MAGNETIC AND FLOW PROPERTIES OF HIGH MAGNETIZATION NANOFLUIDS

Ladislau VÉKÁS, Senior Researcher*

Laboratory of Magnetic Fluids, Center of Advanced Research in Engineering Sciences Romanian Academy – Timisoara Division and National Center for Engineering of Systems with Complex Fluids

"Politehnica" University of Timisoara

Oana Marinică, Researcher

Institute for Complex Fluids
"Politehnica" University of Timisoara

Daniela SUSAN-RESIGA, Assoc. Prof.

National Center for Engineering of Systems with Complex Fluids
"Politehnica" University of Timisoara

Floriana D. STOIAN, Assoc. Prof.

Department of Thermal Machines and Transport and National Research Center for Engineering of Systems with Complex Fluids
"Politehnica" University of Timisoara

Doina BICA, Senior Researcher

Laboratory of Magnetic Fluids, Center of Advanced Research in Engineering Sciences
Romanian Academy - Timisoara Division

Corresponding author: Bv Mihai Viteazul 1, 300222, Timisoara, Romania

Tel.: (+40) 256 403700, Fax: (+40) 256 403700, Email: vekas@acad-tim.tm.edu.ro

ABSTRACT

The paper reviews recent experimental results concerning a wide variety of magnetic nanofluids with high saturation magnetization. Magnetic, rheological and magneto-rheological properties are presented and discussed, especially related to nanostructural processes and colloidal stability in applied magnetic field.

KEYWORDS

Magnetic nanoparticles, magnetic nanofluids, magnetic properties, flow properties

NOMENCLATURE

M_s	[A/m]	saturation magnetization
H	[A/m]	intensity of applied magnetic field
χ_i	[-]	initial susceptivity
$<D_m>$	[nm]	mean magnetic diameter
σ	[nm]	standard deviation
δ	[nm]	thickness of the surfactant layer(s)
D	[nm]	physical diameter of nanoparticles
D_h	[nm]	hydrodynamic diameter of nanoparticles
φ	[-]	physical volume fraction
φ_h	[-]	hydrodynamic volume fraction
φ_ρ	[-]	solid particle volume fraction
φ_m	[-]	maximum hydrodynamic volume fraction
η	[Pa·s]	dynamic viscosity of the magnetic fluid
η_o	[Pa·s]	dynamic viscosity of the carrier liquid
$[\eta]$	[Pa·s]	intrinsic viscosity
p	[-]	fit parameter
A	[-]	coupling coefficient
t	[^{o}C]	temperature

Subscripts and Superscripts

i	initial
s	saturation
m	maximum
h	hydrodynamic
ρ	solid particle
0	carrier liquid viscosity

ABBREVIATIONS

OA	oleic acid
SA	stearic acid
LA	lauric acid
DBS	dodecylbenzensulphonic acid

MA   miristic acid
TPT   thermodynamic perturbation theory
SANS   small angle neutron scattering
MEK   methyl-ethyl-ketone based sample
iso-But iso-butanol based sample
P   pentanol based sample
W   water based sample
Tr30   transformer oil based sample

1. INTRODUCTION

The interest of our group for concentrated magnetic nanofluids, at first, was related to a promising application in the field of hydraulic machines, in particular to the magneto-hydrodynamic torque converters [1, 2, 3]. Later on, also other applications were considered, such as high pressure magnetofluidic rotating seals [4] and bearings [5]. The successful operation of an MHD torque converter is related to the possibility of intense macroscopic rotation of magnetic fluids to be induced by a rotating magnetic field, as a consequence of the non-symmetric character of the stress tensor of a magnetic fluid [6] with magnetic nanoparticles having rigid dipole moment, specific to the so called magnetic fluids with internal rotation [6]. This potential application has foreseen a very high magnetization and low viscosity magnetic fluid. The other two applications need also high magnetization magnetic fluids, but preferably with soft dipoles.

