88th International Bunsen-Discussion Meeting

Magnetic Colloidal Fluids:
Preparation, Characterization, Physical Properties and Applications
July 20 – 22, 2005

Saarland University, Saarbrücken

Organization

R. Hempelmann, H. Janocha, M. Lücke
Saarland University

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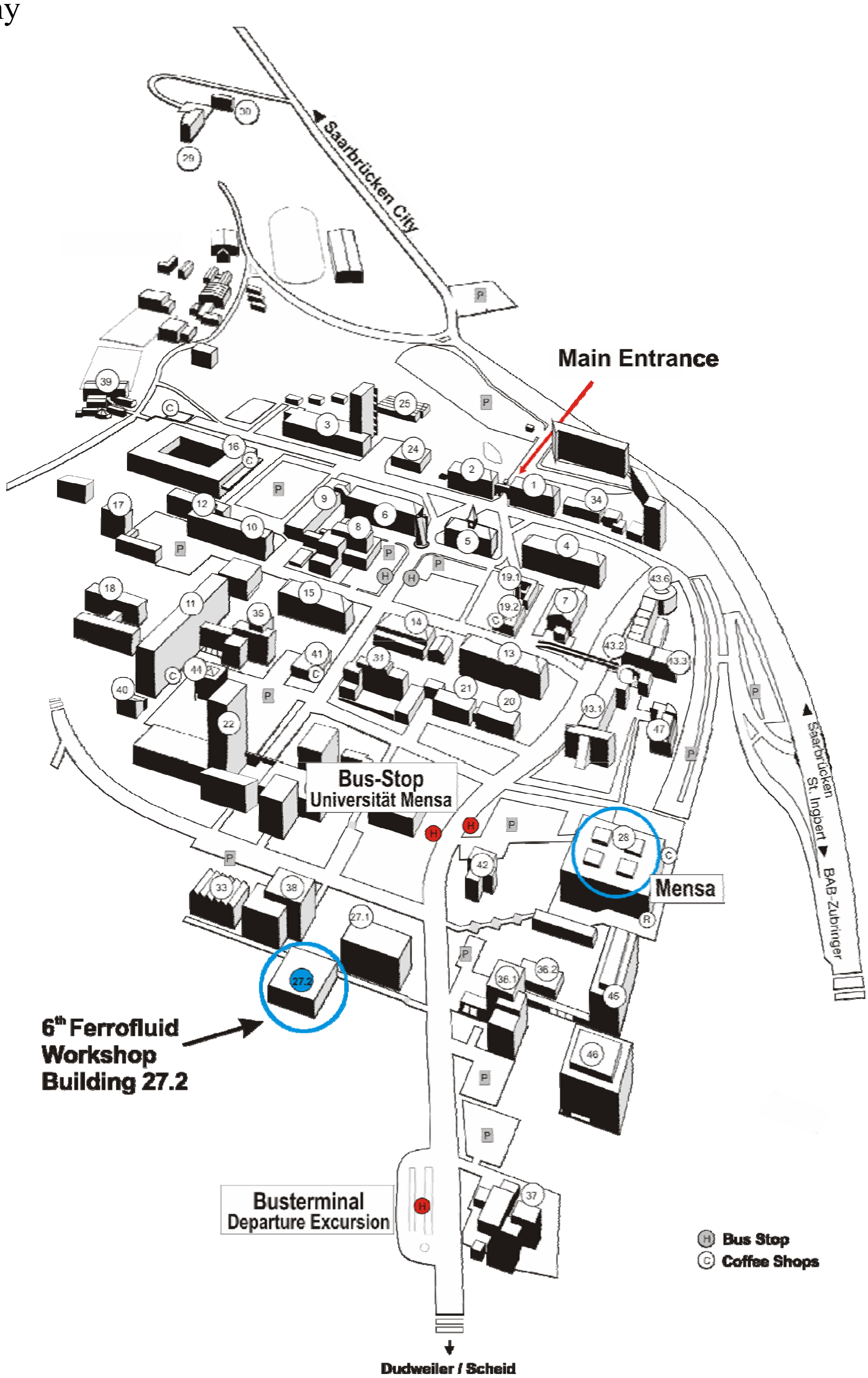

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Time–Table–Overview

Tuesday, 19 July 2005 19:00

Reception in historical Saarbrücken Townhall (at the corner Betzenstraße/ Großherzog-Friedrichstraße, bus stop Johanniskirche or Rathaus)

Wednesday, 20 July 2005	**Thursday, 21 July 2005**	**Friday, 22 July 2005**
9:00 – 10:35 **Synthesis** T1 – T4	9:00 – 10:35 **Modelling I** T14 – T17	9:00 – 10:35 **Biomedical Applications** I T23 – T26
10:35 – 11:00 **Coffee-Break**	10:35 – 11:00 **Coffee-Break**	10:35 – 11:00 **Coffee-Break**
11:00 – 12:20 **Physical Properties** I T5 – T8	11:00 – 12:55 **Modelling II** T18 – T22	11:00 – 12:20 **Biomedical Applications II** T27 – T30
12:30 **Lunch**	13:00 **Lunch**	12:30 **Closing remarks** **Lunch**
14:00 – 15:00 **Physical Properties II** T9 – T10	From 14:30 Uhr **Excursion,** **Dinner**	
15:00 – 17:00 **Postersession** P1 – P66	19:00 **Conference Dinner** **Cloef-Atrium**	
17:00 – 18:00 **Technical Applications** T11 – T13		

Wednesday, 20 July 2005

Synthesis:
Chair: S. Odenbach

T1	9:00	H. Bönnemann	Airstable cobalt-, iron- and iron/cobalt nano-colloids and ferrofluids
T2	9:35	P. Dallas, I. Rabias, D. Niarchos	Preparation of ferrofluids and the effect of organic molecules absorption in the magnetic properties of magnetite nano-particles
T3	9:55	R. Müller, H. Steinmetz, M. Zeisberger, C. Schmidt, R. Hergt, W. Gawalek	Precipated iron oxide particles by cyclic growth
T4	10:15	U. Kreibig, A. Reinholdt, V. Schneider, A. Tillmanns, T. Weirich	An alternative way to ferrofluids
	10:35	Coffee-Break	

Physical Properties I:
Chair: A. Schmidt

T5	11:00	A. Langer, C. Mayer	Characterization of magnetic fluids by nuclear magnetic resonance
T6	11:20	S. Bohlius, H. R. Brand, H. Pleiner	Macroscopic dynamics of uniaxial magnetic gels
T7	11.40	P. Kopčanský, M. Koneracká, M. Timko, I. Potočová, J. Jadzyn	The structural transitions in ferronematics and ferronematic droplets
T8	12:00	G. Mériguet, E. Dubois, V. Dupuis, B. Farago, R. Perzynski	Dynamical properties of ferrofluids in zero and applied magnetic fields
	12.30	Lunch	

Physical Properties II:
Chair: L. Trahms

T9	14:00	A. P. Philipse	Monodisperse magnetic colloids with tunable dipolar interactions
T10	14:35	A. Wiedenmann, R. P. May, D. Dewhurst, J. Haug, A. Heinemann, M. Kammel, U. Keiderling	Time dependence of field induced ordering processes in ferrofluids studied by small angle neutron scattering

Technical Applications:
Chair: N. Bayat

T11	17:00	H. Koser, L. Mao	Modeling a MEMS ferrofluidic pump
T12	17:20	P. Weisgerber, H. Janocha	The ALAS project: Development of a new mounting with magnetorheological fluid for the automotive sector
T13	17:40	H. M. Sauer, E. Cura, L. H. Lie, S. Spiekermann, R. Hempelmann	Microwave induced bonding using nano-scaled ferrites

