

NANO-MICROSTRUCTURED MAGNETORHEOLOGICAL FLUIDS AND ENGINEERING APPLICATIONS

DANIELA SUSAN-RESIGA^{1,2}, PAUL BARVINSCHI¹

Abstract. Magnetorheological (MR) fluids are a category of magnetically controllable intelligent materials, interesting both for fluids science and engineering applications. Conventional MR fluids have several shortcomings and sometimes do not fulfill the requirements of various MR devices. This paper is reviewing some recently developed strategies for improving the composition and MR response of magnetorheological fluids. There are discussed results of varying the composition on nano- and micrometer size level on improving the kinetic stability of MRFs, on tuning their properties, respectively on the control of the (magneto) rheological response and behavior to develop high-performance engineering applications. The advantages of using nano-microstructured magnetoreological fluids compared to conventional MR fluids will be highlighted.

Key words: Ferrofluids, Magnetorheological fluids, Colloidal stability, Magnetorheological behavior, Leakage-free rotating seals, Seismic protection devices, MR brakes, MR clutches.

1. INTRODUCTION

Magnetic suspensions are multiphase systems (with at least two phases) of which at least one phase responds to an applied magnetic field, the material experiencing dramatic changes in their rheological, magnetic, electrical, thermal, acoustic and other mechanical and physical properties. In some cases, the magnetic field-responsive one is the particulate phase (e.g. magnetorheological fluids, ferrofluids), in other cases it is the carrier fluid that is sensitive to the field (e.g. inverse ferrofluids) [1], and sometimes both phases (magnetorheological fluids based on ferrofluids).

Among the magnetically controllable fluids we will focus mostly on the magnetorheological fluids and on ways to improve their magnetic field controllable properties.

Conventional magnetorheological fluids (MRF) are suspensions of multi-domain, non-colloidal, ferromagnetic (Fe, Fe and Co alloys) particles with different morphologies, in a carrier liquid that is not magnetic (mineral oils, silicon oils,

¹ West University of Timisoara, Faculty of Physics, Bvd. V. Parvan, no. 4, 300222, Timisoara, Romania

² Romanian Academy–Timisoara Branch, Bvd. M. Viteazu, no. 24, 300223, Timisoara, Romania

polyesters, watery solutions, gels, etc.) [2–4]. The volume fraction is usually between 0.1 and 0.5.

An applied magnetic field polarizes the multi-domain magnetic particles of the MRF, inducing their aggregation into large aggregates [5], resulting in effects that are important for practical applications: the magneto-viscous effect (MV effect) and the magnetorheological effect (MR effect). These effects are seen in a relative increase of viscosity and yield stress by up to 3 orders of magnitude in moderate magnetic fields. The magnetic moment of the particles is induced by the applied field, therefore it will disappear if the magnetic field is removed, and the suspension will regain its initial state, meaning that the processes of effective change of viscosity and yield stress are reversible. Also, the response to the application of the field is very fast.

Therefore, the fundamental phenomena in conventional MRF (CMRF) magnetorheology are connected to *the possibility of influencing/controlling their structure, and, as a consequence their rheological behavior by using moderate magnetic fields.*

By doing rheological tests and interpreting their results we can build a bridge that connects microscopic behaviours with measurable physical data characteristic for MRFs. For this reason, analyzing the flow behavior of conventional MRF's and constructing rheological models have kept many researchers busy [6–9, 3, 10].

The capacity to control / adapt the properties of CMRFs motivates their use in applications for active vibration control or torque transmission, encountered in various engineering fields [11–20]:

- civil: semi-active dampers for building and bridge foundations, to absorb vibrations generated by earthquakes and the movement of vehicles;
- automotive: clutches, brakes and MR dampers used to reduce vibrations transmitted to passengers, increasing comfort;
- household applications: washing machine dampers;
- sports applications: gym equipment dampers;
- military equipment;
- robotics, etc.

MRFs are also encountered in other areas:

- chemical sensors [21];
- bio-med applications (e.g. artificial joints with built-in dampers to reduce the shocks and vibrations generated by the wearer's movement) [22, 23];
- polishing techniques [24–26];
- sound propagation [27];
- thermal energy transfer control [28, 29];
- electro-optic devices, such as magnetic field sensors, light valves and optic bi-stability devices [30, 31];
- MR orifices and valves in hydraulic circuits;
- rotating seals etc.

The advantages of MRF devices over conventional devices include fast (several milliseconds) response times, better performance, easy design, and in some cases reduced cost.

Despite large progress in this field, conventional MRF's do not yet fulfill all requirements of commercial applications [1, 32]:

- the maximum MR effect is limited by the saturation magnetization of the magnetic phase;
- the speed of the response to the application of the magnetic field is restricted by the characteristics of the coils, and response time is a crucial factor for semi-active control;
- the magnetic phase frequently shows a slight remanence (stays slightly magnetized after the magnetic field is removed);
- MRFs are not stable enough: particle density is significantly larger than the carrier liquid density, and in addition their size is larger than that of particles of ferrofluids, resulting in sedimentation in the long term in the absence of constant stirring [33]; decreasing particle size improves stability, but decreases MV and MR effects;
- once a dense, rigid layer of settled particles has formed, it is difficult to re-disperse them due to the remanent magnetic attraction between the particles;
- thickening during long uses under strong magnetic fields. When MRFs are subjected to large numbers of use cycles, their viscosity in the absence of a magnetic field under high shear stress and shear speed can increase, transforming the suspension into a paste that makes the MR damper improper for use in semi-active control;
- high abrasion due to large particle sizes is a disadvantage in some applications. Erosion due to friction can lead to a structural modification of the suspension's particles which can lead to thickening the MRF, impacting the MR performance [34]; in addition, the walls of the container in which the MRF operates can also be eroded;
- separation of particles from the fluid during use [35]. It has been observed that during "squeeze" mode use, the particles or the carrier liquid can separate perpendicularly to the direction of the compression force, especially when the carrier liquid has a low viscosity, compression speed is low and the applied magnetic field is intense;
- oxidation of particles, which can lead to low shear stresses and large response times [36, 37], and possibly to thickening the MRF during use [38], impacting the performance of the MRF application;
- dependence of MRF viscosity on temperature. Depending on the application, the operating temperature interval can be between -20 °C and 150 °C; outside this interval the viscosity of the MRF is no longer controllable, and semi-active control is compromised [37];
- in MRF's used for sealing problems can arise due to the carrier liquid's (usually oil) viscosity decreasing as temperature increases [37].

The objective of this paper is to discuss some strategies and researchers efforts focused on improving the kinetic stability of MRFs, on tuning their properties, respectively on the control of the (magneto)rheological response and behavior to develop high-performance engineering applications. The advantages of using nano-microstructured magnetoreological fluids compared to conventional MRFs will be highlighted.

2. NANO-MICROSTRUCTURED MAGNETORHEOLOGICAL FLUIDS

2.1. STRATEGIES FOR IMPROVING MAGNETORHEOLOGICAL FLUIDS

The fact that MRFs are key components in many advanced technology applications involving active control of vibrations or torque transmission motivates increasing interest in improving performance through new MRFs formulas:

2.1.1. INVERSE FERROFLUIDS

Among new MRFs formulations, inverse ferrofluids (IFF) have been attracting researchers since the 80's. IFFs are non-magnetic, electro-isolating or electro-conductive microparticle suspensions in a magnetizable carrier liquid [39–45].

The non-magnetic particles being larger by one or two orders of magnitude compared to the ferrofluid's magnetic particles (the carrier fluid), and the ferrofluid can be considered as being hydrodynamically and magnetically continuous. Applying a magnetic field induces a visible magnetic dipole interaction, either attractive or repulsive, between the non-magnetic microparticles. The intensity of this interaction can be controlled by adapting the intensity of the magnetic field, the concentration and morphology of the non-magnetic microparticles, the concentration and magnetization of the carrier ferrofluid's nanoparticles. The microparticle chains that are forming under the action of an external magnetic field are oriented by the direction of the field lines [43].

Skjeltorp has investigated IFFs based on 1–10 μm polystyren spheres suspended in a ferrofluid [39–41], inserted between two glass plates with a gap close to the spheres diameter. When applying a magnetic field parallel to the IFF layer, the particle aggregates took the form of chains oriented by the direction of the magnetic field, and when the field was applied in a perpendicular direction to the IFF layer a hexagonal network of polystyren layers has formed.