The preparation and manifold characterization of high magnetization magnetic nanofluids on non-polar and polar carrier liquids is a problem of current interest and involves, among others, investigations on efficient colloidal stabilization mechanisms, dipole-dipole interactions and particle structure formation processes, the influence of particle volume fraction (magnetization) and agglomerates on flow properties in magnetic field. More recently, other potential uses of magnetizable fluids, like magnetically controlled convective and boiling heat transfer processes [7, 8] or semi-active dampers with magneto-rheological fluid [9], require specially tailored fluids with high magnetization and sometimes with more complex structure on nanometric and/or micrometric scale, compared to usual magnetic nanofluids.

Macroscopically, the introduction of magnetic forces into the fundamental hydrodynamic equations for the quasihomogeneous magnetizable liquid medium gives rise to the magnetohydrodynamics of magnetic nanofluids (ferrohydrodynamics) [10]. Magnetic forces governing these phenomena are completely different from the forces in the classical MHD of electroconductive fluids.

From a microscopic point of view, long-range, attractive van der Waals and magnetic forces are ubiquitous and therefore must be balanced by Coulombic, steric or other interactions to control the colloidal stability of dispersed nanoparticle system, even in intense and strongly non-uniform magnetic field, specific to most of the applications. As the magnetization (particle volume fraction) is raised, the mean interparticle distance diminishes and attractive interactions become stronger. Stabilization mechanisms should prevent the irreversible agglomeration of nanoparticles even in intense non-uniform magnetic field, which is a fundamental requirement, sometimes difficult to be fulfilled. Magnetic and flow properties of magnetic fluids are strongly influenced by structure formation processes and characterize their quality *vs.* envisaged applications.

2. SYNTHESIS OF MAGNETIC NANO-PARTICLES AND MAGNETIZABLE FLUIDS

The synthesis of magnetic fluids has two main steps: (a) the preparation of nano-sized magnetic particles and (b) the subsequent dispersion of the nanoparticles in various non-polar and polar carrier liquids. In what concerns the ferrite nanoparticles, the most efficient route is the chemical co-precipitation process. Details of the multi-step procedures applied at the Laboratory of magnetic fluids from Timisoara are given in [11, 12].

The *synthesis of surfactant covered magnetite nanoparticles* has the following main steps: co-precipitation of magnetite (or other ferrite) (NH_4OH in excess) → subdomain magnetite particles → sterical stabilization (chemisorbtion, e.g. of oleic acid) → phase separation → magnetic decantation and repeated washing → monolayer covered magnetite nanoparticles + free oleic acid → extraction of monolayer covered magnetite nanoparticles (flocculation) → *stabilized magnetite nanoparticles.*

Cobalt ferrite and γ-Fe_2O_3 nanoparticles were also prepared, but applying a somewhat different procedure and using $NaOH$ in excess, instead of NH_4OH. Cobalt ferrite has much larger magnetic anisotropy than magnetite, consequently these nanoparticles correspond to "rigid dipoles".

Besides oleic acid, various other surfactants, with different chain length values, were used to cover nanoparticles to be dispersed in various organic or water carrier liquids, e.g. stearic acid, lauric acid, dodecylbenzen-sulphonic acid or miristic acid.

Further, the procedure of *magnetic nanofluids preparation* depends on the non-polar or polar character of

the organic carrier liquid. The stable dispersion of surface covered nanoparticles requires to properly adapt their interfacial properties to the carrier liquid, which is an important part of the preparation procedure.

The preparation of *water based magnetic fluids* follows a somewhat different route than in the case of organic carriers and requires a more troublesome procedure, due to supplementary difficulties in satisfactory stabilization/dispersion of the magnetic nanoparticles in the strongly polar anorganic carrier. The degree of colloidal stability of water-based magnetic fluids depends on the pH value of the medium and the *electrosteric* stabilization mechanism is specific mainly to water based magnetic fluids. When lauric acid is used as surfactant bilayer, then the preparation process is similar to that outlined for non-polar solvents, because in this case the coprecipitation step is followed also by phase separation.

By applying the procedures described in [11, 12], a wide variety of magnetic nanofluids were prepared on more then 50 different carrier liquids. Specially tailored magnetic fluids were used also to prepare nano-microstructured magnetorheological fluids. In this case micrometer ranged iron particles are dispersed in a concentrated magnetic nanofluid [13], the stability of the suspension being ensured by covering of large Fe particles with magnetite nanoparticles [9].

