

Synthesis and rheology of ferrofluids: a review

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There is no doubt about the potential technological significance of ferrofluids. The promising applications have been identified as dynamic sealing, heat dissipation, damping, and doping of technological materials. Ferrofluids are known as magnetic liquids that are colloidal suspensions of ultrafine, single domain magnetic particles in either aqueous or non-aqueous liquids. In this review article a general classification and the main properties of ferrofluids, description of their synthesis in terms of stability and rheology, and how it is understood in various parts of the science and technology are given. Then the structural changes and rheological properties of these smart fluids under an external stimulus together with a series of applications are presented.

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Introduction

Magnetic fluids are specific subset of smart materials that can adaptively change their physical properties due to external magnetic field. Magnetic control of the properties and behavior of liquids are promising fields for advanced applications and a challenge for basic research. Two main types of magnetic fluids are known since the middle of the 20th century; *magnetorheological (MR) fluids* and *ferrofluids*. The former are suspensions of micrometer-sized particles of magnetizable materials dispersed in a liquid carrier. On the other hand, *ideal* ferrofluids are colloidal suspensions of ultrafine (5–10 nm) single domain magnetic nano-particles such as iron oxide (γ -Fe₂O₃, Fe₃O₄), MnZn ferrites, and Fe and Co in either polar or non-polar liquid carriers [1,2]. Between ~15 and ~40 nm, the magnetic fluid is still considered as ferrofluids. Above this limit, the fluid goes into the limits of the MR fluids. Although conventional or real ferrofluids synthesized by various scientists are in the order of 5–15 nm, Lopez-Lopez and coworkers developed 2 new kinds of

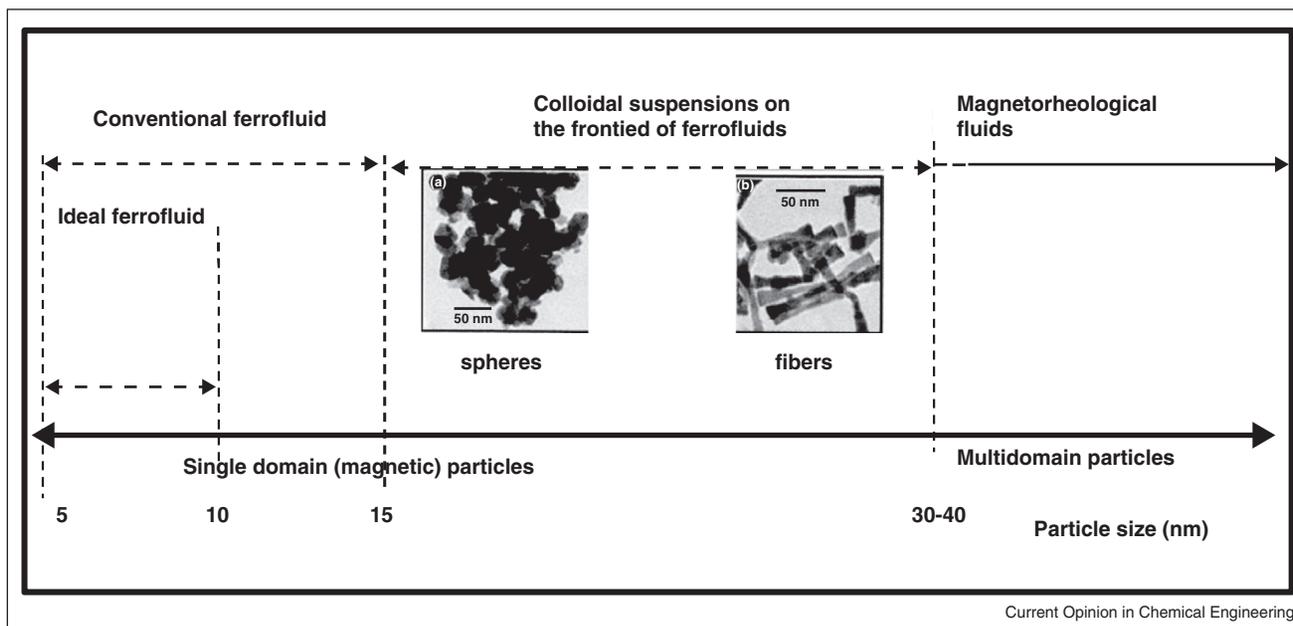
ferrofluids. The features that differentiate them from a conventional ferrofluid is that one of them is composed of spherical particles with larger particle size (diameter = 24 nm) and the other one is composed of fibers. [Figure 1](#) gives their classification of the particles. These fluids have proved to be an advantage from the point of view of the intensity of the MR effect. As a result they develop a larger yield stress and viscosities upon application. However, the stability of these new ferrofluids is worse than the conventional ferrofluids. The reason for this is the magnetostatic forces dominating over Brownian motion. Although they have worse stabilities, these ferrofluids have properties intermediate between conventional ferrofluids and MR fluids. These new fluids could be good candidates for applications with moderate MR effect and stability [3**].