The effect of the magnetic field intensity on the rheological behavior of IFFs with silicon spherical particles of $\sim 104\text{--}378$ nm diameters dispersed in Newtonian ferrofluids with magnetite nanoparticles in a non-aqueous carrier liquid (viscosities between 44 and 559 mPas), volume fractions for the Si particles being in the interval (12.6–26.1)% was analyzed in [46]. Viscosity curves measured with a MCR 501 Anton Paar rheometer for different values of magnetic field intensity

define three regions with different behavior in the presence of the magnetic field (in the absence of the field, the behavior has been Newtonian) – Fig. 1:

- under low shear the effect of the magnetic field is more pronounced, with viscosity increasing with the increase of the field due to the structuring induced by it;
- under medium shear rates we observe shear-thinning behaviour, due to the fragmentation of the field induced particle aggregates;
- at high shear rates viscosity tends towards a constant value that is dependent on the particle volume fraction and the carrier liquid's viscosity (the ferrofluid).

The lines in Fig. 1 are fitted using Sisko's formula:

$$\eta = \eta_{\infty} + c\dot{\gamma}^{-n}, \quad (1)$$

where η_{∞} is the viscosity at infinite (high) shear rate.

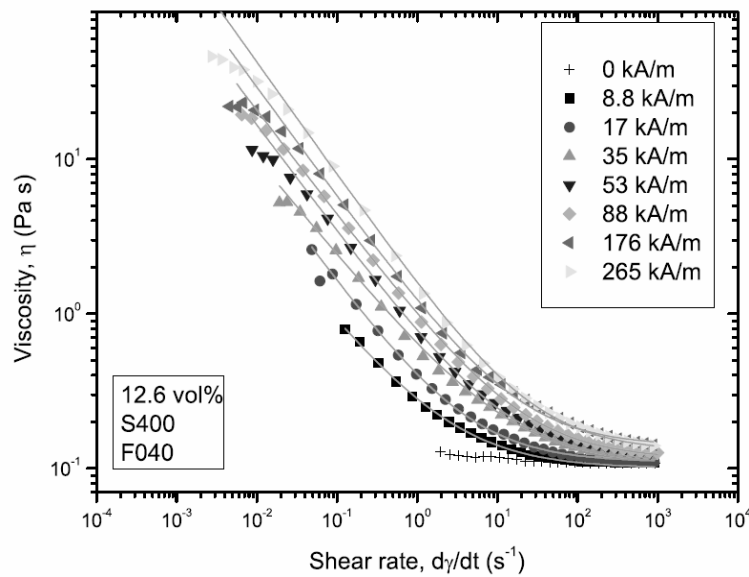


Fig. 1 – Viscosity curves for IFF's based on Si particles with a 378 nm diameter (12.6% volume fraction) dispersed in a ferrofluid with viscosity 44 mPas and saturation magnetization 25.5 kA/m [46]. Reprinted with permission from Ramos J., Klingenberg D.J., Hidalgo-Alvarez R., and de Vicente (2011), *Steady shear magnetorheology of inverse ferrofluids*, *Journal of Rheology* **55**(1), 127–152; doi: 10.1122/1.3523481 (Copyright 2011, The Society of Rheology).

Ramos and collaborators showed that by representing dimensionless viscosity as a function of Mason's number, data measured for different intensities of the magnetic field overlap on a (unique) master curve – Fig. 2.

For the steady shear flow, the Mason number represents the ratio between hydrodynamic and magnetostatic forces in the MRF, and it is expressed as:

$$M_n = \frac{8\eta_C \dot{\gamma}}{\mu_0 \mu_{rC} \beta^2 H^2}, \quad (2)$$

with η_C viscosity of the continuous media, $\dot{\gamma}$ shear rate, μ_0 magnetic permeability of the vacuum, μ_{rC} relative magnetic permeability of the carrier liquid, $\beta = \frac{\mu_p - \mu_{rC}}{\mu_p + 2\mu_{rC}}$ magnetic contrast factor, μ_p relative magnetic permeability of the particles, H intensity of the magnetic field in the suspension.

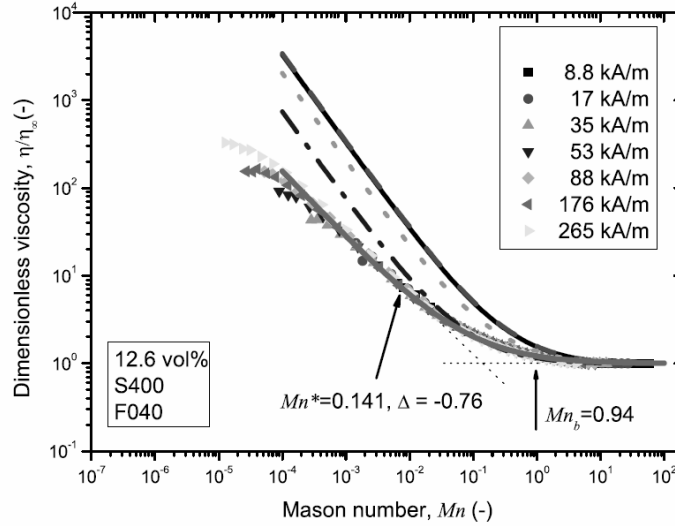


Fig. 2 – Viscosity curves as a function of Mason number for IFFs presented in Fig. 2 [46]. Reprinted with permission from Ramos J., Klingenberg D.J., Hidalgo-Alvarez R., and de Vicente (2011), *Steady shear magnetorheology of inverse ferrofluids*, Journal of Rheology **55**(1), 127–152; doi: 10.1122/1.3523481 (Copyright 2011, The Society of Rheology).

The lines in Fig. 2 represent fits with several models, developed by Martin and Anderson [47], by Vicente and collab. [48], by Gans and collab. [49], Volkova and collab. [50].

The best fit (the pink line) was obtained using the formula proposed in [46]:

$$\frac{\eta}{\eta_\infty} = 1 + M_n^* M_n^\Delta, \quad (3)$$

where Δ is an exponent and M_n^* is the critical Mason number, meaning that value of Mason number corresponding to the transition from magnetic control to hydrodynamic control of the suspension's structure. The values of the fit parameters are presented in Fig. 2, and M_{nb} is the Mason number associated with the breaking of the last doublet.

Ramos and collab. have also analyzed in the same paper the effect of the volume fraction of the Si particles, the effect of the carrier ferrofluid's viscosity and the effect of the particle's size on the rheological behavior and have shown that the viscosity data measured while varying these characteristics of the IFF can also be overlapped on a master curve if the adimensional viscosity curves are represented as a function of the Mason number over the critical Mason number

("dimensionless" Mason number):
$$\frac{\eta}{\eta_\infty} = f\left(\frac{M_n}{M_n^*}\right).$$

Although researchers have found that the MV and MR effects in IFFs are smaller than those in conventional MRFs, the availability of non-magnetic particles that have a very well-defined shape, density and size motivates the IFFs to be frequently utilized as a study model for MR and MV effects.

2.1.2. ADDING ADDITIVES AND SURFACTANTS, COATING PARTICLES WITH AN ORGANIC (USUALLY A POLYMER) AND/OR INORGANIC LAYER

In order to improve the sedimentation behavior of MRFs Weiss and collab. have suggested to add thixotropic additives with hydrogen bonds, polymer-modified metallic oxides, or mixes of these two [51]. The role of the additives is to prevent physical contact between the suspended magnetic particles, contact that can lead to the formation of a dense layer as a consequence of sedimentation, which is difficult to redisperse.

G. van Ewijk has presented the methods to obtain two other types of magnetofluidic composites [52]:

a) The first type is based on magnetite nanoparticles (~ 10 nm) with the dipolar magnetic interaction screened by a SiO_2 layer. In the presence of an external magnetic field, the magnetic moment of the magnetite particles aligns with the direction of the field lines, and forms chains of spheres in that direction. Such MRFs with nano-composite particles with a Fe_3O_4 core and SiO_2 shell (core-shell particles) dispersed in silicon oil were also investigated by Chae's group [53], and they showed the increase in the suspension's stability by reducing the density difference between the particles and the carrier liquid.

b) The second type is based on magnetizable spheres of ~ 1 μm with a SiO_2 core and magnetite nanosphere shell. Because the individual magnetic momentum of the magnetite nanoparticles is randomly oriented in the absence of a magnetic field, the SiO_2 sphere coated with a magnetite nanoparticle layer will not have any magnetic moment. However, under the action of a magnetic field, the magnetic

moment of the nanoparticles will align, giving the composite microparticles a large magnetic moment, aligned with the applied magnetic field. Such MRFs with a water carrier liquid, with nano-composite particles having a SiO_2 core and Fe_3O_4 shell, were prepared and analyzed by Pei and collab. [54], showing once again an increase in kinetic stability due to decreased particle density.