Thursday, 21 July 2005

Modelling I:
Chair: A. Engel

T14	9:00	P. Leiderer	Magnetic colloids as model systems for condensed matter
T15	9.35	I. Szalai, S. Dietrich	Phase diagrams and magnetic susceptibility of ferrofluids
T16	9:55	K. I. Morozov	The dielectric virial expansion and the models of dipolar hard sphere fluids
T17	10:15	A. Zubarev, L. Iskakova	Rheological properties of ferrofluids with drop-like aggregates
	10:30	Coffee-Break	

Modelling II:
Chair: A. Ivanov

T18	11:00	J.-C. Bacri	Magnetic nanoparticles: a tool for micro-rheology in living cells
T19	11:35	V. Becker, A. Engel	Thermal ratchet effect in a rotating ferrofluid
T20	11.55	R. Richter, I. Barashenkov	Two-dimensional solitons on the surface of magnetic fluids
T21	12:15	P. Ilg, M. Kröger, S. Hess	Dynamics and magnetoviscosity of ferro-fluids: Comparison of many-particle simulations and dynamical mean-field theory

T22	12.35	J. P. Huang, Z. Wang, C. Holm	Computer simulations of the structure of colloidal ferrofluids
	13:00	Lunch	
	14:30	Excursion	
	19:00	Conference Dinner	Cloef-Atrium of Orscholz

Friday, 22 July 2005

Biomed I:

Chair: Ch. Alexiou

T23	9:00	U. Pison	Iron particles for medical application
T24	9:35	D. Eberbeck, C. Bergemann, E. Wiekhorst, U. Steinhoff, S. Hartwig, L. Trahms	Aggregation behaviour and specific binding of functionalised magnetic nanoparticles in different suspensions quantified by magneto-relaxometry
T25	9:55	M. Răcuciu, D. Creangă, Z. Olteanu	Photosynthesis under the ferrofluid influence
T26	10:15	M. Schwalbe, C. Jörke, K. Höffken, K. Pachmann, J. H. Clement	Depletion of tumor cells from peripheral blood leukocytes with carboxymethyl dex-trane coated magnetic nanoparticles
	10:35	Coffee-Break	

Biomed II:

Chair: K. Stierstadt

T27	11:00	J. F. B. Santana, M. A. G. Soler, S. W. da Silva, M. H. Guedes, Z. G. M. Lacava, P. C. Morais	Dispersion of in vitro biocompatible magnetic fluids in mice's blood: A Raman analysis
T28	11:20	K. Aurich, S. Priepcke, S. Nagel, W. Weitschies	Monitoring of antigen/antibody inter-actions by magneto-optical relaxation measurements
T29	11.40	C. Peleman, D. Caluwier, J. Cocquyt, P. v. Meeren, J. Baert, M. DeCuyper	Synthesis and cellular uptake of differently charged magnetoliposomes
T30	12:00	O. Brunke, C. Alexiou, I. Hilger, S. Odenbach	Magnetic particle distribution in bio-medical applications-examination by X-ray micro-tomography
	12.30	Closing remarks, lunch	

TALKS

SYNTHESIS

PHYSICAL PROPERTIES

TECHNICAL APPLICATIONS

MODELLING

BIOMEDICINE

POSTER

TALKS

Airstable cobalt-, iron- and iron/cobalt nanocolloids and ferrofluids

H. Bönnemann, S. Behrens, N. Matoussevitch

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and
Forschungszentrum Karlsruhe ITC-CPV, D-76021 Karlsruhe

A size-selective synthesis of Co-, Fe/Co-alloy, and Fe-nanoparticles via the decomposition of low valent carbonyl complexes of the respective transition metals in the presence of aluminum-organic compounds will be discussed. In case of the system $Co/Al(oct)_3$ monodisperse particles of 10 ± 1,3 nm are formed. Subsequent “smooth oxidation” leads to the formation of an oxidic “protecting shell” around the metallic core resulting in stable magnetic nanoparticles exhibiting a remarkable long term stability when exposed to normal atmosphere (air and moisture). Experimental evidence for this including XAS, UPS, MIES data and magnetic properties will be presented. This oxidic coat can be modified with a number of surfactants resulting in air stable magnetic fluids. Using appropriate peptization agents magnetic fluids with high magnetic properties (>170 mT) and high volume concentration (e.g. 10Vol Co) were obtained in organic media.

$Fe(CO)_5$ and $Co_2(CO)_8$ are suitable precursors for the aluminum trialkyl mediated synthesis of Fe and Fe/Co nano sized particles, respectively, which may be peptised in organic solvents to give MFs. The particles have a narrow size distribution which can be conserved during the "smooth" oxidation and subsequent peptisation processes. Besides the well-known surfactants as Korantin SH or LP4 natural Cashew Nut Shell Liquid was tested as a novel surfactant.

First results on the peptisation of metallic particles in polar solvents such as alcohol or water for biomedical applications will also be presented.

Preparation of ferrofluids and the effect of organic molecules absorption in the magnetic properties of magnetite nanoparticles

Panagiotis Dallas, Ioannis Rabias, Dimitrios Niarchos

Institute Of Materials Science, NCSR Demokritos, 15310, Agia Paraskevi, Athens, Greece

Magnetic nanoparticles can be considered as a single magnetic Weiss domain and thus they have superparamagnetic behaviour in the presence of a magnetic field. A rather important feature of these materials is their ability to form ferrofluids (magnetic fluids) with the absorption of proper molecules on their surface, providing them with solubility to organic (organosols) [1a] or polar (hydrosols) [1b] solvents. The absorption of the surfactants alters the surface anisotropy which correlates with the effective anisotropy [2].

Magnetite nanoparticles and magnetite capped with oleic acid or oleylamine were prepared from a biphase water / toluene solution in different starting temperatures. From the XRD patterns the mean size was estimated and the conclusion was that the higher the temperature at the beginning of the reaction the smaller the mean size of the nanoparticles, which is attributed to the simultaneous formation of more nucleous [3]. The exact shape and size of the nanoparticles was estimate from the TEM micrographs. Besides the presence of the surfactants stops the crystal growth thus forming smaller nanoparticles and the size was also controlled by the capping agents leading to cubic shaped nanoparticles.

The nature of the bond between the surfactants and the surface is estimated by IR spectroscopy and their percentage by thermogravimetric analysis performed under oxygen flow. From the differences in the antisymmetric (1517 cm^{-1}) and the symmetric vibration modes (1420 cm^{-1}) of the carboxylic anions in the IR spectra [4] we conclude that the oleic acid interacts by a chelating bidentate mode with the surface cations. Besides a small amount of physisorbed oleic acid was observed from the –COOH vibration at 1721 cm^{-1}. In the case of the pthalic acid we see that a rather significant percent is physisorbed and it forms dimers. The absorption of the oleylamine was also estimated by IR spectroscopy.

The nanoparticles capped with surfactants have a smaller coercive field since the coordination of the surfactants reduce the surface anisotropy and prevent the dipole dipole interactions of the nanoparticles. The same magnetite after the absorption of pthalic acid revealed a reduced coercive field. The same result was observed and for the oleic acid and oleylamine capped magnetite.

The need to understand the magnetic properties and the surface chemistry of magnetic nanoparticles is highly important since their various biomedical applications in drug delivery or hyperthermia.