The research and development on the preparation, characterization and application of the ferrofluids have been studied since mid-1960s which involve the multi-disciplinary sciences of chemistry, fluid mechanics and magnetism. Besides these science fields, because of the small particle size, ferrofluids have involved nanoscience and nanotechnology from their inception.

The most important advantage of these fluids is their ability to achieve a wide range of viscosity in a fraction of millisecond. The off-state viscosity of ferrofluids can go up to 2–500 mPa s depending on the concentration of the solid particles and the carrier liquid. Although they can respond to the action of external magnetic fields, stable ferrofluid show a relatively modest magnetorheological effect, such as increase in yield strength. Since the particle size of the magnetic phase is very small, under ordinary field strengths, thermal agitation gives rise to Brownian forces that can overcome the alignment of the dipoles. Therefore, ferrofluids exhibit field dependent viscosity but they exhibit no yield stress ($\tau_y = 0$) under magnetic fields. The origin of the viscosity enhancement is a single particle effect caused by the magnetic restoring torque experience by each suspending particle, $\mathbf{m} \times \mathbf{H}$, where \mathbf{m} is the magnetic dipole moment of the particle and \mathbf{H} is the magnetic field. This torque favors the alignment of the permanent dipoles along the direction of the applied field and thus opposes the rotation of particles in a shear flow, giving rise to an increase in the viscosity. Viscosity enhancements of $\Delta\eta(B)/\eta(0) \sim 2$ was obtained under applied magnetic fields [4] ([Table 1](#)).

Ferrofluids are quite useful not because of the small increase in their viscosity but their being magnetic fluids: they experience magnetic body forces in magnetic field

Figure 1



Magnetic fluid classification [3**].

Table 1

Some of the properties of ferrofluids [4].

Ferrofluid	
Particle material	Ceramics, ferrites, iron, cobalts, etc.
Particle size	5–10 nm
Suspending fluid	Oils, water
Density (gr/cm ³)	1–2
Off state viscosity (mPa s)	2–500
Required field	~1 kOe
Field induced fluids	$\Delta\eta(B)/\eta(0) \sim 2$
Device excitation	Permanent magnet

gradient, and so positions of a drop of ferrofluid can be controlled which enable them to be widely used in dynamic loudspeakers, computer hardware, dynamic sealing, electronic packaging, aerospace, and bioengineering [5].

The many possibilities of applications of ferrofluids will continue to be explored. In particular biomedical applications are gaining considerable attention. The use of ferrofluids for drug targeting or hyperthermia will certainly be the subject of intense research in the couple of years. This paper will give a brief review synthesis and rheology and latest development of the ferrofluids.

Synthesis of ferrofluids

The magnetic particle content, saturation magnetization, suspension viscosity and surfactants for stabilizing the ferrofluids are important factors that affect the rheological

properties, stability and redispersibility of the ferrofluids. According to the method of colloidal stabilization two main types of ferrofluids are synthesized: water-based ionic fluids and surfacted organic media based ferrofluids [1].