Therefore, MRFs with strongly magnetizable core-shell structured particles [55–57] were proven to be more stable and with more durable properties than conventional MRFs, but with the cost of a lower yield stress.

Analyzing the effect of adding three additives (oleic acid, Al stearate and Si nanoparticles) on sedimentation and redispersability of particles in MRFs, López-López and collab. have shown that oleic acid and Al stearate have similar effects: even if they do not impede sedimentation, the irreversible agglomeration of particles is severely reduced and their redispersability is significantly easier [58]. Adding gel-forming agents such as Si nanoparticles prevents deposition of Fe particles when MRFs are at rest, but in shear conditions the gel is destroyed, and the Fe particles can still sediment. The Si-Fe sediments that form prevent particle redispersing. It has also been observed that applying a magnetic field reduces sedimentation rate and eases redispersion.

Improving MRFs stability with Fe particles in silicon oil by adding a thixotropic additive (Si nanoparticles) was investigated with rotational and oscillatory tests by de Vicente's group [59]. Also, in order to reduce sedimentation, Lim and collab. have suggested an MRF containing a mix of spherical Fe carbonil particles and Si coated Fe carbonil particles [60], which has improved the dampening performance of a small size MR damper. Due to the increased affinity between the coated particles and the silicon oil the response time was also improved.

Olabi and Grunwald have used sodium and lithium stearate as thixotropic additives in order to improve stability [61], while Ashtiani and collab. have added stearic acid to the carrier liquid as an additive, which led to the formation of a gelatinous network which helped prevent sedimentation [62].

López-López and collab. used different surfactants (oleic acid, Al stearate, lecithin, Na dodecyl-sulphate, etc.) as stabilizers in MRFs with spherical Fe microparticles in polar and non-polar carrier liquids to obtain a good dispersion of the particles [63].

Even for water based MRFs stability can be improved using surfactants, e.g. glycerol/sodium stearate [64], polyacrylic acid [65] or wormlike surfactant mycelium (above a critical concentration the structure of surfactant molecules shifts from spherical to a long cylindrical one, known as wormlike) [66].

2.1.3. USING HIGH VISCOSITY AND VISCO-ELASTIC CARRIER LIQUIDS; USING FATTY BASE LIQUIDS; PREPARATIONS OF MAGNETIC GELS AND ELASTOMERS

Segovia-Gutiérrez has investigated an MRF based on a Boger fluid [67].

Boger fluids are a mix of a polymer solution diluted with a solvent of high viscosity, and they show visco-elasticity (the viscoelastic moduli G' , G'' are

different from zero), but has a constant viscosity, independent of shearing rate. The justification for using a Boger fluid as a carrier liquid for an MRF is the high viscosity (that can reduce the particle sedimentation phenomenon) and the intrinsic visco-elastic properties.

The MRF studied by Segovia-Gutiérrez contained Fe carbonil particles (5% vol) in a Boger fluid (prepared by dispersing polyacrylamide in double-distilled water, mixed with high-viscosity glucose syrup). The rheological behavior was non-Newtonian in the absence of a magnetic field (viscosity was ~ 103 Pas), and shear-thinning in the presence of a field. The MV effect was ~ 4 orders of magnitude for a magnetic field of 350 kA/m.

Choi and collab. also showed that using a highly viscous carrier liquid with pronounced shear-thinning behavior insure long-term stability of MRFs [68].

Also, using fatty (lubricated) carrier liquids with a well defined yield stress improved sedimentation behavior without significantly affecting the intensity of the MR effect [69].

Improvements can also be obtained by using magnetic compounds that are the solid equivalent of ferro-fluids and MRFs, namely magnetic gels [70] and magnetic elastomers [71]. Magnetic elastomers are produced by dispersing magnetic particles in a polymer solution or melt and applying a magnetic field before crosslinking. Responsive composite materials at magnetic fields, obtained by dispersing superparamagnetic particles in polymers were used to study colloidal aggregation [72] and optic traps [73] as well as other magneto-electro-rheological applications.

Electrically conductive composites can be obtained by introducing graphite microparticles in an elastomer matrix, which can be used in the fabrication of magneto-resistors, sensors and magnetic field transducers [74,75].

2.1.4. THE EFFECT OF PARTICLE SIZE AND CONCENTRATION; PREPARING BIDISPERSE MRFs

Many researchers have investigated the effect of particle size and concentration of conventional MRFs [31, 76, 77]. Reducing particle sizes to a few hundred nanometers [78, 79] in order to diminish the effect of sedimentation and abrasion had repercussions on the intensity of the MR effect.

Rosenfeld and collab. [77] have comparatively investigated three MR fluids with the same solid mass concentration (60% mass): one based on nanometric Fe powder (~ 15 - 25 nm), another based on micrometric powder (~ 45 μm) and a third being a nano-micro mixture (50/50%), with the Fe particles of spherical shape. In the absence of a magnetic field, the nano and hybrid fluids showed a Bingham yield stress higher than the fluid with iron microparticles. The situation changes in the presence of a magnetic field, when the highest yield stress is present in the fluid with micrometric iron particles, which is, however, not the most stable of the three. The plastic viscosity has a similar behavior.

The distribution of magnetic particle sizes has proved to have a great influence on kinetic stability of MRFs [2, 76, 80].

Wereley and collab. have prepared a bidisperse MRF by dispersing nanometric and micrometric iron particles in hydraulic oil [76]. The main results are summarized below:

a) MRF with Fe microparticles, under the action of a magnetic field, develop a relatively coarse network of chains or sheets, revealing a significant yield stress; b) the nanoparticle fluid develops a fine network, yet strong, suggesting a low yield stress; c) in the bidisperse fluid distinct chains of microparticles are formed, while the nanoparticles seem to fill the gaps between the microparticles. This indicates the possibility of preparing such MRFs with high yield stress, low sedimentation speed and improved redispersability (compared to a fluid which contains only microparticles) due to the thermal convection of the nanoparticles.

Wereley and collab. demonstrated the advantages of bidisperse fluids using rheological, magneto-rheological and sedimentation investigations: replacing 20% of a MRF conventional Fe particles (of micron size) with nanoparticles, the sedimentation speed decreases by approximately one order of magnitude, and the yield stress under intense magnetic fields can increase by over 15%.

The advantages of using extreme bidisperse fluids were also proven in the case of water based suspensions with nano- and micro-metric magnetite particles by Viota and collab. [81]. For a given microparticle concentration, the increase in nanoparticles concentration leads to an increase of the yield stress for all investigated values of the applied magnetic field's intensity. This increase is attributed to the formation of heterogeneous aggregates that improve the stability of the suspension and favor the formation of well-arranged structures induced by the field.

Improving the MR effect, stability and redispersability were also demonstrated in the case of using bimodal suspensions at the single-multidomain limit [82]. Particles of both sizes were made of Fe, and the carrier liquid was polyalphaolefin oil. The experimental results show a satisfactory correlation to those obtained from 3D finite element simulations. The physical reason for this improvement is the coating of the larger particles with small particles, due to remnant magnetization of the latter.

2.1.5. THE EFFECT OF PARTICLE MORPHOLOGY

Beside spherical carbonyl iron particles, magnetizable particles of other shapes have been synthesized in order to produce MRFs with high yield stress and low sedimentation rate [83].

The paper [84] presents and characterizes a new type of MRF with low sedimentation, that contains plate shaped Fe microparticles, which play an important role in improving yield stress, flow behavior and sedimentation stability. The properties of this MRF were validated by testing a small scale damper that can be used to control washing machine vibrations.