1) a) A.B Bourlinos, A.Simopoulos, D.Petridis, Chem.Mater. 14 899-903 b) Yokoi H., Kantoh T., Bull.Chem.Soc.Jpn. 1993, 66, 1536
2) Andrew Boal, Kanad Das, Mark Gray, Vincent Rotelo, Chem.Mater. 2002, 14, 2628
3) Taeghwan Hyaeon, Chem.Comm. 927-934, 2003
4) Claude Rocchiccioli Deltcheff, Raymonde Franck, Valerie Cabuil, Rene Massart J.Chem.Research (1987) 1209-1210

Precipitated iron oxide particles by cyclic growth

R. Müller, H. Steinmetz, M. Zeisberger, C. Schmidt,
R. Hergt, W. Gawalek

Institut für Physikalische Hochtechnologie Jena

Magnetic fluids may undergo considerable heating if subjected to ac-magnetic fields which may be useful for application in magnetic particle hyperthermia proposed as a tumour therapy. Enhancement of the specific loss power (SLP) of magnetic ac-losses would allow a reduction of the tissue load with magnetic material (magnetite or maghemite) and improve the reliability of therapy. During remagnetisation of magnetic nanoparticles in an ac-field several types of loss processes (hysteresis losses, Néel- or Brown relaxation) may appear which depend strongly on the mean particle size [1] and the size distribution width [2].
To influence and improve the mean size as well as size distribution new approaches in preparation are promising where nucleation and growth of the particles can be influenced independently (what is hardly possible by the usual wet precipitation method) or where a further growing is possible on small given particles without further nucleation. The second way was shown for a non-aqueous system (decomposition of metal-organic compounds) by Philipse et al. at ICMF10, Guaruja, 2004.
First experiments on iron oxide powders by such a cyclic method based on „conventional“ precipitation from Fe-salt solution have shown the feasibility of cyclic growth in an aqueous system. An increasing mean particle size (in the range from about 10 to 30nm) with increasing number of cycles (up to four) is confirmed by XRD. Magnetic parameters of the saturation hysteresis loop and magnetic hysteresis losses calculated from minor loops (by VSM) will be shown. The coercivity increases with the particle size, whereas the hysteresis losses at small applied fields (11kA/m) reveal lower values again if the particles are magnetically too hard.

1) R. Hergt, et al., Recent Res. Devel. Mat. Sci., 3 (2002) 723-742
2) R. Hergt et al., J. Magn. Magn. Mater., 280 (2004) 358-368

An alternative way to ferrofluids

U. Kreibig[1], A. Reinholdt[1], V. Schneider[1], A. Tillmanns[2], T. Weirich[3]

[1]*Physikalisches Institut IA, RWTH Aachen /BRD*
[2]*Physikalisches Institut IIA, RWTH Aachen /BRD*
[3]*Gemeinschaftslabor für Elektronenmikroskopie, RWTH Aachen / BRD*

Conventional Ferrofluids consist of „wet-chemically“ prepared Magnetite or Cobalt colloids, stabilized by some - often unknown - organic stabilizer molecules in the liquid embedding medium. In a short sequence, we shall demonstrate several of our ferromagnetic nanoparticle systems, produced by this way [1].
We proposed recently [2], as a probably simpler and, probably, better defined alternative, a two-step preparation route:
Step 1: „Physical“ particle preparation in our high power, seeding gas supported, laser evaporation/ablation cluster source LUCAS, followed by (almost) adiabatic expansion from the working pressure of $p \leq 3$ atm to $p \approx 1.1$ bar . The required extremely high particle production rate is rendered feasible by applying a 250 W Nd:YAG laser. To reduce or prevent particle agglomeration, the particles can be partly oxidized.
Step 2: Immersion of the particles into a proper embedding liquid, at environmental pressure. The particles have to be stabilized to reduce or prevent agglomeration.

Step 1 was already successfully exercised with LUCAS [3]. We prepared Ni particles (which usually remain unconsidered for ferrofluids because of high preparation difficulties) of about 6 nm mean diameter and deposited them from the free cluster beam behind the adiabatic expansion nozzle onto quartz glass. First we characterized the pure and the partly oxidized Ni-particles by extended TEM, HRTEM and SAED analysis. Second, magnetic properties were measured, with surprising results. The results of both investigations shall be presented in the talk in detail.
Step 2 has not yet been performed, since the UHV chamber construction has to be altered considerably. We, nevertheless want to present the project already now, in order to encourage other research groups to try their own.

1) „Aerosole, Hydrosole und kompakte Clustermaterie: Optische und magnetische Untersuchungen an neuartigen Werkstoffen“ J.Wollgarten, Diploma Work, I. Physikalisches Institut der RWTH-Aachen (1997)
2) DFG-SPP1104 Colloquium Abstracts 2003
3) M. Gartz, C. Keutgen, S. Kuenneke, U. Kreibig *Eur. Phys. J.* D9, 127 (1999)
U. Kreibig in *Handbook of Surfaces and Interfaces of Materials* Vol.3 (ed. H. Nalva), Academic Press, 2 (2001)
A. Stepanov, M. Gartz, G. Bour, A. Reinholdt, U. Kreibig *Vacuum* 67, 223 (2002)

Characterization of magnetic fluids by nuclear magnetic resonance

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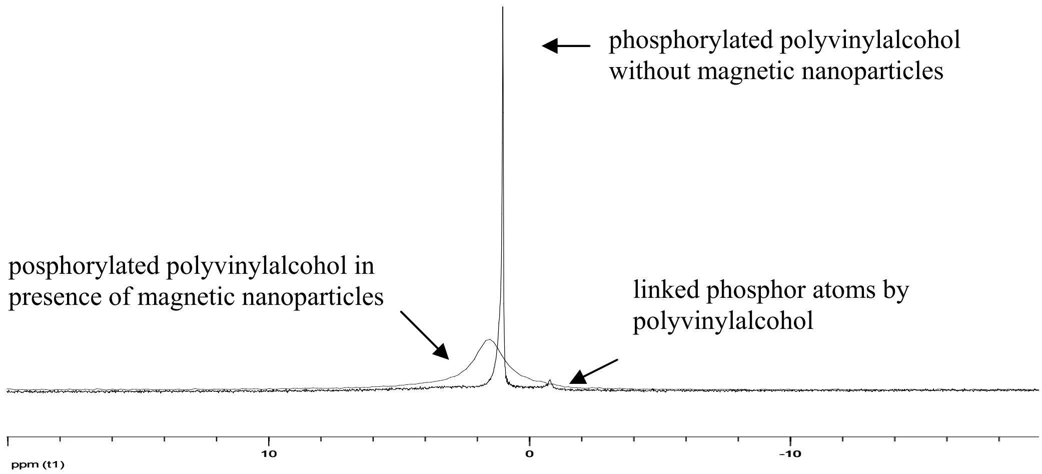
Institute of Chemistry, University of Duisburg-Essen, 47057 Duisburg, Germany

In order to provide information on particle arrangement of magnetic fluids and to analyse self diffusion of the fluid medium, nuclear magnetic resonance experiments on mobile spins in the liquid phase are applied. The obtained resonance frequencies and line shapes depend on a various factors, like particle concentration, particle magnetization, particle agglomeration and sample geometry.
The influence of the self diffusion on the NMR line shape is analysed by numerical simulation of the spectra. This method presents a tool for the observation of changes in the dispersion structure or molecul mobility.
Magnetic fluids consist of nanosized magnetic particles coated with a surfactant dispersed in a fluid medium (Rosenzweig, 1985). In an external magnetic field, the magnetisation of the individual particles of such a fluid leads to a complex superposition of the stray fields. Thereby, a characteristic variation of the field strength with the spatial position is caused (Gonzalez et al., 1998; Terheiden et al., 2003). A numeric algorithm for the simulation of NMR spectra has been developed that accounts for all these influence together with the self diffusion of the spins in the fluid medium.
In the past, various NMR analyses on these confirmed the dependence of the line width and line shape of the NMR signal on the self diffusion of the observed spins in the presence of equally distributed magnetic particles. Generally, decreasing self diffusion leads to a line broadening and/or a line asymmetry (Terheiden et al., 2003). To obtain a line asymmetry it is necessary to decrease the self diffusion of the spins below a certain value in order to minimize motional averaging.
Phosphate has been tied to polyvinylalcohol by phosphorylation and is characterized by ^{31}P-NMR.