In what follows, the magnetic and flow properties of magnetic fluids are reviewed, depending on their concentration, composition and colloidal stability.

3. MAGNETIC PROPERTIES VS. STABILITY

Magnetization curves can be extensively used for the study of both particle interactions and agglomerate formation, processes that strongly influence the rheological and magnetoheological behaviour of magnetic fluids. Saturation magnetization, initial susceptivity*,* full magnetization curves ($M = M(H)$ or $M/M_s(H)$) and magneto-granulometric analysis (mean magnetic diameter and standard deviation), at various values of the volumic concentration of magnetic nanoparticles, give an insight on microstructural characteristics of various samples to be compared [14-17]. For example, the initial susceptivity is influenced by particle diameter and size distribution, particle interactions, existence of preformed aggregates during the preparation process and aggregate formation at zero field or induced by the applied magnetic field. As it was shown in detail in [18], the comparison of the dependence of the initial susceptibility on the physical volume fraction, φ, of magnetic nanoparticles for samples of different types of magnetic fluids, re-

veals interesting microstructural aspects. For example, particle agglomerates, which behave as large particles, will determine this part of the magnetization curve.

Composition details, in particular the quality of the surfactant used, as well as its particular adsorbtion properties to the nanoparticle surface, influence particle agglomeration processes. It was shown in [18] that double layer sterically stabilized pentanol magnetic fluids have smaller initial susceptivity, compared to other, less well stabilized samples of the same concentration and quasilinear $\chi_i = \chi_i(\varphi)$ dependence, at least for small φ values, i. e. agglomerates are practically absent in this case.

In order to exemplify, in Fig. 1 [19], water based magnetic fluid samples, with various magnetization and different double layer coating of particles, are compared to a high magnetization pentanol based sample, with *OA+DBS* double layer covered magnetite nanoparticles. The concentrated pentanol sample has the lowest initial susceptivity, as a qualitative examination of the initial, low field part, of the magnetization curves show. This behavior is a consequence of the degree of colloidal stability of samples, i.e. water based samples are, in general, less stable than the pentanol based one and the existing agglomerates are at the origin of the relatively great values of χ_i. An interesting exception is the *MA+DBS* double-layer stabilized water based sample, which is close to the pentanol based one, however it has four times lower magnetization (concentration). The very good stability of pentanol based samples is evidenced also by comparison with non-polar, transformer oil based magnetic fluids, Fig. 2 [18]. The quasilinear dependence in the case of pentanol based samples denotes that dipolar interactions and the fraction of agglomerates are negligible. Full curves are fits to the thermodynamic perturbation theory (*TPT*) driven model [18]. Also, the comparison reveals that technical grade oleic acid surfactant used in the case of transformer oil samples is less efficient than the chemically pure *OA* layer, which is better chemisorbed to the surface of nanoparticles.