Magnetic phase

Although pure metals (Fe, Co, Ni) possess the highest saturation magnetization, they are extremely sensitive to oxidation, hence the magnetic particles such as ferrites like magnetite (Fe_3O_4), maghemite ($\gamma\text{-Fe}_2\text{O}_3$), or others (stoichiometric formula: $\text{MO}\cdot\text{Fe}_2\text{O}_3$, M divalent ion = Mn, Zn, Ni, Co, Fe) are commonly used in ferrofluids. Among these, nano size iron oxide is most widely used magnetic phase in ferrofluids. Various approaches have been explored for synthesis and characterization of high quality magnetic iron oxide nanoparticles. For example, sol-gel pyrolysis method was performed by Laokul *et al.* [6], Xu *et al.* [7], and Bica [8] synthesized nanoparticles by thermal reductive decomposition method. Mechanical alloying is another technique performed by Waje *et al.* [9]. Hydrothermal technique was also used by various scientists to synthesize ferrite nanoparticles [10,11]. However, the chemical method of coprecipitation of ferrous and ferric ions from solutions by addition of an alkali is a method which is very often used to prepare nanoparticles due to its low cost and simplicity [12]. Size reduction could be another method where magnetic powder of micron size is mixed with a solvent and a dispersant in a ball mill in order to grind for a period of several weeks [13].

Magnetic properties of the magnetic nanoparticles play the most crucial role in the rheology of the ferrofluids. As mentioned above, typical size of the magnetic nanoparticle is on the order of 10 nm which has magnetic single-domain and they possess magnetic moment μ . When subjected to an applied magnetic field, B , the magnetic moments have a potential energy U , given by Eqn. (1).

$$U = -\mu B \quad (1)$$

In order to reduce U the nanoparticles orient parallel to the applied magnetic field lines. When the applied field is removed, magnetic moments become randomized and magnetization becomes zero. If the applied magnetic field has a gradient then the nanoparticles will experience a force F and move toward the region of greatest field intensity. This force is proportional to the applied magnetic field, B , and the magnetic moments of nanoparticles, μ .

$$F = \nabla B \cdot \mu \quad (2)$$

Another important parameter that affects the magnetic properties of the ferrofluid is the Curie temperature, T_c , above which the magnetic particles lose their magnetization.

Carrier liquid

The function of the carrier liquid is to provide a medium in which the magnetic powders are suspended. A choice of carrier liquid depends on the type of the application. Ferrofluids used in different research and technology fields have been synthesized in carrier liquids such as water, silicone oil, synthetic or semi-synthetic oil, mineral oil, lubricating oil, kerosene and combinations of these and many other polar liquids [14]. Boiling temperature, vapor pressure at elevated temperature, freezing point are important parameters to consider when choosing the carrier liquid. The carrier liquid should be non-reactive with the magnetic phase and also with the material used in the device.

In recent years, studies on ferrofluids using ionic liquids have been reported which seems to be promising field of the study [15^{*},16^{*},17^{*}]. Ionic liquids are composed of ions and liquid and they have melting points near room temperature, or below 100 °C [18]. Huang and Wang prepared ionic liquid based ferrofluids containing coated and uncoated CoFe_2O_4 particles. They found that stability of the magnetic colloid was increased for the coated particles with the excess oleic acid. Oleate bilayer surrounding the particle surface contributed to steric stabilization [15^{*}]. Rodriguez-Arco and co-workers reported synthesis of magnetic fluids consisting of magnetite nanoparticles dispersed in an ionic liquid with addition of different additives. They used citric acid,

oleic acid, and humic acid as additives to provide the stability. Among these, oleate double layer gave rise to a true stable ferrofluid, whereas no or short term stability are achieved with the other additives. They showed that steric repulsion was needed to ensure long term stability and the best way to achieve this is to use surfactants adsorbed on the surface of the particles with tails compatible with the liquid carrier [16^{*},17^{*}]. Ionic fluids are considered to be stable, environmentally friendly and due to their low flammability, negligible vapor pressure, they are good candidate for colloidal media for nanoparticles.