Macroscopic dynamics of uniaxial magnetic gels

S. Bohlius[1,2], H. R. Brand[2], H. Pleiner[1]

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Over the last few years ferrogels became a promising class of materials for applications in many fields. Ferrogels are chemically cross-linked polymer networks that are generated using a ferrofluid as a solvent. To produce uniaxial gels, the cross-linking process is performed in an external magnetic field [1,2]. In this situation the nanosized ferromagnetic particles form columns and fibers that are larger than the network mesh size, because the stabilizing coating of the particles is reduced in its efficiency due to the low pH value needed to start the cross-linking process. These chains are fixed in the network, interacting in a way that is so far only partially understood, leading to a frozen-in magnetization which in turn gives rise to several effects in external shear and magnetic fields.
Here we generalize the set of hydrodynamic equations for isotropic ferrogels [3] to uniaxial ferrogels. Ferromagnetic gels are uniaxial, if the frozen-in magnetization denotes the only preferred direction. They show on the one hand similarities to other anisotropic gels, like nematic elastomers, and to isotropic ferrofluids and ferrogels, but the combination of preferred direction, magnetic degree of freedom and elasticity makes them unique and very special.
Prominent features [4] are the relative rotations between the magnetization and the elastic network, which couple dynamically flow, shear, and magnetic reorientation. As a result, shear flow in a plane that contains the frozen-in magnetization induces a rotation of the magnetization, not only within the shear plane, but also out of the shear plane. This behavior is qualitatively different from that of other types of materials. Another outstanding aspect of the hydrodynamics of this material is the difference between the mass current density (mass density times velocity) and the momentum density due to a nonvanishing magnetization vorticity. Unheard of in other classical condensed phases, it is known from some uniaxial quantum fluids, where, however, experiments on this aspect are almost impossible.
Finally we looked at an oscillating external magnetic field that induces not only an oscillation of the magnetization in the direction of the external field, but also oscillating shear strains. The latter are found in planes that contain the frozen-in magnetization and either the external field or the third, perpendicular direction. In addition, the external field also induces a magnetization component perpendicular to both the field and the frozen-in magnetization. The reversible transport coefficient that governs this effect vanishes with the magnetization and is, thus, characteristic for this type of ferromagnetic gel.

1) Z. Varga, J. Feher, G. Filipcsei, and M. Zrinyi, Macromol. Symp. 200, 93 (2003)
2) D. Collin, G.K. Auernhammer, O. Gavat, P. Martinoty, and H.R. Brand, Macromol. Rapid Commun. 24, 737 (2003)
3) E. Jarkova, H. Pleiner, H.-W. Müller, and H.R. Brand, Phys. Rev. E 68, 041706 (2003)
4) S. Bohlius, H.R. Brand, and H. Pleiner, Phys. Rev. E 70, 06411 (2004)

The structural transitions in ferronematics and ferronematic droplets

P. Kopčanský, M. Koneracká, M. Timko, I. Potočová, J. Jadzyn

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Ferronematics are colloidal suspensions of fine magnetic particles in nematic liquid crystals. Their response to external magnetic field sufficiently exceeds that of pure nematics, what encourages investigators in the study of their properties and of the possibilities of their application. The presence of magnetic admixture shifts, compared with pure nematics, the threshold fields of structural transitions invoked by external magnetic or electric fields. The investigation of the changes of these threshold fields is useful for the estimation of the type of anchoring of nematic molecules on magnetic particles surfaces in studied ferronematic. Our previous observations of structural transitions in ferronematics based on thermotropic nematics (8CB, 7CP5BOC, ZLI 1695) confirmed the presence of soft anchoring of nematic molecules on magnetic particles in thermotropic ferronematics [1,2]. This type of anchoring, introduced by Burylov and Raikher [3], permits the boundary condition $n \perp m$ (n-nematic director, m-magnetic moment of magnetic particle), forbidden for the rigid anchoring with $n \| m$, as considered by Brochard and de Gennes [4]. The soft anchoring is characterized by finite value of the surface density of anchoring energy W at the magnetic particle - nematic boundary.

In our present work we study the structural transitions and estimate the type of anchoring and anchoring energy value in MBBA-based ferronematic and in ferronematic droplets, formed in solutions of nematogenic 6CHBT with fine magnetic particles dissolved in phenyl isocyanate. The experiments with ferronematic droplets were inspired by the work of Kedziora et al [5], who observed a droplet phase of nematic crystal dissolved in nonpolar medium in the vicinity of isotropic-nematic transition. We prepared ferronematic droplets, which are magnetically active, the size of the droplets can be easily controlled by the change of temperature and of the molar fraction of dissolved liquid crystal. The magneto-dielectric measurements of various structural transitions in this new system enabled us to estimate the type of anchoring and to find the anchoring energy of nematic molecules on magnetic particles surfaces in droplets.

1) I.Potočová, P.Kopčanský, M.Koneracká, L.Tomčo, M.Timko, J.Jadzyn, G.Czechowski, J.Magn.Magn.Mater. 252, 150 (2002)
2) P.Kopčanský, I.Potočová, M.Koneracká, M.Timko, J.Jadzyn, G.Czechowski, A.G.M.Jansen, phys.stat.sol. (b) 236, 450 (2003)
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Dynamical properties of ferrofluids in zero and applied magnetic fields.

G. Mériguet[1], E. Dubois[1], V. Dupuis[1], B. Farago[2], R. Perzynski[1]

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The ferrofluids studied here are colloidal dispersions of nanocrystals of maghemite in water, stabilized by electrostatic repulsion. Their structure is now well known without field: the interparticle interaction can be continuously tuned by varying the osmotic pressure π, the ionic strength I, and the dipolar interaction through the size of the nanoparticles. These systems exhibit a phase diagram similar to that of molecular systems (gas, liquid, fluid and solid phases exist) [1]. Moreover, their structure under a magnetic field is relatively well understood for volume fractions up to 15% [2].

With these well-defined systems, we firstly explore the dynamical behaviour while increasing the volume fraction Φ from the fluid phase towards the solid phase, which is a glass due to the remaining polydispersity. The rotational dynamic of the nanoparticles, probed by magneto-induced birefringence relaxation experiments [3], shows a spectacular slowing down up to 9 orders of magnitude while increasing Φ from 15 % to 30 %.

Secondly, we explore the translational dynamic for volume fractions up to 15 %, and several interparticle potentials using Neutron Spin Echo (NSE). We perform experiments both without and with a magnetic field. On the micron scale, the collective diffusion under magnetic field is anisotropic [4]. On the scale of the interparticle distance, the only technique available to probe the translational dynamics in such systems is NSE. Under an applied magnetic field, we probe the anisotropy of the diffusion, and link it to the local structure of the dispersions measured with Small Angle Neutron Scattering.