The magnetoviscous and viscoelastic properties of some bidisperse MR suspensions with plate shaped Fe particles of different size ($2\ \mu\text{m}$, respectively $19\ \mu\text{m}$) and mass fractions ($W_{\text{small}}/W_{\text{large}}$ is 0.67, 1.5, respectively 4.0) in a non-magnetic carrier liquid (a hydrocarbon) were investigated in [85]. It was observed that the initial susceptibility and saturation magnetization are higher in the case of larger particles. Increasing the particle concentration for small particles improves the formation of strong chains. If the fluid contains particles of two different sizes, microscopic studies revealed the formation of longer chains due to the presence of the smaller particles, therefore an improvement of interactions between particles and as a consequence a higher yield stresses and higher elastic moduli. These experimental results are important for design of MRF based devices, for estimating the dependency of the damping force on the applied magnetic field in the case of a damper used in a vehicle, or torque in the case of a braking system using an MRF.

De Vicente and collab. have investigated the rheological and magnetorheological behavior in oscillatory tests with low amplitude for spherical magnetite particle MRFs, and for rod-shaped particle MRFs (Fig. 3), with the same intrinsic magnetic and crystallographic properties and similar size distributions, for magnetic fields with $H = 0\text{--}800\ \text{kA/m}$ [86].

MRFs with rod shaped particles show better MR properties in oscillatory shear conditions in the linear viscoelastic domain (LVE): they need a less intense magnetic field in order to form structures, and develop a higher elastic modulus.

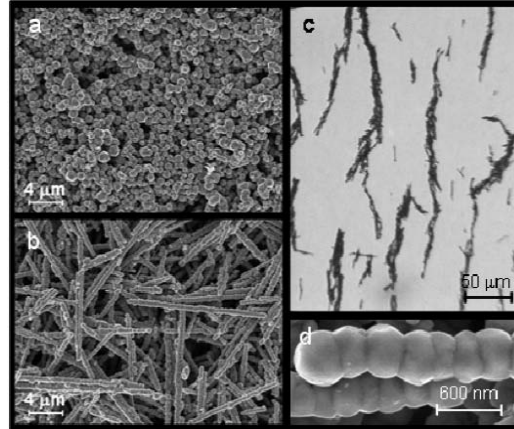


Fig. 3 – SEM images of: a) spheres ($680 \pm 150\ \text{nm}$); b) magnetite rods (sizes $560 \pm 120\ \text{nm}/6900 \pm 3800\ \text{nm}$) dispersed in MRFs; c) typical structure induced in MRFs containing microrods by magnetic fields; d) magnetite microrods detail [86]. Reprinted from de Vicente J., Segovia-Gutiérrez J.P., Andablo-Reyes E., Vereda F. (2009), *Dynamic rheology of sphere- and rod-based magnetorheological fluids*, The Journal of Chemical Physics **131**(19), 194902; doi: 10.1063/1.3259358, with the permission of AIP Publishing.

In [87], the effect of the particles' shape on the MR performance of the MRF under the influence of a magnetic field was investigated for spherical, plate shaped and rod shaped particles, giving details also about the procedure of obtaining the particles of different morphologies. MRFs with rod-type particles showed higher yield stress and elastic modulus.

However, it has been observed that the effect of the particles' shape is negligible for high concentrations of particles and/or high intensity magnetic fields. The effect of the particle shapes and field intensity over the yield effect manifested by the three MRF types is shown in Fig. 4.

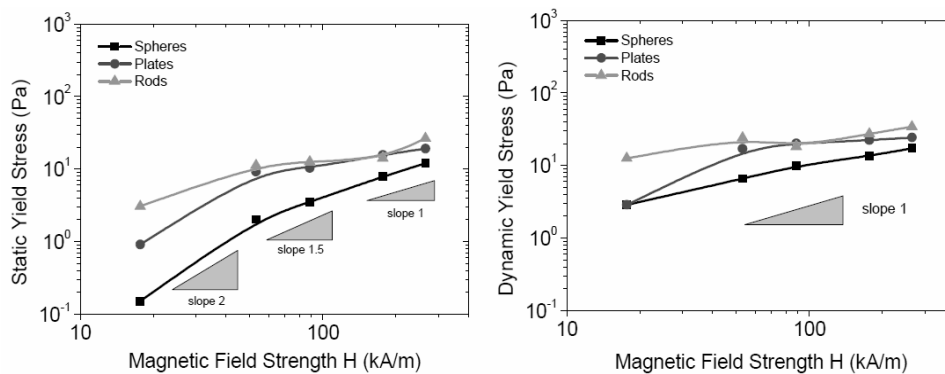


Fig. 4 – The effect of particle shape on yield stress (static and dynamic Bingham type) for different values of magnetic field intensity. Particle volume fraction is 1% [87]. Reprinted with permission from de Vicente J., Fernando V., Segovia-Gutiérrez J.P., del Puerto Morales M., and Hidalgo-Alvarez R. (2010), *Effect of particle shape in magnetorheology*, *Journal of Rheology* **54**, 1337–1362; doi: 10.1122/1.3479045. (Copyright 2010, The Society of Rheology).

It seems that the non-spherical particles tend to align with the long axis along the field lines, and when the field intensity increases they experience a stronger internal field, the magnetization process being faster and the structuring stronger for field intensities below saturation intensity.

The shear viscosity of these three suspensions measured for different magnetic field intensities was represented as a function of the Mason number – Figs. 5 a, b, c. The following approximations were used:

- since the volume fraction of the particles was very low (1%), the magnetic field inside the suspension was considered to be the applied magnetic field;
- the structures induced by a moderate magnetic field in the low concentration MRFs were considered to be chains with a thickness equal to particle size.

Figures 5 a, b, c show a good overlapping of data on a single master curve, for a large range of values of magnetic field intensities (in the linear magnetic regime), especially in the case of suspensions with spherical and plate-shaped particles.

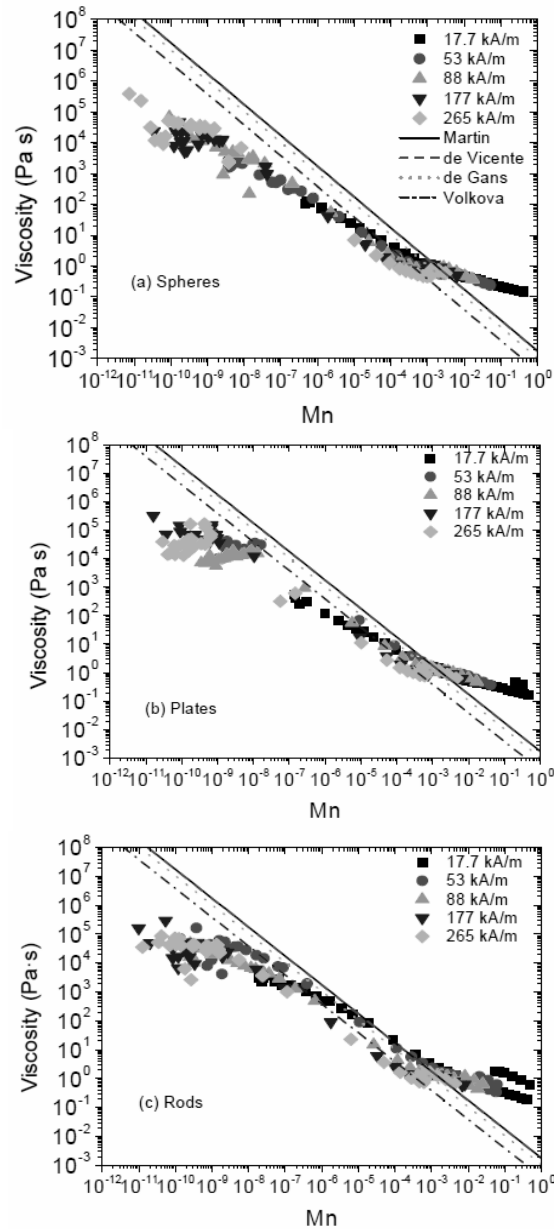


Fig. 5 – Shear viscosity as a function of Mason number for a large range of values of magnetic field intensities and three different particle shapes: a) spheres; b) plates; c) rods [87]. Reprinted with permission from de Vicente J., Fernando V., Segovia-Gutiérrez J.P., del Puerto Morales M., and Hidalgo-Alvarez R. (2010), *Effect of particle shape in magnetorheology*, Journal of Rheology **54**, 1337–1362; doi: 10.1122/1.3479045. (Copyright 2010, The Society of Rheology).

