, no magnetic peak but can move with magnetic field |
| | 7 | Partial miscibility, dark yellow and transparent upper liquid, oil-like thin layer on surface, unclear interface, no magnetic peak but still can move with magnetic field |
| | 10 | Partial miscibility, dark yellow and transparent upper liquid, oil-like thin layer on surface, unclear interface and rust fine granular, no magnetic peak but still can move with magnetic field |
| | 51 | Partial miscibility, dark reddish brown and transparent upper liquid, sticky floating layer on surface, honeycomb rust hole caused by acid corrosion ferrofluid producing H ₂ on interface, magnetism significantly decreased but still can move with magnetic field |
| | 279 | Partial miscibility, dark brown translucent upper liquid, point floating objects on surface, honeycomb rust hole like coral on interface, magnetism declined further, but hardly move with magnetic field |
| Magnetic grease | Mixed instantly | Immiscible, colorless and transparent upper liquid, distinct interface, magnetic grease attached to beaker bottom, no magnetic peak but can move with magnetic field |
| | 7 | Immiscible, light yellow and transparent upper liquid, distinct interface, magnetism decreased obviously but still can move with magnetic field |
| | 10 | Immiscible, dark yellow and transparent upper liquid, distinct interface, magnetism significantly decreased but still can move with magnetic field |
| | 51 | Immiscible, apricot and transparent upper layer, distinct interface, magnetic grease is pure white, almost no magnetism, magnetic core was dark brown, retained little magnetism |

Table 7 (continued)

| Ferrofluids | Days | Phenomenon of corrosion at pH=1 |
|---|-----------------|---|
| Perfluoropolyether oil-based ferrofluid | 279 | Immiscible, smell, grease changed into white brittle solid, magnetism and viscoelasticity almost completely lost, grease corroded by acid completely |
| | Mixed instantly | Immiscible, distinct interface, colorless and transparent upper liquid, bright black ferrofluid, magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 7 | Immiscible, distinct interface, colorless and transparent upper liquid, bright black ferrofluid, magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 10 | Immiscible, distinct interface, colorless and transparent upper liquid, bright black ferrofluid, magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 51 | Immiscible, distinct interface, light yellow upper liquid, bright black ferrofluid, no magnetic peak but can move with magnetic field, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 279 | Immiscible, distinct interface, light yellow upper liquid, bright black ferrofluid, magnetic decreased slightly, weaker magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |

3.7 Corrosion by pH = 1 Acid and pH = 14 Alkali

In addition to air, water, neutral, and noncorrosive media, strong acid and alkali chemical materials are inevitable in ferrofluid applications. Acid and alkali tolerance test is not only essential for ferrofluid selection according to application condition but also necessary for personal safety and risk control in potential hazard. pH = 1 acid and pH = 14 alkali are prepared with HCl and NaOH. Take 10 ml water-based ferrofluid, kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid and perfluoropolyether oil-based ferrofluid in a small beaker each, add acid and alkali respectively, then stir the mixture and observe the phenomenon over time.

3.7.1 Water-Based Ferrofluid Corroded by pH = 1 Acid and pH = 14 Alkali

Water-based ferrofluid is miscible with pH = 1 acid and pH = 14 alkali in any ratio equal to dilution, and mixture appearance will not change, as shown in Fig. 12. With increase amount of acid or alkali, magnetic particle concentration decreases and magnetism is reduced. When the ratio of acid or alkali to ferrofluid is more than 5:1, water-based ferrofluid will no longer respond to magnetic field. In addition, water-based ferrofluid changed from bright black to dark brown gradually and finally became reddish brown due to corrosion. Fe_3O_4 reacted with HCl into FeCl_2 and FeCl_3 , and Fe_3O_4 partially oxidized to Fe_2O_3 too. Therefore, water-based ferrofluid is easiest to be destroyed by acid and alkali.

3.7.2 Organic Ferrofluid Corroded by pH = 1 Acid

Based liquid of kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid, magnetic grease, and perfluoropolyether oil-based ferrofluid are organics relative to water-based ferrofluid. The organic ferrofluid and magnetic grease corroded by acid or alkali are more complex than water-based ferrofluid. All ferrofluids have magnetic peaks in magnetic field before corrosion. Continuously monitor the phenomenon of mixture surface, two-phase interface, magnetic property corroded by pH = 1 and 14 when the mix instant, 7, 10, 51, and 279 days later and record the results as follows:

Figure 13 shows the front view of ferrofluids submerged in hydrochloric acid solution at pH = 1.