Additives

In the synthesis of the ferrofluids, dispersants must be chosen to minimize the agglomeration of the particles and to increase colloidal stability which is important in the applications of the ferrofluids. The additives must also be chosen to match the dielectric properties of the carrier liquid. Various surfactants such as silica [19], chitosan [20], polyvinyl alcohol (PVA) [21], and ethylene glycol [22], are usually used to coat the nanoparticles and to enhance dispersibility in aqueous medium. Oleic acid (OA) is a commonly used surfactant to stabilize magnetic nano particles synthesized by traditional co-precipitation method [14]. Besides oleic acid, citric and tartaric acids are also used to obtain stable ferrofluids over a large pH range (pH 3–11) [23]. Anti-oxidation additives may also be added to prevent oxidation. In water based MR fluids pH control additives are also used. Surfactant from renewable material obtained from cashew nut shell liquid was synthesized by Baretto and co-workers [24]. This new colloidal suspension may be important in biological and industrial applications.

Ferrofluids are widely used in biomedical applications, such as hyperthermia [10]. Table 2 provides a list of materials that could be used to stabilize the nanoparticles [25,26].

Stability of the ferrofluids

Magnetic nanoparticles tend to aggregate due to strong magnetic dipole–dipole attraction between particles. Stability of the magnetic colloid depends on the thermal contribution and on the balance between attractive (van der Waals and dipole–dipole) and repulsive (steric and electrostatic) interactions. Table 3 gives the summary of the forces and the criteria for stability of the colloids.

Under the magnetic field, the magnetic energy derives the particles to higher intensity regions, on the other hand thermal energy forces particles to wander around in the whole liquid. The stability against segregation is favored by the high ratio of the thermal energy to the magnetic energy. Stability against settling due to gravitational field is given by the ratio between gravitational energy and magnetic energy. The equations and the ratios of these energies are given in Table 3.

Table 2
Different polymers and molecules that can be used for nanoparticle coating to stabilize the ferrofluid with some biological applications [25,26].

Polymers/molecules	Advantages
Polyethylene glycol (PEG)	Non-covalent immobilization of PEG on the surface improves the biocompatibility
Dextran	Stabilizes the colloidal solution
Polyvinylpyrrolidone (PVP)	Stabilizes the colloidal solution
Fatty acids	Colloidal stability, terminal functional carboxyl groups
Polyvinyl alcohol (PVA)	Prevents coagulation of particles, giving rise to monodisperse particles
	Increase the stability and biocompatibility of the particles and also helps in bioadhesion
Polyacrylic acid	Good for cell biology, for example, targeting to cell
Polypeptide	
Phosphorylcholine	Poorly complement and coagulation activation, colloidal solution stabilizer
Chitosan	A natural cationic linear polymer that is widely used as non-viral gene delivery system, biocompatible, hydrophilic, used in agriculture, food, medicine, biotechnology, textiles, polymers, and water treatment
Gelatin	Used as a gelling agent, emulsifier hydrophilic, biocompatible, natural polymer

The two basic attractive interactions between the magnetic particles are dipole–dipole and van der Waals–London interactions. The ratio of thermal energy to dipole–dipole contact energy must be greater than unity dipole dipole interaction. The diameter of the particle is given in Table 3. When all the parameters are plugged into the equation ($D \leq (72kT/\pi\mu_0M^2)^{1/3}$), the particle size is $D \leq 7.8$ nm. The normal ferrofluids with the particle size of 10 nm are in the limits of agglomeration. van der Waals forces arise due to the fluctuating electric dipole–dipole forces. Preventing of contact is another necessity if a stable colloid is to be obtained [27].