Magnetic properties, the magnetization curve and especially its low field part, give an insight to the nanostructure of magnetic fluids and indicate the presence of agglomerates. Pre-existing agglomerates, e.g. in water based magnetic fluids or field induced reversible or irreversible agglomerates in organic carriers, have a significant influence on the long-term stability and flow properties of magnetic fluids. These data may be correlated with *direct probing* of nanostructures in magnetic fluids by small angle neutron scattering (*SANS*) [20].
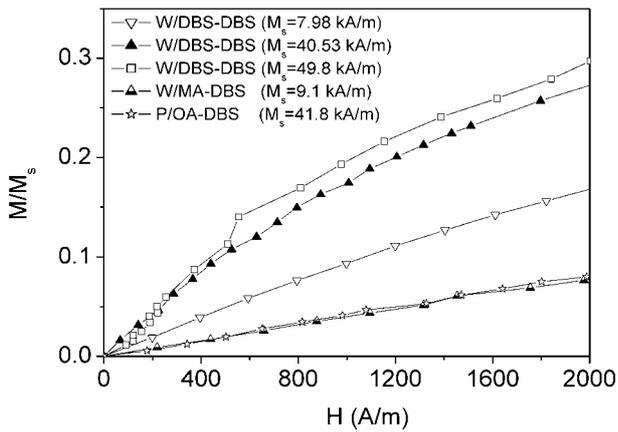
Figure 1. Reduced magnetization curves at low fields for water and pentanol based samples
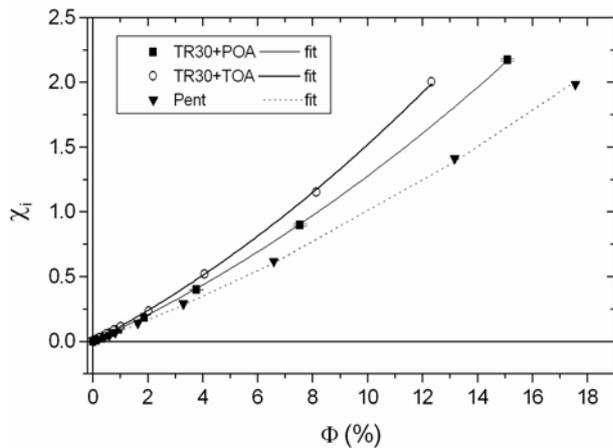

Figure 2. Dependence of initial susceptivity on physical volume fraction of magnetic nanoparticle

3.1. Flow properties. Magnetoviscous effect vs. colloidal stability

The chemisorbed first layer and for polar carriers, the physically adsorbed secondary surfactant layer gives rise to an "effective" surface covering of nanoparticles, whose thickness have to be added to the physical dimension of the nanoparticles when the particle motion relative to the carrier liquid is under consideration. This motion of the surfactant covered nanoparticles is determined by the "hydrodynamic" diameter, $D_h=D+2\delta$, of particles. At the nanostructural level, the hydrodynamic diameter and the characteristic dimension and of agglomerates, if any, will determine the flow properties of magnetic fluids. Macroscopically, the "hydrodynamic" volume fraction of magnetic particles and the applied magnetic field will be the main factors influencing the flow behaviour. The flow properties will ultimately reflect also the degree of colloidal stability of various kind of magnetic fluids.

□ *Dependence of viscosity on the hydrodynamic volume fraction*

The dependence of nondimensional viscosity, η / η_o on the solid particle volume fraction may be determined using the Vand formula, as shown in [17]. In order to determine the hydrodynamic volume fraction, i.e. the effective volume fraction of the double-layer-covered Fe_3O_4 nanoparticles, the experimental data were fitted to the Vand formula using the fit parameter $p = \varphi_h / \varphi_\rho$:

$$\eta / \eta_o = exp\left[\left(2.5\,p\varphi_\rho + 2.7\,p^2\varphi_\rho^2\right)\right]/ \left(1 - 0.609\,p\varphi_\rho\right) \tag{1}$$

The fitted $\eta / \eta_o = f\left(\varphi_\rho\right)$ curves at various temperatures, together with the detailed results of the fits with the formulae of Vand, Krieger-Dougherty, Quemada, Chong, Rosensweig and Chow, for various hydrocarbon and alcohol based samples, may be found in [15-18], while in [21], a thorough comparative analysis of these formulae relative to the flow behavior of a wide variety of magnetic fluids was presented. Using the fitted p values, the effective mean surfactant layer thickness may be obtained, $\delta = (p^{1/3} - 1)D / 2$.

The values of the maximum hydrodynamic volume fraction were determined by fitting the data to the well-known two-parameter formula of Krieger-Dougherty:

$$\eta / \eta_o = \left(1 - \varphi_h / \varphi_m\right)^{-[\eta]\varphi_m} \tag{2}.$$

For spherical particles $[\eta] = 2.5$, while for other shapes its value is greater. Fits to various magnetic fluids gave values for the intrinsic viscosity between 2.7 and 3.0.