Figure 13 only exhibits the upper liquid color and morphology change after corrosion. Planforms in Figs. 14, 15, 16, 17 and 18 show the mixture surface and two-phase interface contact with acid, which are more visual to compare the changes before and after corrosion.

Fig. 19 Phenomenon of five ferrofluids corroded by NaOH at pH = 14



The phenomenon of kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid, magnetic grease, and perfluoropolyether oil-based ferrofluid corroded by pH = 14 alkali the mix instant, 7, 10, 51, and 279 days later are listed in Table 7.

3.7.3 Organic Ferrofluid Corroded by pH = 14 Alkali

Figure 19 shows the front view of kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid, magnetic grease, and perfluoropolyether oil-based ferrofluid corroded by pH = 14 alkali.

Figure 19 only exhibits the upper liquid color and morphology change after corrosion. Planforms in Figs. 20,

21, 22, 23 and 24 show mixture surface and two-phase interface contact with alkali, which are more visual to compare the changes before and after corrosion.

The phenomenon of kerosene-based ferrofluid, engine oil-based ferrofluid, ester-based ferrofluid, magnetic grease, and perfluoropolyether oil-based ferrofluid corroded by pH = 14 alkali when the mix instant, 7 and 12 days later are listed in Table 8.

By comparison of the phenomenon, organic-based ferrofluid corroded by pH = 1 acid is different from corroded pH = 14 alkali; the specific analysis is as follows:

As shown in Fig. 25, ferrofluid divided into many small droplets after acid added with stir. Then small droplets automatically merged together and restored to liquid phase

Fig. 20 Kerosene oil-based ferrofluid corroded by NaOH at pH = 14 when mixed, 7 and 12 days



Fig. 21 Engine oil-based ferrofluid corroded by NaOH at pH = 14 when mixed, 7 and 12 days

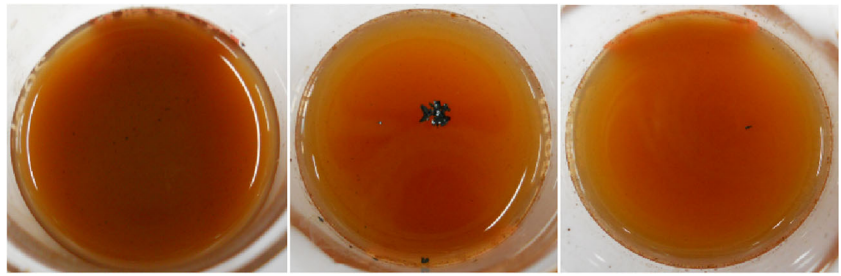


Fig. 22 Ester-based ferrofluid corroded by NaOH at pH = 14 when mixed, 7 and 12 days

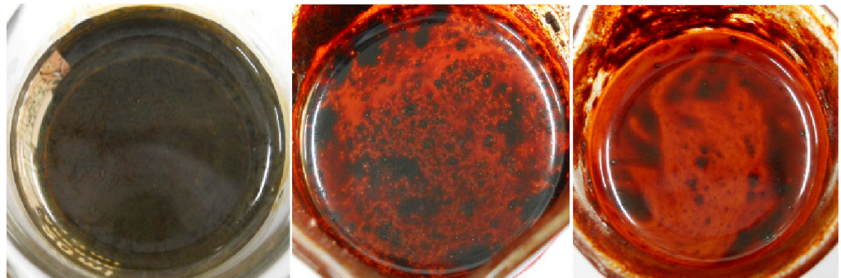


Fig. 23 Magnetic grease corroded by NaOH at pH = 14 when mixed, 7 and 12 days



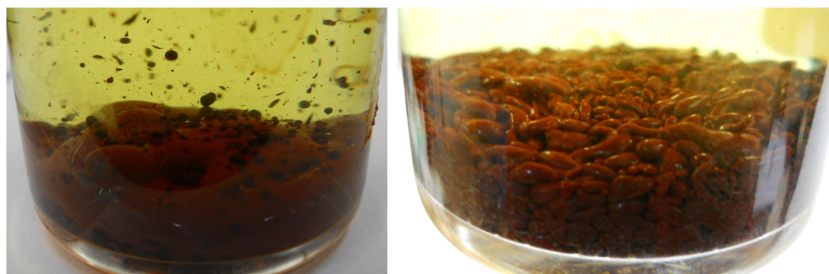
Fig. 24 Perfluoropolyether oil-based ferrofluid corroded by NaOH at pH = 14 when mixed, 7 and 12 days