Besides the Brownian motion, electrostatic and steric repulsions are the main mechanisms supporting the ferrofluid colloidal stability. Electrostatic interaction is the dominant mechanisms in ionic ferrofluids whereas steric repulsion is the dominant mechanism supporting the colloidal stability in organic based ferrofluids [15]. Figure 2 gives the graphical representation of the long range magnetic interaction of particles and short range van der Waals attraction energy that are strongly related with the agglomeration and settling of particles in magnetic suspensions (Figure 2).

The agglomeration of powders suspended in a liquid can be prevented by creating mutually repelling charged double layers or by physically preventing the close

approach of particles by steric hindrance of the molecule adsorbed onto the particle surface (Figure 3) [15,27]. As the thickness of the adsorbed polymer increases, the size of the magnetic particle decreases and as a result the stability of the magnetic particle dispersion increases [15,27]. Huand and Wang showed that by excess oleic acid in their ferrofluid, stable magnetic colloid was achieved by steric repulsion [15,27]. The surfactants could be long chain molecules. If the particles are dispersed in a non-polar medium, as oil, one layer of surfactant is needed to form an external hydrophobic layer. The polar head of the surfactant is attached to the surface of the particles. On the other hand, if the particles are dispersed in apolar medium, as water, a double surfactant of the particle is needed to form a hydrophilic layer around them [28]. The functional group at the end could be cationic, anionic, or nonionic and is attached to the outer layer of the magnetic particles by either chemical bonding or physical or combination. Huang and Wang showed that stable ferrofluids cannot be achieved by uncoated magnetic particles [15,27]. Figure 3 gives the adsorption of the molecule and increase of the hydrodynamic radius.

The tail of the surfactant provides a permanent distance between the particles and compatibility with the fluid. One of the classic examples of an agent for dispersing magnetite particles into hydrocarbons is oleic acid.

Table 3
Forces acting on the magnetic nanoparticles and the criteria for stability of the colloids. [27].

Interaction force	Equation	Stability
Magnetic field energy	$E_{\text{mag}} = \mu_0 M_0 V H$	$kT/\mu_0 M V H \geq 1; D \leq (6kT/\pi\mu_0 M)^{1/3}$
Gravitational field	$E_{\text{grav}} = \Delta\rho V g h$	$\Delta\rho V g L/\mu_0 M H \ll 1$
Dipole-dipole contact energy	$E_{\text{dip}} = (\mu_0 M_0^2/12)V$	$12kT/\mu_0 M_0^2 V \geq 1; D \leq (72kT/\pi\mu_0 M^2)^{1/3}$
van der Waals	$E_{\text{vdW}} = -A/6[(2/(l^2 + 4l)) + (2/(l + 2)^2) + \ln((l^2 + 4l)/(l + 2)^2)]$	
Steric repulsion	Short ranged repulsive force	

μ_0 is the permeability of the free space ($4\pi \times 10^{-7}$ H m), M is the magnetization, H is the magnetic field, k is the Boltzmann's constant (1.38×10^{-23}) N m K⁻¹, $\Delta\rho$ ($=\rho_{\text{solid}} - \rho_{\text{liquid}}$), $g = 9.8$ m/s², A is the Hamaker constant (for Fe, Fe₂O₃ and Fe₃O₄ in hydrocarbon), $A = 10^{-19}$ Nm, l is the separation between particles and D is the diameter of the particle.