In [22, 23] T.S.Chow performed a thorough theoretical analysis of concentrated suspensions, taking into account the contribution of many-body particle interactions on the effective viscosity. On the basis of a liquid lattice model, the low-shear limiting viscosity resulted as

$$\eta / \eta_o = exp\left(\frac{2.5\varphi_h}{1 - \varphi_h}\right) + \frac{A\varphi_h^2}{1 - A\varphi_h^2\varphi_m} \tag{3}.$$

Without considering dipole-dipole type interactions between particles, the theoretical value of A was determined to be 4.67.

The theoretical formula (Eq. 3) obtained by Chow proved to be well fitted by viscosity data for various magnetic fluids, but only of *high degree of colloidal stability*, as it follows from detailed investigations on various type of magnetic fluids [21]. The fitted values of A are relatively close to the theoretical one for $\varphi_h \le 0.45$ and at temperature values above 50^oC.

At higher values of the hydrodynamic volume fraction, the resulting A value is lower, evidencing the role of dipolar interactions at close packing. At high volume fraction values the mean interparticle distance is decreasing and approaches the minimum at the maximum hydrodynamic volume fraction, approx. *0.60-0.65*. The distance between the magnetic core of nanoparticles will depend on the thickness of the stabilizing layer, δ. The analysis of this dependence made for a large variety of mono- and double-layer sterically stabilized magnetic fluids [21] indeed evidence the influence of δ.
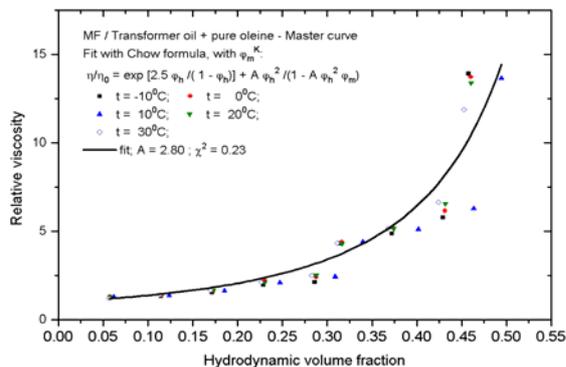
Figure 3. Dependence of relative viscosity on the hydrodynamic volume fraction for a set of transformer oil based magnetic fluids, with nanoparticles stabilized with chemically pure oleic acid monolayer. Fit to Chow formula (for data obtained for $t = -10^oC \div 30^oC$)

In Fig. 3 and Fig. 4 the results of fits to the Chow formula (3) are given for two different type of magnetic fluids, one with non-polar carrier (transformer oil) and monolayer surfacted magnetic particles (Fig. 3) and an other, with polar carrier (heptanol) and double layer covered particles (Fig. 4). Both sets of samples showed excellent colloidal stability.

The results of the fits give for the coupling parameter A rather different values, *A=2.80* for the monolayer stabilized sample and *A=4.52* for the magnetic fluid with double-layer covered nanoparticles, i.e. the last is practically the same as the theoretical value *4.67* valid for non-interacting particles. These results clearly reveal the role of the mean thickness δ of the stabilizing layer(s). For polar samples, like the heptanol based one, the value of δ is almost twice greater than the thickness of *OA* monolayer specific for the non-polar transformer oil samples (*2.25 nm* vs. *1.6 nm* [21]). Taking into account the above difference in the interparticle distance at close packing of particles, i.e. at high saturation magnetization values, the influence of dipolar interactions in the case of transformer oil samples have to be much more significant and this explains the reduced value of A obtained in this case.
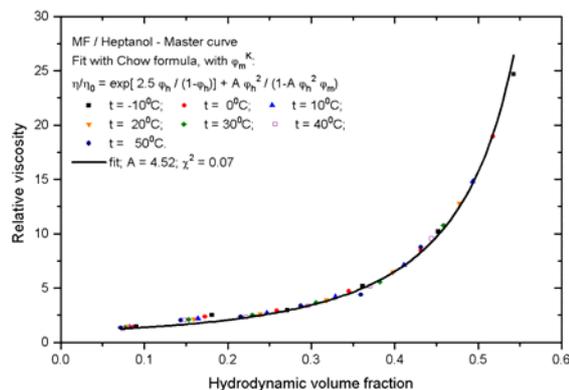

Figure 4. Dependence of relative viscosity on the hydrodynamic volume fraction for a set of heptanol based magnetic fluids, with nanoparticles stabilized with chemically pure oleic acid and dodecylbenzen-sulphonic acid double-layer. Fit to Chow formula (for data obtained for $t=-10^oC \div 50^oC$)

From detailed analysis of similar data for several sets of non-polar and polar magnetic fluid samples it follows [20, 21] that the effective thickness δ and, consequently, the coupling parameter A, are slightly temperature dependent.