A comparative study of the magnetorheology of conventional MRFs with Fe particles and MRFs with porous and rough Fe particles was done by Vereda and collab. [88]. The MR suspensions had the same mass concentration of Fe, the particles had similar sizes and magnetization curves, but the rheological behavior was different due to the particle porosity in one of the fluids. The voids in these particles lead to a reduced density and a smaller average volume magnetization. Also, for the same mass content, the porous particles occupy a larger volume fraction. The volume fraction for the same mass concentration was double for the MRFs with porous particles compared to the MRFs that had smooth particles, and the average volume magnetization for each particle was roughly half of that of a smooth particle. Thus, for the same Fe mass content viscosities and yield stresses (static and dynamic) were measured to be smaller for MRFs with porous particles.

The rheological properties of the two suspensions can be acceptably correlated by applying the adequate correction – by correctly estimating the volume fraction of the particles and effective volumetric magnetization. In the case of the suspension containing the porous particles with a volume fraction of 2.1% an atypical dilatant behavior was detected, which can be caused by the roughness of the surface of the porous particles. Using the finite element method, it has been shown that the particle's surface roughness can affect the field induced particle magnetization, and the interactions between the particles [89, 90].

Using a rheometer with a parallel plate MR cell, Bell and collab. have investigated through rotational tests, the MR properties of some suspensions containing 260 nm diameter Fe micro-wires with different length distributions ($5.4 \pm 5.2 \mu\text{m}$, respectively $7.6 \pm 5.1 \mu\text{m}$) dispersed in silicone oil, with volume fractions of 2, 4 and 6% [91]. The yield stress in the presence of a saturation magnetic field was maximum (8.2 kPa) for MRFs containing $7.6 \mu\text{m}$ wires with a 6% volume fraction, and the degree of sedimentation was significantly lower than in the case of conventional MRFs. It has been observed that rod-shaped wires improve magnetorheological performance.

López-López, Kuzhir and Bossis studied in detail, experimentally [92] and theoretically [93] the shearing magneto-rheology of some suspensions of non-Brownian magnetic micro-fibers with different concentrations in a wide range of magnetic field intensity values. The results were compared with those obtained for conventional MRFs. They found that magnetic fiber suspensions show an improved MR effect, explained by the presence of friction between the fibers, dependent on the applied magnetic field. In order of establishing the relevance of this friction, they investigated the microscopic structure of these suspensions using an optical microscope. In the absence of an applied magnetic field, the fibers form an entangled network with relatively isotropic orientation. When a magnetic field is applied, the fiber network deforms and tends to align with the direction of the field. The solid friction of the fibers prevents a full alignment of the fibers with the field,

and the network remains entangled. Theoretically, they considered four different structures of the magnetic fiber suspensions: column, zigzag, three dimensional stochastic structures and almost planar stochastic structures and found that practically the main contribution to the yield stress comes from the solid friction between the fibers, and not from the dipolar magnetic interactions between the fibers. The lowest yield stress was obtained in the zigzag structure, and the highest for the column structure.

Experimental results and analytic calculations based on the finite element method reported by Vereda and collab. showed that the MR effect of an MRF can also be improved by using faceted particles [94]. The improvement can be partially attributed to more intense magnetostatic forces between the faceted particles. Magnetite particles with an average size of $(4.4 \pm 1.4) \mu\text{m}$, synthesized according to the procedure described in [95], showed faceted morphologies, the octahedron being the dominant shape – Fig. 6.

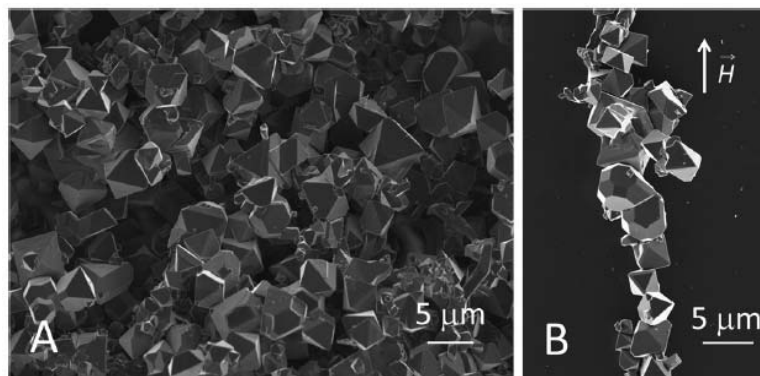


Fig. 6 – A) SEM micrograph of magnetite particles used in MRF preparation; B) SEM micrograph on a linear aggregate formed when these particles were dried in the presence of a DC uniaxial magnetic field [94]. Reprinted from Vereda F., Segovia-Gutiérrez J.P., de Vicente J., and Hidalgo-Alvarez R. (2016), *Faceted particles: An approach for the enhancement of the elasticity and the yieldstress of magnetorheological fluids*, *Applied Physics Letters* **108**(21), 211904; doi: 10.1063/1.4952394, with the permission of AIP Publishing.

Finite element method calculations for simple systems with two particles have shown that improvements to the strength of the MRFs structure can be partially attributed to the stronger magnetic forces between faceted particles due to the planar contacts, which are closer than point contacts between spherical particles.

Lee and collab. compared the MR and sedimentation properties as a function of particle shape for MRFs containing spherical Fe carbonyl particles, respectively flake-shaped particles [96]. Although the saturation magnetization was smaller in the case of the suspension containing the flake-shaped particles, the rheological

properties (shear stress, viscosity, elastic modulus) under the effect of an external magnetic field were superior to those of the spherical particle suspension. In addition, the kinetic stability was better in the case of the Fe carbonyl flake suspension, suggesting the importance of the particle's anisotropy towards the MR performance of a suspension.

For such suspensions containing flake-shaped Fe particles, Upadhyay and collab. demonstrated an additional 20% improvement in thermal conductivity (under the action of a magnetic field) compared to a commercial MRF containing spherical particles with a volume fraction larger by almost 20% [97]. This improvement was attributed to the increase in contact area for the flake-shaped particles when they form chains, which insures a faster heat transfer. The improvement is even higher if magnetic nanoparticles are added to such a suspension, which reduce the friction between the flake-shaped particles, acting as a lubricant.

Improved sedimentation stability was also obtained in the case of dimorphic MRFs, obtained by partially replacing the suspension's microparticles with particles of other shapes. Ngatu and collab. have prepared such MRFs by substituting a part of the spherical Fe particles (diameters of $8 \pm 2 \mu\text{m}$ and volume fraction between 50 and 80%) with Fe nano-wires with a diameter of 230 nm and a length distribution of $7.6 \pm 5.1 \mu\text{m}$ [98]. Sedlacik and collaborator's dimorphic MRF was obtained by replacing part of the spherical Fe carbonyl microparticles with rod-shaped Fe particles, both types of particles being coated with polysiloxane [99]. The reduction in short-range attraction between the carbonyl microparticles and the rod-shaped particles led to an improvement in redispersability. Lim and collab. have also added graphite nanofibers in a suspension containing Fe carbonyl particles, demonstrating the increase in sedimentation resistance and an improved stability towards flocculation, with no significant macroscopic changes in MR behavior [60]. The nanofibers filling the gaps between the Fe carbonyl microspheres reduce the physical contact between them, impeding the formation of a dense layer following sedimentation.

2.1.6. FERROFLUIDS BASED MAGNETORHEOLOGICAL FLUIDS (FF-MRFs)

Among the different types of MRFs, Fe particle suspensions based on ferrofluids represent promising candidates for exploiting both the advantages of ferrofluids (kinetic stability), as well as those of conventional MRFs (strong MR and MV effects) [17, 100], and they were investigated in a large number of papers [81, 101–109].

How do FF-MRFs behave?

In the absence of a magnetic field only the ferrofluid's magnetic nanoparticles have magnetic moments, and those are randomly oriented. These

coated nanoparticles are practically permanent nanomagnets that will coat the Fe microparticles, preventing their direct contact. As a consequence, there will be a reduction in the formation of solid sediments, and redispersability is improved [101, 103, 110]. Due to the coating of the Fe microparticles with magnetite nanoparticles the abrasion effect is also reduced, the ferrofluid acting as a lubricant.

The presence of an external magnetic field magnetizes the Fe microparticles, and the magnetic moments tend to align with the field lines. As a result, the suspension will have a very large magnetization, even for a relatively low Fe particle concentration, which means it will have a good sealing capacity. Resulting from the intensification of the interactions of the multidomain particles owed to the base ferrofluid's magnetic permeability, the MR and MV effects are stronger than those of conventional MRFs with the same solid volume fraction [111, 112, 103]. This leads to a particularly good damping capacity.