Figure 2

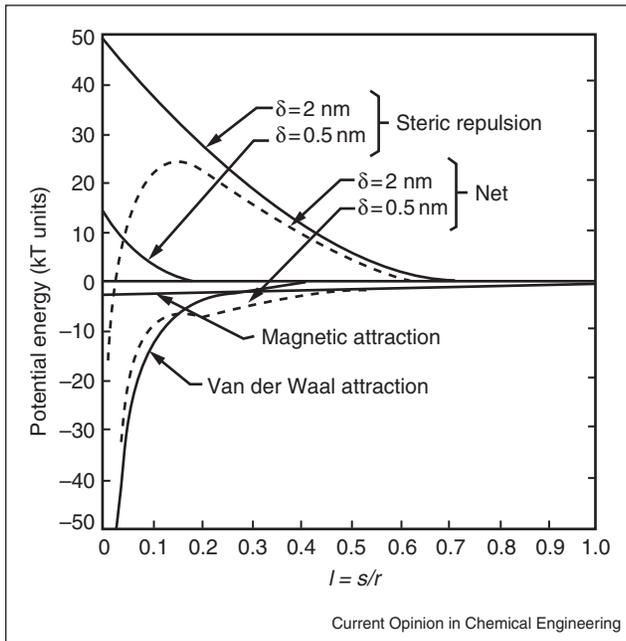


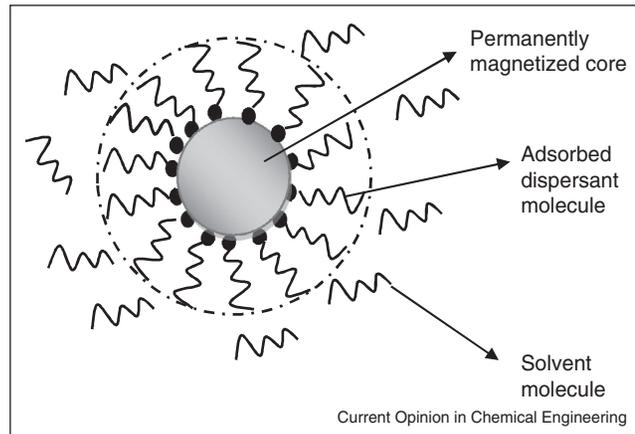
Illustration of magnetic attractive energy, van der Waals attractive energy and steric repulsion energy and the net potential energy for different lengths of adsorbed molecule, δ . The ratio between the interparticle distance and the radius of the particle is denoted as ' $l = s/r$ ' [27].

Although the particles are coated with polymeric binders or coated with adsorbed surfactants, the magnetic attractive force causes reagglomeration of the particles within time. This agglomeration process is more irreversible for the particles larger in size and magnetic moment.

Rheology of ferrofluids

The rheological properties of ferrofluids and their influencing factors are of importance in the fundamental research of ferrofluids and have significant impact on the applications. Viscosity of ferrofluids is one important issue of rheology. Many factors such as solid content, particle size distribution, pH value, temperature and surfactants influence the viscosity.

Figure 3



Adsorption of the long chain molecules onto the particle surface provides steric repulsion increasing the hydrodynamic radius [27].

Albert Einstein proposed a simple equation to calculate the viscosity of particle suspensions. The resulting formula (Eqn. (3)) relates the mixture viscosity η to the carrier fluid viscosity η_0 and solid volume fraction ϕ . The equation is only valid for dilute systems where $\phi < 0.5$.

$$\frac{\eta}{\eta_0} = 1 + 2.5\phi \tag{3}$$

After the proposal of Einstein, many models have been developed to predict viscosity. One of these models was proposed by Brinkman which gives a viscosity equation extended by Einstein's equation for concentrated suspensions. Batchelor expanded Eqn. (3) to include terms of order ϕ^2 where ϕ is the volume fraction. In this model, Brownian motion of the particles interaction with each other is taken into consideration and those interactions will affect the viscosity of the entire system. Bicerano *et al.* [30] examined the viscosity of suspensions of different hard bodies, and proposed a formula for the relative viscosity that provides a smooth transition between the dilute and the concentrated regimes and is valid for low-shear. The summary of these models are given in Table 4 [29].

Table 4

Comparison of the viscosity models [29].

Viscosity models	Expression	Description
Einstein	$\frac{\eta}{\eta_0} = 1 + 2.5\phi$	For dilute suspensions
Brinkman	$\frac{\eta}{\eta_0} = \frac{1}{(1-\phi)^{2.5}}$	Extended Einstein's equation for concentrated suspensions
Batchelor	$\frac{\eta}{\eta_0} = 1 + 2.5\phi + 6.5^2\phi^2$	Consider the effect of Brownian motion on bulk stress
Bicerano <i>et al.</i>	$\frac{\eta}{\eta_0} = 1 + \eta\phi + k_H\phi^2$	η is the virial viscosity, k_H is Huggins coefficient

η is the suspension viscosity and η_0 is the viscosity of the base fluid and ϕ is the volume fraction of the solid loading.