Table 8 Phenomenon of five ferrofluids corroded by NaOH at pH = 14

| Ferrofluids | Days | Phenomenon of corrosion at pH = 14 |
|---|-----------------|---|
| Kerosene-based ferrofluid | Mixed instantly | The upper liquid immediately turned into reddish brown turbid when mixed instantly and changed into dark brown after oscillation. No flottage on surface, magnetism decreased |
| | 7 | Dark brown turbid upper liquid, oil flottage on surface, magnetic particles precipitated partly from ferrofluid and became brown, magnetism decreased obviously |
| | 12 | Red brown turbid upper liquid, oil flottage on surface, magnetic particles precipitated completely from ferrofluid and became brown and loose, almost no magnetism, ferrofluid was damaged |
| | Mixed instantly | The upper liquid immediately turned into reddish brown turbid when mixed instantly, small amount of black dot floated on surface, magnetic decreased |
| Engine oil-based ferrofluid | 7 | Reddish brown upper liquid, a black sticky flottage on surface, magnetic particles precipitate partly from ferrofluid and become brown, magnetism drop obviously |
| | 12 | Brown turbid upper liquid, oil flottage on surface, magnetic particles precipitated completely from ferrofluid, dark brown magnetic particles formed irregular agglomeration, ferrofluid was damaged, almost no magnetism |
| Ester-based ferrofluid | Mixed instantly | The upper liquid immediately turned into reddish brown after oscillation, small oily materials floated on surface, ferrofluid color at interface became dark brown, unclear interface, magnetism decreased |
| | 7 | Deep red upper liquid, lots of reddish brown sticky material attached to surface and beaker wall, magnetic particles precipitated largely from ferrofluid and became brownish gray, ferrofluid was damaged, magnetism declined |
| Magnetic grease | 12 | Brown upper liquid, lot of red brown sticky material attached to surface and beaker wall, only brown honeycomb and loose magnetic particles not ferrofluid, magnetism declined |
| | Mixed instantly | Immiscible, the upper liquid layer was colorless and transparent, clear interface, magnetic grease attached to beaker bottom, no change after oscillation |
| Perfluoropolyether oil-based ferrofluid | 7 | Immiscible, the upper liquid layer was colorless and transparent, clear interface, magnetic grease attached to beaker bottom, magnetic grease at two phase interface changed from black to gray, many fine holes on magnetic grease surface, magnetism declined |
| | 12 | Immiscible, the upper liquid layer was colorless and transparent, clear interface, magnetic grease attached to beaker bottom, magnetic grease at 2-phase interface from yellow to white, many fine pores on surface, magnetism further weakened |
| | Mixed instantly | Immiscible, colorless and transparent upper liquid, distinct interface, ferrofluid was bright black, magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 7 | Immiscible, colorless and transparent upper liquid, distinct interface, ferrofluid was bright black, magnetic peak, not sticky on beaker wall because of both hydrophobic and oleophobic |
| | 12 | Immiscible, colorless and transparent upper liquid, distinct interface, ferrofluid was bright black, not sticky on beaker wall because of both hydrophobic and oleophobic, dark black ferrofluid protective film at two-phase interface, still magnetic peak |

Fig. 25 Phenomenon of hydrocarbon-based ferrofluid stirred by HCl at pH = 1



forming an obvious interface. The upper phase is transparent after about 10 min of rest, which means that based liquid is immiscible with acid solution. Going through initial corrosion, a protective layer will form at interface to slow down further corrosion to internal ferrofluid.