1) F. Cousin, E. Dubois, V. Cabuil, Phys. Rev. E, 68, 0211405, 1-9 (2003).
2) F. Gazeau, E. Dubois, J.C. Bacri, F. Boué, A. Cebers, R. Perzynski, Phys. Rev E, 65, 031403-1 15 (2002).
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Monodisperse magnetic colloids with tunable dipolar interactions

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To investigate the effect of dipolar interactions on static and dynamic properties of magnetic colloids, well-defined particles with a tunable magnetic moment are essential. Several recently developed strategies to synthesize such model colloids will be discussed. Results include surfactant-stabilized magnetite particles from a seeded-growth method, silica cores with a cobalt-ferrite shell, and magnetite cores embedded in latex spheres.

Various features such as reversible dipolar chain formation in zero-field, and the effect of dipolar attractions on particle dynamics can now be studied quantitatively in terms of particle interactions which may be varied from an isotropic repulsion to a dominating dipolar attraction.

Time dependence of field induced ordering processes in ferrofluids studied by small angle neutron scattering

A. Wiedenmann[1], R. P. May[2], C. Dewhurst[2], J. Haug[1], A. Heinemann[1], M. Kammel[1], U. Keiderling[1]

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Small Angle Neutron Scattering (SANS) investigations of surfactant stabilized Cobalt ferrofluids with Co concentrations above 1 vol. % have shown that inter-particle interactions induced by an external magnetic field gives rise to pseudo-crystalline ordering. The Cobalt core-shell particles are arranged in hexagonal planes, with the magnetic moments aligned parallel to the [110] direction which is defined by the external magnetic field [1,2].

Here we report on time-resolved SANS investigations using polarized neutrons which aimed to follow the dynamics of the ordering and disordering processes. For this purpose the ferrofluid samples were placed in a homogenous magnetic field perpendicular to the incoming neutrons which allows the in-plane correlation peaks to be observed. SANS patterns have been measured in very short time slices between 50 ms up to few seconds, triggered by a signal from the magnetic field when the set point of the magnetic field was reached. This stroboscopic technique allowed the time evolution of ordered domains during ordering and relaxation processes to be studied under various conditions. The analysis of the 2D pattern showed i) the reversibility of the processes, ii) the overall dynamics of the underlying slow relaxation processes and iii) the nature of different types of ordered domains.

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Modeling a MEMS ferrofluidic pump

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We present a new micro-ferrofluidic device design that achieves over 1.8 mm/sec average flow speed in a 100 µm high channel. This device has no moving mechanical parts and very low power requirements; it is suitable for fully-integrated microfluidic circuits. The design is based on spatially travelling sinusoidal magnetic fields, which offer the advantage of utilizing both magnetic force and magnetic torque pumping(1) to achieve high flow speeds. Our approach involves solving the magnetization constitutive equation with coupled linear and angular momentum conservation equations(2) to model the behavior of ferrofluids in microchannels in the presence of spatially traveling magnetic fields. An iterative, second-order central-differencing scheme has been employed to calculate the magnetic field, particle spin velocity and overall flow velocity inside the ferrofluid. Flow velocity for ferrofluids depends partly on the spatial period of the traveling wave and the channel dimension; the frequency of the input excitation precisely controls the flow speed. Flow can be reversed by changing the travelling magnetic field direction. Maximum flow velocity is achieved when the product of the excitation wavenumber and height of the ferrofluid channel approaches unity, and the excitation frequency is close to the reciprocal of the relaxation time constant of magnetic particles. Figure 1 shows a measured ferrohydrodynamic pumping peak around 1kHz for oil-based ferrofluid. This relaxation time is determined by the nanoparticles' effective hydrodynamic diameter, which depends in part on the type and size of the chemicals coating their surface.

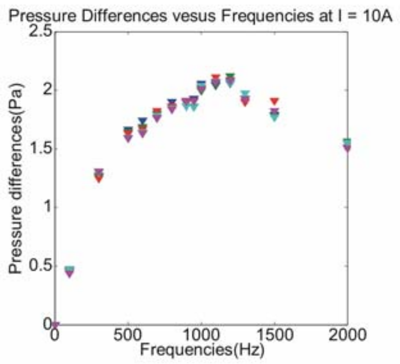

Figure 1. Pumping pressure vesus applied excitation frequencies as measured for the oil-based ferrofluid at 10A current amplitude. The corresponding magnetic field amplitude just inside the windings is about 8000A/m.

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The ALAS project: Development of a new mounting with magnetorheological fluid for the automotive sector

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Driving comfort of an automobile requires, among other things, the damping of large vibration amplitudes of the engine in the range of 5...15 Hz and good vibration isolation for high frequency excitation (small damping, low stiffness). The path for finding a solution was paved by the Freudenberg KG through the innovation of hydraulic mountings in 1977 and led from switchable hydraulic mountings (Freudenberg KG) to active vibration absorbers with electrodynamic actuators (Continental AG) or piezoelectric actuators (Volkswagen).

Within the scope of the project ALAS (acronym for „*Aktives Lagerungssystem mit magnetorheologischer Flüssigkeit (MRF) für den Automobil-Sektor*“), a new mounting with magnetorheological fluid was to be developed and tested. Its stiffness had to be quickly alterable.

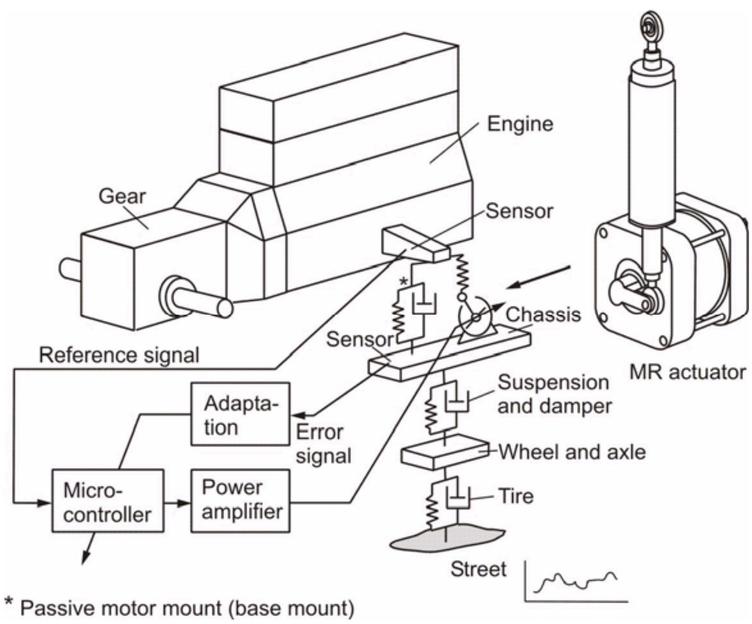

Fig. 1: Representation of the total MR fluid mounting system.

The result of the cooperation of six project partners (Freudenberg KG, BMW AG, FhG ISC, Fuchs Europe, *D*ASS* mbH and LPA) is the active mounting system depicted in Fig. 1. All additional components, such as signal sensing and processing, closed-loop control and the amplifier as well as the mounting system itself, were at first implemented in a special actuator-sensor system at the LPA and successfully examined there before they were installed and tested in an experimental vehicle at a test station provided by our project partner BMW. The intended effect, the damping of the vibration movement within the applicable range, was proven.

LPA will present a detailed description of the actuator-sensor system with the involved MR fluid mounting system and will submit the test results.

The ALAS project was sponsered by the Federal Ministry of Education and Research BMBF in Germany (*Bundesministerium für Bildung und Forschung, Förderkennzeichen 03N3105C*).