Under the influence of a uniform magnetic field, larger nanoparticles and nanoparticle groups from the base ferrofluid are attracted to the ferro-magnetic microparticles, forming clouds around them and leading to an efficient repulsion between these microparticles [113, 114]. The formation of the nanoparticle halo is favored when the carrier liquid is a steric stabilized ferrofluid with nanoparticle agglomerations [114], and is inhibited in the case of ferrofluids with no agglomerations, like in the case of electrostatically stabilized water based [115] or ionic fluid [114].

In the presence of an irregular magnetic field, as opposed to conventional MRFs, FF-MRFs show Rosensweig instabilities similar to those of ferrofluids, inducing a structure that is interesting for their use as sealing fluids or MR fluids. This phenomenon was evidenced through X-ray computer micro-tomography investigations [116]. The reconstructed 3D images show the migration of Fe microparticles towards more intense magnetic field regions, without them separating from the base ferrofluid, which permits the formation of the seen peaks and the use of this type of composite in MF seals with low rotation speeds for high pressure differential conditions.

The paper [111] compares the rheological and magneto-rheological behavior of an extreme bidisperse (nano-micro) magnetizable fluid – sample D1 – and a commercial MRF (MRF-140CG, Lord Co., USA) with the same volume fraction of the solid magnetic content. It was shown that the magnetic nanoparticles in the FF-MRF improve the MV effect of the extreme bidisperse magnetic fluid compared to the effects manifested by a conventional MRF with the same solid magnetic content that only has micrometric particles – Fig. 7. The apparent yield stress normalized by the square of the saturation magnetization has a more pronounced increase with the increase of the magnetic field's intensity, although the MR effect is greater for the FF-MRF – Fig. 8.

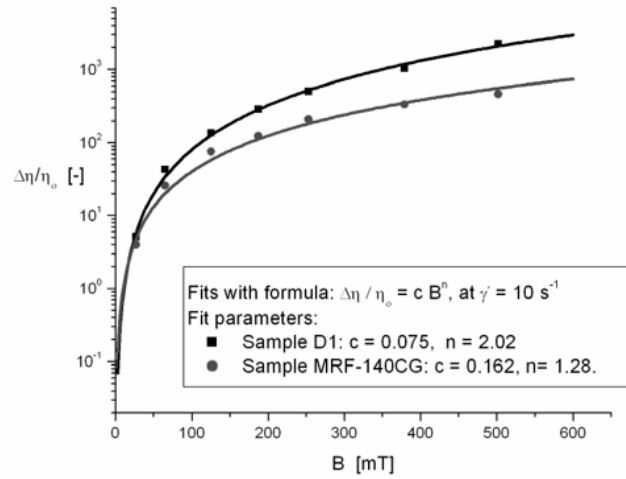


Fig. 7 – MV effect as a function of magnetic field induction at constant shear rate [111]. Reprinted from Journal of Magnetism and Magnetic Materials, **322** (20), Susan-Resiga D., Vékás L., and Bica D., *Flow behaviour of extremely bidisperse magnetizable fluids*, 3166–3172. (Copyright 2010, with permission from Elsevier).

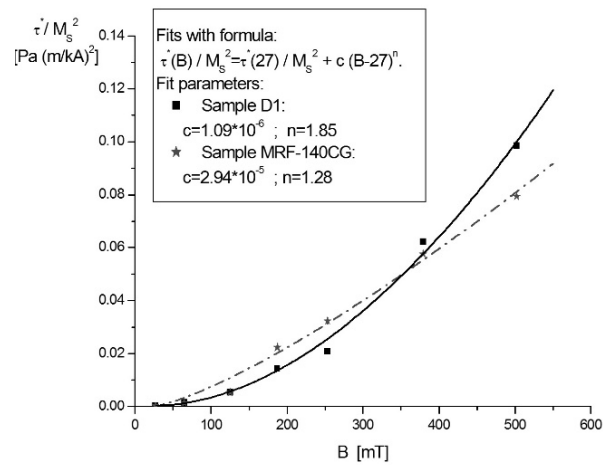


Fig. 8 – Apparent yield stress normalized by the square of the saturation magnetization as a function of the magnetic field's induction – both samples [111]. Reprinted from Journal of Magnetism and Magnetic Materials, **322** (20), Susan-Resiga D., Vékás L., and Bica D., *Flow behaviour of extremely bidisperse magnetizable fluids*, 3166–3172. (Copyright 2010, with permission from Elsevier).

Comparative investigations were also done for the nano-micro bidisperse fluid MRF-LM5 (to which commercial additives were added, in order to prevent sedimentation of particles, and to improve dispersion) and the commercial sample MRF-132 DG (Lord Co., USA), both having a similarly high solid content [33].

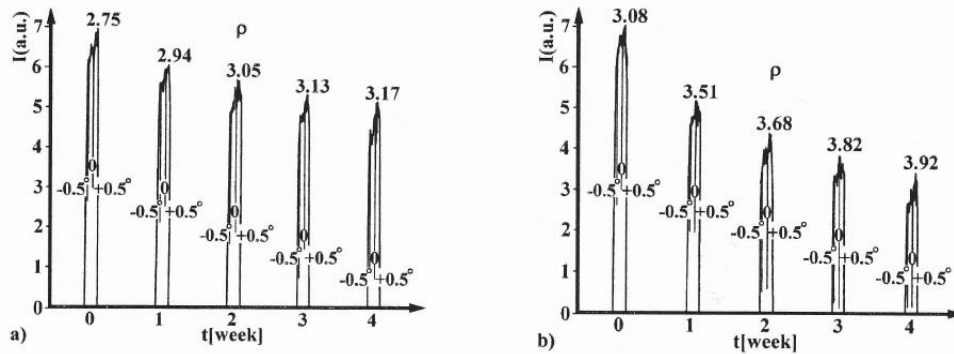


Fig. 9 – Gravitational sedimentation analysis by X-ray transmission over 4 weeks:
a) MRF-LM5; b) MRF-132DG [33].

They showed that the addition of additives in prepared fluids improves stability, reduces sedimentation rate compared to the commercial sample – Fig. 9. X-ray diffraction investigations were done using a Dron-3 type diffractometer with X-rays generated by a molybdenum anode. However, this improvement has proved to be in the detriment of the MV effect (which is smaller), and probably the MR effect too, which was not analyzed in the mentioned paper.

A great advantage of FF-MRFs is the possibility of controlling/tuning their properties/behavior by modifying the composition at the two hierarchical levels, nano and micro [100]. Ample investigations on the rheological and magnetorheological behavior of FF-MRFs with different volume fractions of magnetite nanoparticles in the carrier ferrofluid and Fe microparticles are reported in [112, 107, 106]. The 27 samples analyzed in [112, 107] cover a large range of volume fractions – nano (2.75% – 22.90%) and micro (4% – 44%), the total solid volume fraction reaching 56.8%. The total hydrodynamic volume fraction (which also includes the contribution of the chemo-absorbed organic layer on the surface of the magnetite particles) reached a value possible only due to the extreme bidisperse nature of the suspension, namely 85%.

The ferrofluids in which the Fe particles were dispersed did not have agglomerates, so they were very stable, and therefore they had a Newtonian behavior in the absence of an external magnetic field. This has insured that a low viscosity could be maintained and the MV and MR effects that were researched could be reproduced. The advantages of using FF-MRFs were demonstrated by comparing the intensity of the MR and MV effects for different values of volume

fractions for nano (φ) and micro (Φ_{Fe}) particles for different values of magnetic field induction, with the intensity of these effects in the case of a commercial MRF, namely MRF 140-CG (Lord Co., USA).