When alkaline is added, kerosene-based ferrofluid, engine oil-based ferrofluid and ester-based ferrofluid appear reddish brown turbid after mixing instantly. The phenomenon is caused by reaction of NaOH with oleic acid (surfactant), so insoluble magnetic particles lost protection immediately then precipitate into the mixture, as shown in Fig. 26. Alkali cause far more serious damage to ferrofluids than acid, leaving only magnetic particles; ferrofluid is no longer liquid. Acid corrosion rate is slow, and although property is weakened, ferrofluids continue to work until complete acid corrosion.

Furthermore, HCl and NaOH are strong electrolyte, H^+ , Cl^- , Na^+ , and OH^- ions are 100% ionized. The ions directly participate or promote chemical reaction with based liquid, surfactant, or magnetic particles, resulting in ferrofluid damage.

Ferrofluid service life (corrosion at pH = 1) can be predicted according to experimental results above: the life of magnetic grease, ester-based ferrofluid, kerosene-based ferrofluid, and engine oil-based ferrofluid are approximately 10, 35, 50, and 200 days, respectively. Perfluoropolyether oil-based ferrofluid's theoretical life may be longer than 500 days. The ability of resistance to acid cor-



Fig. 26 Phenomenon of hydrocarbon-based ferrofluid stirred by NaOH at pH = 14

rosion: perfluoropolyether oil-based ferrofluid > engine oil-based ferrofluid > kerosene-based ferrofluid > ester-based ferrofluid > magnetic grease > water based ferrofluid.

Magnetic particles precipitate instantly from ferrofluid when contact with pH = 14 NaOH solution, so kerosene-based and engine oil-based ferrofluid are firstly damaged. Service life of ester-based ferrofluid is approximately 7 days and magnetic grease is about 15 days. Perfluoropolyether oil-based ferrofluid has not been destroyed basically during experiment time. The ability of resistance to alkali corrosion: perfluoropolyether oil-based ferrofluid > magnetic grease > ester-based ferrofluid > engine oil-based ferrofluid > kerosene-based ferrofluid > water based ferrofluid.

Magnetic properties of perfluoropolyether oil-based ferrofluid before and after corrosion are shown in Fig. 27. Saturation magnetization is 308.15 Gs before corrosion and decreases to 246.134 Gs after 279 days of corrosion. Remanence and coercivity are approximately zero, which means that corrosion does not change superparamagnetism. The initial magnetization curves coincide, and the initial susceptibility is equal. Corrosion only reduces saturation magnetization, which is permitted in the applications.

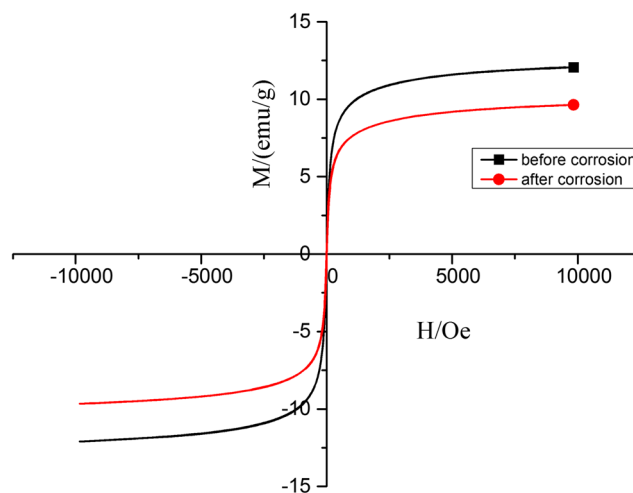


Fig. 27 Magnetization curves of perfluoropolyether oil-based ferrofluids before and after corrosion

4 Conclusions

Applications of conventional hydrocarbon-based ferrofluids are limited. Perfluoropolyether oil-based ferrofluid is stable in magnetic field; the saturation magnetization is 582.46 Gs. The average volatile rate is low, and the ferrofluid can work between -40 and 200 °C without oxidation or decomposition. Perfluoropolyether oil-based ferrofluid cannot be damaged by water or organic solvents. The ferrofluid also can survive in $\text{pH} = 1$ acid or $\text{pH} = 14$ alkali without corrosion. Perfluoropolyether oil-based ferrofluid has unique chemical stability, acid and alkali resistibility, high- and low-temperature bearing, and heat oxygen stability. It is a promising material to work in many potential applications.

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