The change in the yield stress in magnetic fluids is given by the Bingham Model [30]. Eqn. (4) gives the Bingham model.

$$\tau = \tau_0 + \dot{\gamma}\eta \quad (4)$$

where τ is the shear stress, τ_0 is the yield stress, $\dot{\gamma}$ is the shear rate and η is the viscosity. When the shear stress is smaller than the yield stress, there is no fluid motion; otherwise, the shear rate is directly proportional to the difference of shear stress and yield stress.

The Carson model given by Eqn. (5) takes account of both the yield stress threshold and the shear thinning behavior and it is based on a structure model of the interactive behavior of solid and liquid phases of a two-phase suspension [30].

$$\sqrt{\tau} = \sqrt{\tau_y} + \sqrt{k\dot{\gamma}} \quad (5)$$

where k is a Carson model constant. The Carson model shows both yield stress and shear-thinning non-Newtonian viscosity. The Herschel–Bulkley (H–B) Model also takes the yield stress and shear thinning into consideration and it is given by Eqn. (6).

$$\tau = \tau_0 + m\dot{\gamma}^n \quad (6)$$

where m and n are regarded as model factors. There are 2 kinds of viscosity variations for ferrofluids when the shear rate increases: Newtonian and shear thinning behaviors. Shear thinning of well-dispersed suspensions can be linked to the modification in the structure and arrangement of interacting particles [31]. Shearing may cause the particles to orient in the direction of flow and its gradient which will cause agglomerates to break and hence reduce the amount of solvent immobilized by the particles which will lead to the lowering of the apparent viscosity. Hong *et al.* reported shear thinning behavior of water-based Fe_3O_4 ferrofluids. In their study, the viscosity of their water based ferrofluids with or without magnetic field were correlated using H–B model [32]. Shear thinning behavior was also reported for various types of ferrofluids [33,34].

A ferrofluid remains flowable in the presence of the magnetic field, even when it is fully magnetized. Yet, the rheology is affected by the presence of the magnetic field. Viscosity of the ferrofluids shows an increase with the increase in the magnetic field as well as the solid concentration [33,35]. The increase in the viscosity under magnetic field can be due to the formation of particle chains in the direction of the magnetic field. As the magnetic field increases the interaction between nanoparticles becomes stronger and the particle chains become longer. As the length of the chains increases, the resistance of the fluid to flow increases so the viscosity increases.

Eqns. (3) and (4) have analyzed the MR response of ferrofluids through the values of the field-induced yield

stress. However, in the case of ferrofluids it is more common to quantify their rheological response to applied magnetic fields through the magnetoviscous effect (MVE), which can be defined as [36]

$$M = \frac{\eta_H - \eta_0}{\eta_0} \quad (7)$$

where η_H and η_0 are the viscosities of the ferrofluid at a given magnetic field (H) and at zero magnetic field, respectively. This dimensionless magnitude quantifies the gain in viscosity that can be reached at a given shear rate upon application of a magnetic field of certain strength. Actually, for most technological applications (e.g. MR dampers and bearings) this magnitude has more interest than the yield stress, since the ferrofluid (or MR fluid) must work in the flow regime, where the yield stress is not relevant [3**].

Summary

Ferrofluids represent advanced, complex and multi-component materials synthesized using sophisticated pathways. Improvements in understanding of physics and chemistry and materials science of these materials will help to develop new ferrofluids and devices based on smart technology. Researches on the applications and synthesis of ferrofluids will continue to be explored in the coming years. Especially biomedical application has been progressing rapidly. Ferrofluids for drug targeting or hypothermia will certainly be subject of intense research in the next years.

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