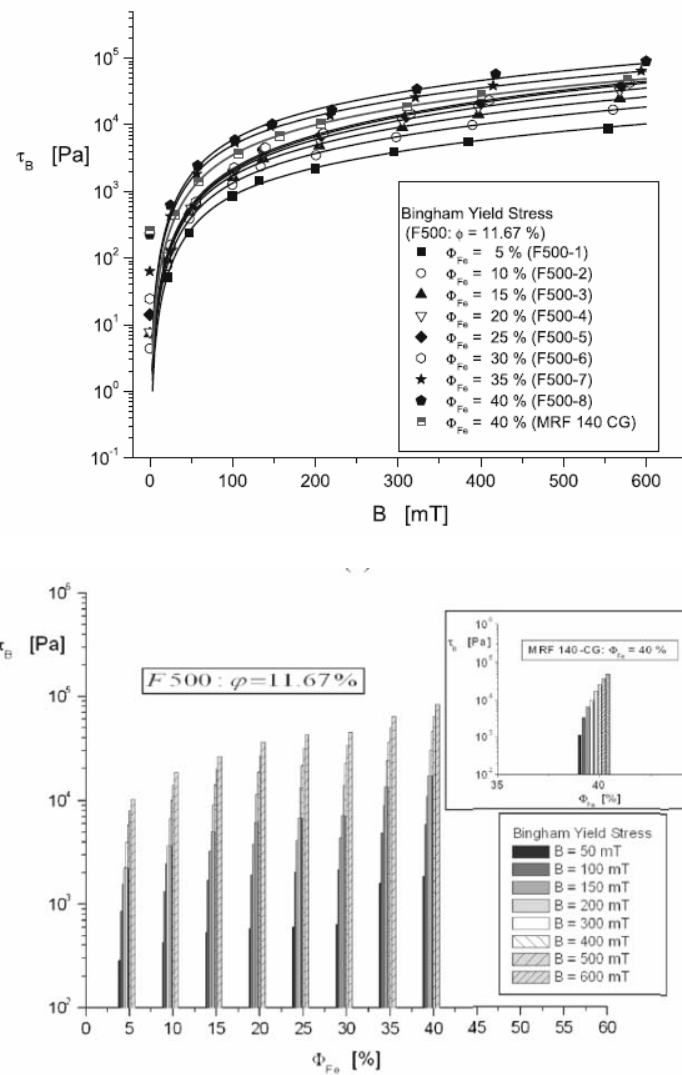


Fig. 10 – Influence of magnetic field induction and Fe particle volume fraction on Bingham yield stress for the F500 set composite samples [107]. Reprinted by permission from Springer Nature Customer Service Centre GmbH, Springer Nature, Rheologica Acta, *Ferrofluid-based magnetorheological fluids: tuning the properties by varying the composition at two hierarchical levels*, Susan-Resiga D., and Vékás L., **55** (7), 581–595, 2016.

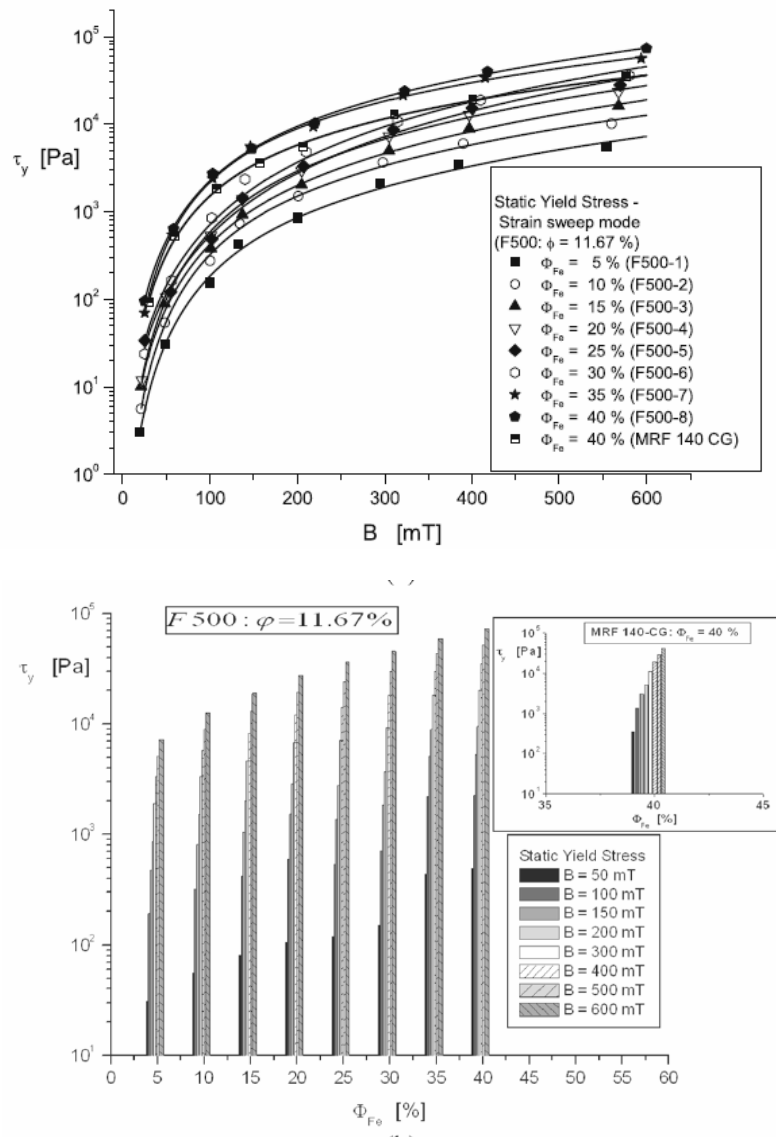


Fig. 11 – Influence of magnetic field induction and Fe particle volume fraction on static yield stress for the F500 set composite samples [107]. Reprinted by permission from Springer Nature Customer Service Centre GmbH, Springer Nature, Rheologica Acta, *Ferrofluid-based magnetorheological fluids: tuning the properties by varying the composition at two hierarchical levels*, Susan-Resiga D., and Vékás L., **55** (7), 581-595, 2016,

Figures. 10 and 11 show the dynamic yield stress (Bingham type) respectively the static yield stress, as a function of the magnetic field induction and the volume fraction of Fe microparticles for the composite set F500 (transformer oil based ferrofluid with 11.56% volume fraction of magnetite nanoparticles, saturation magnetization $M_S = 500$ G, and with different volume fractions of Fe microparticles), compared with MRF 140-CG. The samples of sets F100 and F1000 have similar behaviors.

A saturation tendency of the MR effect can be observed for high concentrations of Fe particles in the presence of intense magnetic fields [107], where particle agglomerations reach maximum size in the given conditions [46]. Similar results were obtained when investigating the MV effect.

Therefore the flow behavior of FF-MRFs is controllable by varying the micro and nano concentrations, as well as the applied magnetic field induction, choosing the optimal $\varphi : \Phi_{Fe} : B$ combination in order to fulfill the requirements of practical applications.

Like in the case of inverse ferrofluids and conventional MRFs, the apparent viscosity data measured for different values of magnetic field induction and different shear rates can be successfully correlated using Mason number M_n for FF-MRFs. Using the approximation of average magnetization and Mason number formula deduced in the case of inverse ferrofluids by Klingenberg and collab. [117], for the case of FF-MRFs the following expression for M_n was obtained [108]:

$$M_n = 72 \frac{\eta_{FF} \dot{\gamma} \Phi_{Fe}^2}{\mu_o \mu_{rFF} [\langle M_{FF-MRF} \rangle - \langle M_{FF} \rangle]^2}. \quad (4)$$

Here η_{FF} represents the continuous media viscosity (the base ferrofluid), $\mu_o = 1.26 \cdot 10^{-6} N/A^2$ is the magnetic permeability of the free space, μ_{rFF} is the relative magnetic permeability of the continuum phase, $\dot{\gamma}$ is the shear rate, Φ_{Fe} is the Fe microparticle volume fraction, $\langle M_{FF} \rangle$ and $\langle M_{FF-MRF} \rangle$ are the average magnetization for the ferrofluid respectively for the bidisperse composite, their values being taken from the magnetization curves. Expression (1) is valid for the entire range of magnetic field induction values and contains only experimentally measured data.

Representing dimensionless viscosity data as a function of Mason number, $\frac{\eta_{app}}{\eta_\infty} = f(M_n)$, we can observe that they overlap on a single master curve for each sample – e.g. Fig. 12 for sample F100–2.