Microwave induced bonding using nanoscaled ferrites

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Dispersions of superparamagnetic nanoparticles (<50 nm in diameter) in polymer matrices exhibit high microwave absorption rates due to ferromagnetic resonance [1]. This feature makes them interesting for industrial bonding processes of plastics substrates, since large amounts of energy can be injected into adhesives through the bonding parts without any mechanical contact [2, 3].
A cost efficient process has developed to produce nanoscaled superparamagnetic iron oxides (SPIOs) - doped with further metal ions - to be used as mw absorbing heaters in, e.g., epoxy or polyurethane adhesives, or in hotmelts. Depending on the composition of these nanoferrites their Curie temperature can be tuned to values between 140°C and 200°C. Upon heating the bonding systems to these temperatures, the collective magnetisms of the nanoparticles collapses and they cease to absorb mw energy, which represents an intrinsic overheat protection inside the bonding line. The optimal exploitation of the advanced mw properties of the SPIOs requires a very specific microwave technique, which has also been developed by SusTech and which can be integrated into a large variety of industrial bonding processes. By doping the iron oxides with transition metal ions the SPIOs are optimized with respect to microwave absorption as well as for good dispersibility in various polymer mixtures.
Beyond the chemical and physical principles a brief overview over related bonding techniques and various applications of the mw bonding system will be given.

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Magnetic colloids as model systems for condensed matter

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Colloidal particles in suspension provide – apart from numerous practical applications – versatile model systems for studying basic questions in condensed matter physics. Due to their intrinsic length and time scales in the micrometer and millisecond range they are readily accessible for measurements by means of light scattering or video microscopy, the particle density can easily be varied over orders of magnitude, and also other relevant parameters can be tuned, in particular the interaction energy between the particles, which can range from values much smaller to values much larger than kT.

We report here on experiments with spherical superparamagnetic colloidal particles of well-defined size on the order of a few micrometers, which form 2-dimensional systems at a substrate-water interface. The positions of the particles are recorded by a CCD camera, and the trajectories obtained from these measurements provide all the essential information about the statics and dynamics of such systems. Due to their magnetic properties the particles can in this case be tuned in their interaction quite conveniently by applying a proper magnetic field perpendicular to the sample plane, which gives rise to adjustable (repulsive) magnetic dipole moments of the particles. We have used the "simulations" obtained by means of this "analogue computer" to study problems like crystallization and melting of colloidal ensembles in confined geometry. The interaction between the particles can directly be derived from histograms of the particle positions. It could be shown that phase transitions in 2-dimensional classical "dots" exhibit a rich behavior, for which each particle counts. In addition, we have also carried out dynamics studies in externally applied potential gradients in order to investigate the transport of classical particles through channels or "wires" of various configurations.

Phase diagrams and magnetic susceptibility of ferrofluids

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Ferrofluids are colloidal suspensions of ferromagnetic particles dispersed in a solvent. As long as one is not aiming for a quantitative description of ferrofluids but for general phenomena and trends the so-called Stockmayer model has turned out to be rather useful (see Refs. [1] and [2]). It considers spherical particles interacting with Lennard-Jones potentials plus pointlike permanent dipoles at the center. These fluids are often polydisperse, therefore the binary Stockmayer fluid mixture model gives a reasonable description of bidisperse ferromagnetic fluids and as such provides also a first approximation of actual polydisperse ferrocolloids. For these fluids the magnetic dipole moments of the particles are proportional to the particle volume. Here, we apply a modified mean-field density functional theory to determine the phase behavior of bidisperse ferromagnetic fluids both in the absence of and as a function of an external magnetic field. On the basis of systematic numerical calculations we construct the global phase diagrams of these systems in the three-dimensional thermodynamic space of temperature, pressure, and chemical potential difference of two components. The vapor-liquid – like, isotropic liquid – isotropic liquid, isotropic liquid – ferromagnetic liquid first-order phase transitions are investigated. We discuss how the topology of the phase diagrams changes upon varying the two dipole moments of the two species as well as their sizes. The magnetic susceptibility of the isotropic vapor, isotropic liquid, and ferromagnetic liquid phases is calculated from the corresponding magnetization curves (see Ref. [3]). The behavior of the magnetic susceptibility along and in the vicinity of the phase boundaries is also discussed.

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The dielectric virial expansion and the models of dipolar hard-sphere fluids

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Fluid of dipolar hard spheres of diameter d with embedded point dipoles of magnitude m is the basic model in statistical mechanics of polar liquids. The classical theories by Debye, Onsager and Wertheim determine its dielectric constant ε as a function of single parameter $y=4\pi nm^2/9kT$, where n is the particle number density. At the same time, the results of the models differ significantly and the corresponding expansions ε over y do not coincide starting from terms of order $\sim y^3$. Now it is well-established that ε depends on two parameters – y and $\lambda=m^2/d^3kT$ [1]. According to the modern point of view, the influence of λ on ε is weak until $\lambda<2$ and becomes significant for higher values of λ [2,3]. It is worth-noticing that at $\lambda>3$ the formation of dipolar chains is observed in number of numerical experiments.
Here we develop the virial expansion technique (VET) to determine the dielectric constant of dipolar hard-sphere fluid. Our aim is threefold. First, the VET allows bringing into agreement different approaches and to determine explicitly ε as a function of both dipolar parameters – y and λ. Second, the VET admits to estimate the range of applicability of different analytical models such as the mean-spherical model and the association theory. Finally, number of studies demonstrates that the dielectric constant might be described by simple polynomial dependences on λ and y [4-6]. We assume that retaining in the virial series a few terms only is sufficient for adequate description of the dielectric constant at least at small and intermediate values of λ.
Financial support provided by the INTAS Grant 03-51-6064 is gratefully acknowledged.

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Rheological properties of ferrofluids with drop-like aggregates

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Many experiments demonstrate that bulk drop-like aggregates, consisting of millions of ferroparticles, appear in ferrofluids when temperature is low (however about the room temperature) and/or applied magnetic field is high enough. Appearance of these drops must influence significantly on rheological and other physical properties of these systems.

We present results of theoretical study of the drops influence both on the effective viscosity of ferrofluids and on elastic properties of these systems, filing thin flat gaps. For maximal simplification of analysis, we consider idealistic monodisperse model of real polydisperse ferrofluid. We treat appearance of the drops as specific condensation phase transition in ensemble of the particles. It is well known, from the classical theory of the phase transitions, that at the final stage of the separation, a system, initially methastable, presents two simply connected coexisting phases. However, in the shear flowing system, hydrodynamical viscous forces eliminate volume of growing domains (drops) of the dense phase by a certain maximum volume. That is why the separated system presents an emulsion of the drops, which are elongated along applied magnetic field. We estimated the maximal volume and shape of these drops as well as their influence on the effective viscosity of the ferrofluid. Estimates show that even when volume concentration of these drops is small (less than one per cent), they can provide very strong, more than order of magnitude, increase of the viscosity η of ferrofluid. When applied field H is weak, the viscosity increases as $\delta\eta \sim H^{4/3}$, then tends to a saturation.

If the stable drops are large enough, they overlap the channel of flow and present “bridges” between the channel walls. In this case appearance of quasi-elastic and yield stress effects in ferrofluid are expected. We estimated the shear elastic modulus of the ferrofluids with the bridges as well as of yield stresses of these systems. Estimates show that when the shear strain is high enough, the drop-like bridges break down into two separate drops, which are shorter than the gap. Obviously, this moment can be associated with the change of the elastic to fluid regime of rheological behaviour of ferrofluid. Estimates of the yield stress σ_y demonstrate, that when the field H is weak, $\sigma_y \sim H^{4/3}$, then tends to a saturation. For typical ferrofluids the saturated yield stress is estimated as 0.5-1Pa, depending on concentration of magnetic particles, which are able to form the bulk structures.