Fits of the type given in Figs. 3 and 4 are possible only for magnetic fluids of high colloidal stability, like hydrocarbon or alcohol (C3-C9) based samples, but not for water based magnetic fluids, which are much less stable. Indeed, if we compare the flow properties of pentanol and water based magnetic fluids, qualitative differences show up. In Fig. 5 the shear rate dependence of viscosity of such samples are compared [20]. The strong decrease of viscosity observed for the medium magnetization (*LA+DBS; 22.69 kA/m*) water based sample denotes the existence of agglomerates, preformed during the preparation process.
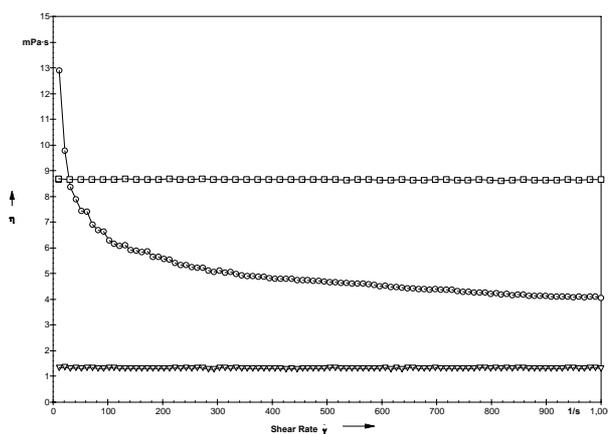

Figure 5. Shear-rate dependence of the viscosity for water-based and pentanol-based magnetic fluid samples: Δ *triangles* - water (*LA+LA; 6.37 kA/m*); ° *circles* - water (*LA+ DBS; 18.71 kA/m*); □ *squares* - pentanol (*OA+DBS; 22.29 kA/m*)

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□ *Magneto-rheological behavior*

Under the influence of magnetic field, magnetic fluids increase their effective viscosity owing to the supplementary dissipation due to the motion of "rigid dipole" type particles relative to the surrounding carrier liquid. Much more important is the effect of pre-existent or field induced agglomerates on the flow properties in magnetic field. In the case of magnetite magnetic fluids, the "rigid dipole" type dissipations are negligible, because for magnetite nanoparticles below *10 nm*, the relaxation of magnetization is mainly due to Nèel mechanism.

The effect of magnetic field on flow properties were investigated using special magneto-rheological (MR) measuring cells, with cylindrical or plate-plate geometry [17].

In Fig. 6 there are compared the magneto-rheological behavior of two organic liquid based magnetic fluids [19].

The methyl-ethyl-ketone based sample is highly polar and it is characterized by intense field induced process of agglomeration, while the iso-butanol based sample is less polar and it is of high colloidal stability, similar to pentanol based magnetic fluids. The strongly shear rate dependent viscosity of the *MEK* sample denotes the role of agglomerates in changing the flow properties. On the contrary, only a small, below *10%*, viscosity increase was observed for the *iso-But* sample, with stable colloidal structure.

The relative increase of effective viscosity, *[η(B)-η(0)]/η(0)*, is a very useful quantity in describing the magnetoviscous effect [24]. In [24] the significant role of the microscopic make-up of ferrofluids on their flow behavior is argumented by detailed experimental data. From the measured data for the *MEK* and *iso-But* samples, the relative increase of effective viscosity is given in Fig. 7 [19] for two different shear rate values. The viscosity increase is practically zero for the highly stable *iso-But* sample.