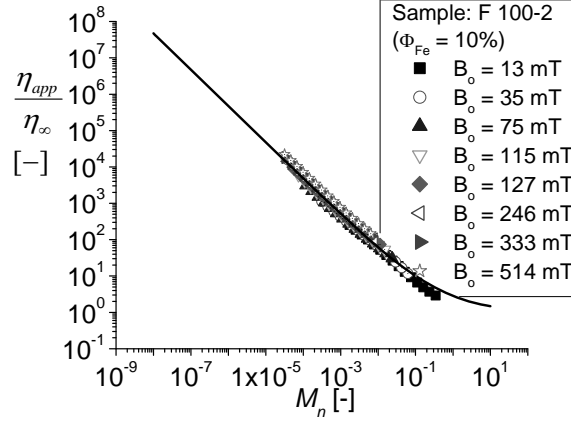


Fig. 12 – Dimensionless viscosity as a function of Mason number, $\frac{\eta_{app}}{\eta_{\infty}} = f(M_n)$, for sample F100-2 at all values of magnetic field induction. Critical Mason number obtained from the fit: $M_n^* = 0.471$ [108]. Reprinted with permission from Susan-Resiga D., and Vékás L., *Ferrofluid based composite fluids: Magnetorheological properties correlated by Mason and Casson numbers*, Journal of Rheology **61**(3), 401–408 (2017); doi: 10.1122/1.4977713. (Copyright 2017, The Society of Rheology).

Because in the case of FF-MRFs the magnetic interactions between the particles are strong, the flow/viscosity curves follow Casson's model [118], the reunited data from Fig. 12 was correlated with Casson formula in its adimensional form:

$$\frac{\eta_{app}}{\eta_{\infty}} = M_n^* M_n^{-1} + 2\sqrt{M_n^* M_n^{-1}} + 1. \quad (5)$$

The fit parameter M_n^* represents critical Mason number, it corresponds to the transition from magnetic to hydrodynamic control of the structure (this transition takes place at the particular value $\dot{\gamma} = \frac{\tau_C}{\eta_{\infty}}$ of shear rate) and depends on the particle volume fraction:

$$M_n^* = 72 \frac{\eta_{FF} \Phi_{Fe}^2}{\mu_o \mu_{rFF} [\langle M_{FF-MRF} \rangle - \langle M_{FF} \rangle]^2} \cdot \frac{\tau_C}{\eta_{\infty}}. \quad (6)$$

The master curves $\frac{\eta_{app}}{\eta_{\infty}} = f(M_n)$ obtained for samples with different nano and micro volume fractions do not overlap. However, we can obtain a more general master curve by representing the adimensional viscosity data for all samples as a function of the inverse of Casson number, C_a (which represents the ratio of magnetic and viscous forces): $\frac{\eta_{app}}{\eta_{\infty}} = f\left(\frac{M_n}{M_n^*} = \frac{1}{C_a}\right)$ – Fig. 13.

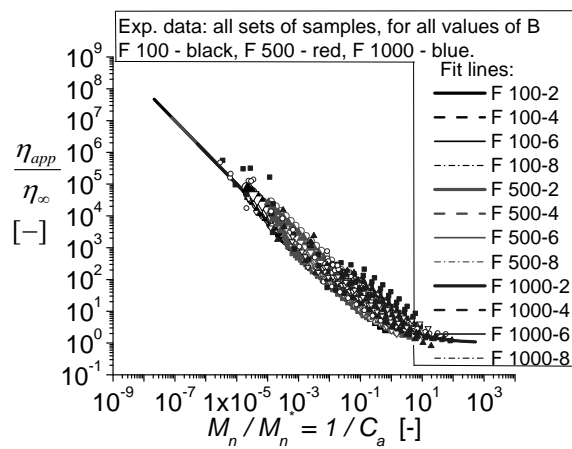


Fig. 13 – Master curve $\frac{\eta_{app}}{\eta_{\infty}} = f\left(\frac{M_n}{M_n^*} = \frac{1}{C_a}\right)$ obtained for different values of magnetite and Fe volume fractions, and for different values of applied magnetic field induction [108]. Reprinted with permission from Susan-Resiga D., and Vékás L., *Ferrofluid based composite fluids: Magnetorheological properties correlated by Mason and Casson numbers*, *Journal of Rheology* **61**(3), 401–408 (2017); doi: 10.1122/1.4977713. (Copyright 2017, The Society of Rheology).

The concentrated samples in set F1000 show a larger dispersion of experimental points – Fig. 13 – probably due to the formation of more complex particle agglomerates compared to the simple chains, and which influence flow behavior.

The master curve in Fig. 13 is useful:

- to determine the apparent viscosity in the case of FF-MRFs with compositions that do not have any associated experimental data;
- to choose an optimal composition for an FF-MRF in order to design a certain MR application.

Similarly, based on the approximation of the average magnetization, for the same FF-MRF samples (with different values for nano and micro volume fractions)

a single master curve was obtained representing the static yield stress as a function of characteristic magnetic stress [109] – Fig. 14:

$$\tau_{char} = \frac{1}{48} \cdot \frac{\mu_o \mu_r FF [\langle M_{FF-MRF} \rangle - \langle M_{FF} \rangle]^2}{\Phi_{Fe}^2}. \quad (7)$$

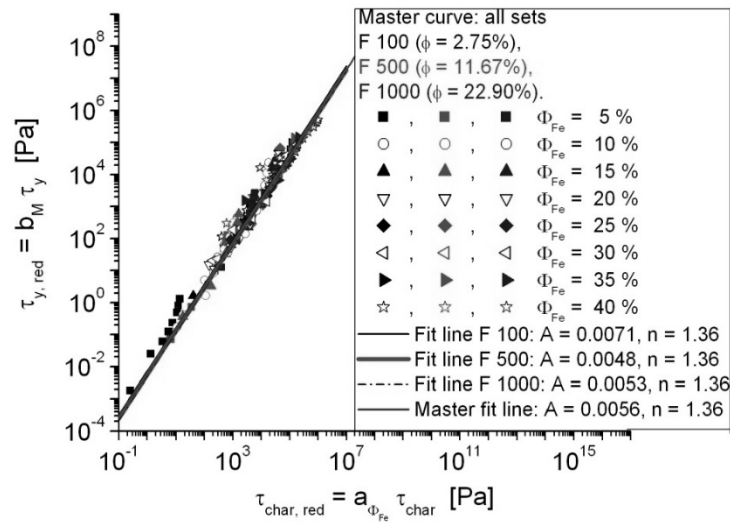


Fig. 14 – Master curve $\tau_{y,red} = f(\tau_{char,red})$ obtained for all samples with different nano and micro concentrations, at different values of magnetic field induction. $a_{\Phi_{Fe}}$ și b_M are horizontal and vertical translation coefficients of experimental data [109]. Reprinted with permission from Susan-Resiga D., and Barvinschi P., *Correlation of rheological properties of ferrofluid-based magnetorheological fluids using the concentration-magnetization superposition*, Journal of Rheology **62**(3), 739–752 (2018); doi: 10.1122/1.5017674. (Copyright 2018, The Society of Rheology).

This master curve, with the same utility as that in Fig. 13, indicates for the first time in literature the concentration – magnetization superposition in the case of FF-MRFs. The results obtained using the finite element method (FEM) in order to calculate the characteristic magnetic stress, are well correlated to experimental results.

Another interesting aspect highlighted in [109] is that for Fe particle volume fraction $\Phi_{Fe}^* = 30\%$ the data reveals a change in magnetorheological behavior for the FF-MRF samples of all three sets (F100, F500, F1000), most likely due to the

transition from chain-type agglomerates (induced by the magnetic field) to more complex structures.

2. 2. APLICATIONS OF NANO- MICROSTRUCTURED MRFs

Due to the obvious advantages of using nano-microstructured MRFs, they tend to gradually replace conventional MRFs in practical applications, such as:

- linear and disc-type dampers [119, 120];
- anti-seismic protection MR systems [18, 121, 122];
- MR brakes [123, 124] and clutches [125] for hydraulic pumps and turbines
- rotating seals and vibration dampers [106];
- polishing techniques [126, 127, 128, 25];
- rotating leak-less seals for gas environments [129];
- hydraulic systems for helicopter landing gears [130];
- dampers for helicopter rotors [131];
- magnetoresistors, transducers and magnetic field sensors [74, 75];
- optic traps [73];
- biomedical applications [132] and others.

3. CONCLUSIONS

Nano-microstructured magnetorheological fluids combine the advantages of ferro-fluids – a good colloidal stability – with those of conventional magnetorheological fluids – intense MR and MV effects and more control levers for flow behavior (applied magnetic field induction, volume fractions for nano and microparticles). In addition, compared to conventional MRFs, bidisperse fluids show reduced abrasion and better re-dispersability of particles.

The current tendency for FF-MRFs is to replace conventional MRFs in practical applications.

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