Magnetic nanoparticles: a tool for microrheology in living cells

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We have developed new tools to measure the local rheological behaviour of complex fluids, based on rotation of a magnetic probe that has been extended to the measurement of the local viscoelasticity of soft materials on microscopic scales. This new technique of microrheology is based on either the oscillation of chains of micron-sized magnetic particles or on their piloted rotation towards the direction of a permanent magnetic field. Chains oscillations are detected over a 0.001-100Hz frequency range using light scattering although the chains' rotation towards a permanent magnetic field is observed with a microscope allowing a local determination of viscoelastic properties on the scale of the chains of particles. We have demonstrated the accuracy of both assays with a micellar Maxwellian solution and validated theoretical predictions.

The development of the magnetic probe rotational microrheology allowed exploring the cell interior rheological properties.

On one hand, we used the spontaneous endocytosis performed by living cells to internalize 8 nm diameter magnetic nanoparticules which concentrate inside 0.5 µm diameter intracellular vesicles, endosomes, becoming magnetic (5000 nanoparticles per endosome). These magnetic vesicles attract each other and form chains within the cell when submitted to an external magnetic field. We have demonstrated that these chains were valuable tools to probe the intracellular dynamics at very local scales. The viscoelasticity of the chain microenvironment is quantified in terms of a viscosity and a relaxation time by analyzing the rotational dynamics of each tested chain in response to a rotation of the external magnetic field.

We find that the cell interior is a highly heterogeneous structure, with regions where chains are embedded inside a dense viscoelastic matrix and other domains where chains are surrounded by a less rigid viscoelastic material. When one compound of the cell cytoskeleton is disrupted (microfilaments or microtubules), the intracellular viscoelasticity becomes less heterogeneous and more fluid-like, in the sense of both a lower viscosity and a lower relaxation time.

On the other hand, we used the phagocytosis of 2.8 µm diameter magnetic beads by specialized cells (amoebae) to explore the response of the actin cytoskeleton to a controlled shear rate. We measured the response of pairs of magnetic phagosomes to a permanent rotation of the magnetic field, at a given frequency F. The motion of the pair is then dominated by the long time viscous flow behavior of the medium surrounding the pair.

Our major finding is that the measured viscosity decreases with the applied frequency, which is the shear rate suffered by the pairs. Moreover, this shear-thinning behavior follows a simple power law which is directly correlated with the presence and rigidity of actin filaments: the viscosity falls more quickly with shear rate when actin filaments are stabilized although when the actin cytoskeleton is disrupted, the viscosity’s dependence in frequency is much less pronounced. Finally, when a myosin motor is surexpressed inside the cells, both the value of the viscosities and the exponent of the shear-thinning behavior increase.

Thermal ratchet effect in a rotating ferrofluid

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Rotational Brownian motion of a colloidal magnetic particle in a ferrofluid under the influence of an external magnetic field may give rise to a noise induced rotation of the ferrofluid particle due to the rectification of thermal fluctuations[1,2]. Via viscous coupling the associated angular momentum is transferred to the carrier liquid and can be measured as a macroscopic torque.
We generalize the model introduced in [1] to the case of a rotating ferrofluid and study the interplay between the ratchet effect and the non-zero vorticity of the flow. This also allows to describe the influence of the noise-driven rotation on the novel instability discussed in [3].

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Two-dimensional solitons on the surface of magnetic fluids

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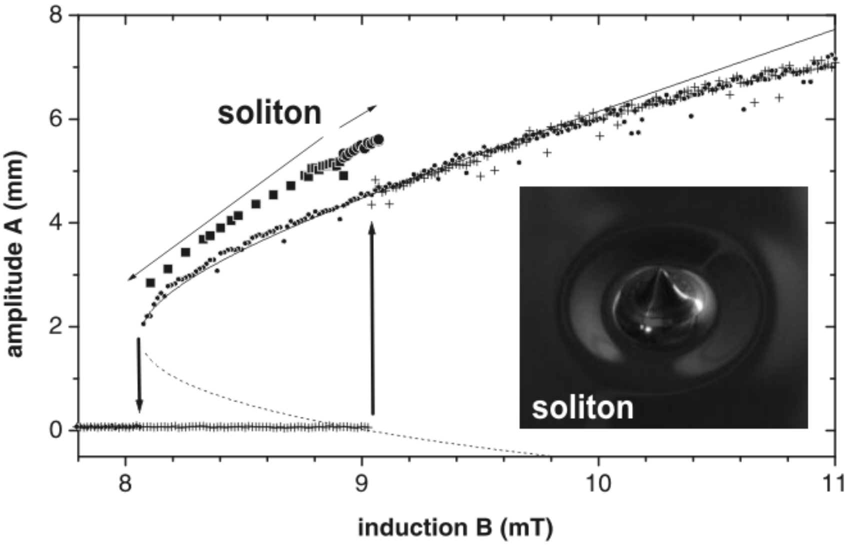
To date, the list of experimentally detectable 2D localized structures was confined mostly to vortices in superfluids, superconductors, and other media on one hand and dissipative solitons in nonequilibrium systems on the other. While the stability of the former is due to their nontrivial topology, the latter come into beeing via the balance of strong dissipation and energy gain. Examples include current filaments in gas discharge systems [1], oscillons in fluids and granular materials [2], breathing spots in chemical reactions [3], and feedback and cavity solitons in optics [4]. Despite some encouraging theoretical insights, the question of whether two-dimensional nontopological solitons can arise in *purely conservative systemes* has remained open.

We report an experimental observation of a strongly localized, stable stationary soliton on the surface of magnetic liquid in the stationary magnetic field. It can be initiated via local application of a magnetic field in the bistable regime of the Rosensweig instability. The figure displays the top-to-bottom amplitude of the hexagonal array of liquid crests, as measured by means of radioscopy [5]. Moreover the range of stability of the soliton and the soliton itself (see inset) are shown. In our contribution we investigate the interaction of solitons and their binding to molecule like structures [6]. Eventually we present a stabilization mechanism, which was discussed before in the context of wave front locking [7].



The experiments have been financially supported by *Deutsche Forschungsgemeinschaft* under grant Ri 1054/1-4.

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Dynamics and magnetoviscosity of ferrofluids: comparison of many-particle simulations and dynamical mean-field theory

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Extensive equilibrium and nonequilibrium simulations on a model ferrofluid are performed in order to study systematically the effect of a magnetic field on dynamical and viscous properties of ferrofluids. A many-particle system is considered where the particles interact with each other by steric and dipolar forces. In addition, the particles are exposed to a magnetic field. Friction and Brownian forces due to the solvent are included into the dynamics [1,2]. The time evolution of the system is solved numerically by Brownian Dynamics simulations. The nonequilibrium simulations are performed in a planar Couette flow. Different volume fractions and dipolar interaction strengths are considered, which correspond to ferrofluids containing ferromagnetic particles of different sizes and in different concentrations.
The field and concentration dependence of the nonequilibrium magnetisation and the viscosity coefficients are found to depend on the strength and orientation of the internal magnetic field [1]. The simplified model of non-interacting magnetic dipoles [3] describes the nonequilibrium magnetisation and the rotational viscosity, but does not account for configurational viscosity contributions and normal stress differences. Improved mean-field models [4] that overcome these limitations show good agreement with the simulation results for weak dipolar interactions where the models should apply. Comparisons to simulation results for various interaction strengths allows to determine the range of validity of the mean-field models.