However, for the *MEK* based sample, the relative increase is more than two-fold at the lower shear rate value. Increasing the shear rate, the agglomerates are progressively destroyed and the effective viscosity change reduces. The magnetorheological effect observed for the two magnetic fluids clearly evidence the existing differences in colloidal stability of samples. The double surfactant layer of *DBS* is much more efficient in the case of i-butanol, then for the much more polar *MEK* carrier.

The influence of the field-induced agglomerates on the flow behavior is well illustrated in Fig. 8 in the case of a *MEK* based sample. Increasing the magnetic induction *B* of the applied field, the initially Newtonian character of the flow successively changes to strongly non-Newtonian.
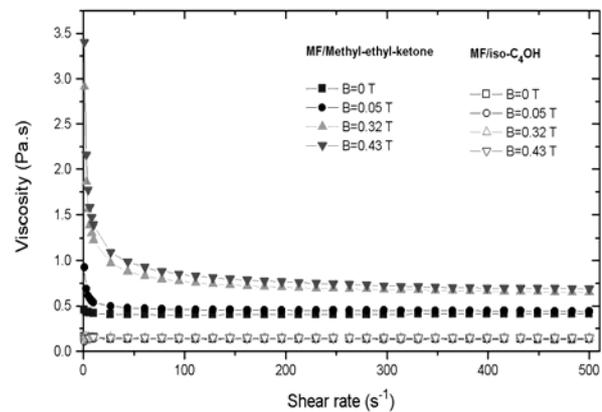

Figure 6. Viscosity vs. shear rate at various values of magnetic induction *B* for magnetic fluids with methyl-ethyl-ketone and i-butanol as carrier liquids: stabilizant double layer *DBS+DBS*; saturation magnetization of samples ≈*39.8 kA/m* (MR cell with plate-plate geometry)
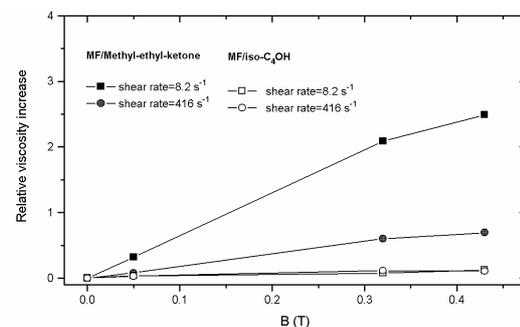

Figure 7. Relative increase of effective viscosity vs. magnetic induction of the applied magnetic field: magnetic fluids with *MEK* and *iso-But* as carrier liquids; stabilizant double layers: *DBS+DBS* (*MEK*) and *OA+DBS* (*iso-But*); saturation magnetization of samples ≈*39.8 kA/m* (MR cell with plate-plate geometry)
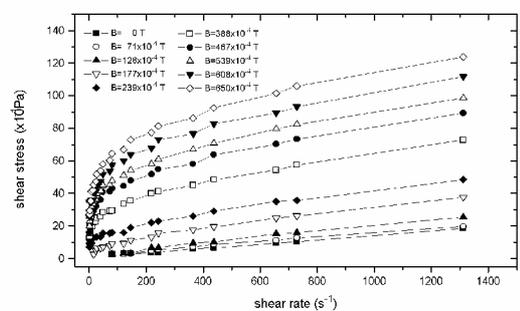

Figure 8. The shear stress-shear rate flow curves for a *MEK* based sample of medium magnetization (25.48 kA/m) under the influence of magnetic field (cylindrical MR cell)

4. CONCLUSIONS

The magnetic and flow behavior of concentrated magnetic nanofluids with high saturation magnetization depend on composition details and preparation procedure, especially on the stabilization/dispersion process of magnetic nanoparticles in various carrier liquids.

High saturation magnetization samples, up to the upper physical limit specific to magnetite nanoparticles (approx. *79.62 kA/m*), were obtained and analyzed. Their colloidal stability, as well as magnetic and flow properties, allowed us to use them in high performance rotating seal manufacturing [25], magnetically controlled heat transfer processes [26] and sensor and transducer [27] applications, as well as for synthesis of magnetic nanocomposite materials [28].

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