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Computer simulations of the structure of colloidal ferrofluids

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The structure of a ferrofluid under the influence of an external magnetic field is expected to become anisotropic due to the alignment of the dipoles into the direction of the external field, and subsequently to the formation of particle chains due to the attractive head to tail orientations of the ferrofluid particles. Knowledge about the structure of a colloidal ferrofluid can be inferred from scattering data via the measurement of structure factors. We have used molecular dynamics simulations to investigate the structure of both monodispersed and polydispersed ferrofluids. The results for the isotropic structure factor for monodispersed samples are similar to previous data by Camp and Patey that was obtained using an alternative Monte Carlo simulation technique, but in a different parameter region. Here we look in addition at bidispersed samples and compute the anisotropic structure factor by projecting the q-vector onto the XY and XZ planes separately, when the magnetic field was applied along the z axis. We observe that the XY-plane structure factor as well as the pair distribution functions are quite different from those obtained for the XZ plane. Further, the 2D structure factor patterns are investigated for both monodispersed and bidispersed samples under different conditions. In addition we look at the scaling exponents of structure factors. Our results should be of value to interpret scattering data on ferrofluids obtained under the influence of an external field.

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Iron particles for medical application

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Biologically available iron is a functional constituent of proteins such as haemoglobin and myoglobin and us such essential for living. Technologically produced iron particles with size in the nanometre range have unique physical-chemical properties. Nanostructured iron as naked particle, as magnetic label, or as functionalized surface-core entity has been used with success as contrast agent in a number of magnetic resonance imaging (MRI) applications and for cell tagging *in vitro* and *in vivo*. Successful medical application of iron particles as diagnostic or therapeutic agent depends on their colloidal stability and biocompatibility in physiological environments such as the circulation, tissue or cell culture. In addition, quantitative data demonstrating lack of toxicity and distribution pattern in various compartments of the body are crucial. Because these data are scare, the ultimate utility of nano-sized iron particles for medical application is still not established.

Aggregation behaviour and specific binding of functionalised magnetic nanoparticles in different suspensions quantified by magnetorelaxometry

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Functionalised nanoparticles are intended to bind specifically to targets in biological systems. The binding of magnetic nanoparticles (MNP) can be quantified by magnetorelaxometry (MRX) as has been shown for non-biological (modified latex spheres) and biological (yeast cells) systems [1].

Magnetorelaxometry (MRX) is an integral and sensitive method to quantify the binding and aggregation behaviour of MNP. In contrast to flourescence and radioactive assays, MRX allows to distinguish between bound and unbound MNP without washing steps.

Non-aggregated well separated probes are required for immunological detection tasks. Therefore we have investigated the aggregation of magnetic nanoparticles in a biologically relevant milieu. The quantification of specific binding in practice requires the investigation of the nonspecific binding as well.

We studied the effect of different MNP coatings (polar, nonpolar, biological active) in different suspending media such as aqua dest., phosphate buffer solution (PBS) and serum on the aggregation behaviour of the magnetic probes. We modelled an aggregate as a single MNP with a large hydrodynamic diameter. Assuming a lognormal distribution of the diameters, we estimated the fraction of aggregated MNP as well as mean and width of the aggregate size distribution. Suspending MNP with polar surfaces, the aggregate size is nearly the same as in water, but the amount of aggregates is higher than in water (e.g. 2-3 times higher for Resovist). Among the investigated MNP-Suspensions Resovist (Schering, Germany) and DDM128 (Meito Sangyo, Japan) show relatively weak aggregation of MNP in water as well as in serum.
We set up a biological specific streptavidin-biotin binding system with DDM128 or μ-MACS (Miltenyi, Germany) as magnetic probes. The binding efficiency varies with the type of MNP and is decreased in aged samples: After 1 month the binding efficiency of DDM128 decreases from 90% to about 50%.
The MRX as an integral method is suitable to measure the yield of functionalisation of MNP.

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Photosynthesis under the ferrofluid influence

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The young plantlet growth was studied during the first 12 days of life after germination in the presence of the ferrofluid and daily addition of correspondent ferrofluid concentrations in the culture medium. The ferrophase was obtained by iron oxides precipitation from auto-catalytic reaction of ferrous and ferric salts in alkali medium. The ferrofluid was constituted by coating the small ferrophase particles with citric acid and further dispersion in water. The photosynthesis rate was followed by means of the chlorophyll ratio (chlorophyll a/chlorophyll b), provided by standard spectrophotometric assay (Meyer-Bertenrath method, JASCO type device). The ferrofluid influence upon each chlorophyll type as well as upon the total carotene pigment level was also studied. Complementary data have been extracted from the spectrophotometric assay of the nucleic acid average content. The data provided by the chlorophyll ratio measurement offered the main insight into the photosynthesis complex processes since they revealed the response of the LHC II system (Light Harvesting Complex II) to the external stimuli. The results obtained by us have shown that low ferrofluid concentrations (and correspondingly low ferrophase aliquots) are actually able to influence the plant growth during their early ontogenetic stages, while higher ferrofluid concentrations could have an indirectly effect of some toxin releasing. It seems that the tylakoidal membranes where the assimilatory pigments (mainly chlorophylls and carotenes) are located are sensitive to the physical-chemical stress related to the ferrofluid supply. As for the cell nucleus, the assay of the nucleic acid content, could give some data concerning the capacity of the plant growth under the ferrofluid influence though the correlation with the data representing the LHC II system is not easy to interpret. The statistic analysis was accomplished by considering five repetitions of every measurement in ferrofluid and control samples. Average values of ferrofluid samples and control ones have been compared by means of t-test. Statistic significance was revealed in comparison to the significance threshold of 0.05. We also noticed the differences between the plant species in the frame of the comparative study objective carried out regarding the biological sources.

Depletion of tumor cells from peripheral blood leukocytes with carboxymethyl dextrane coated magnetic nanoparticles

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We could show, that the interaction of magnetic nanoparticles with living cells is cell-type specific, especially comparing epithelial-type tumor cells and human peripheral leukocytes *in vitro*. This observation could be of interest for the aim of eliminating residual tumor cells from peripheral blood of tumor patients.

Leukocytes were prepared by erythrocyte lysis from whole blood samples of patients and healthy volunteers for *in vitro* experiments. The breast cancer cell line MCF-7 was kept under standard cell culture conditions. Cells were inoculated with magnetic core/carboxymethyl-dextran (CMD) nanoparticles with an average magnetite/maghemite core TEM-size varying between 3 and 15nm. Magnetically labeled cells were separated by MACS. The separated cells were analyzed by FACS and Laser Scanning Cytometry. Tumor cells were detected with anti-human epithelium-antigen (HEA)-FITC.

Isolated leukocytes or tumor cells were incubated with magnetic nanoparticles for different time periods (0-20 min) with or without different concentrations of human plasma (0-25%). The interaction of tumor cells with the magnetic nanoparticles led to a more rapid labeling of these cells in contrast to leukocytes. In addition, the interaction was affected only by plasma levels above 5%, whereas the leukocytes showed a dramatic reduction in interaction with the nanoparticles already in the presence of 0.5% plasma. Based on these results, whole blood samples from patients suffering from solid tumors were treated with erythrocyte lysis buffer in order to enrich the leukocyte fraction and to remove the plasma faction. The remaining cells were incubated with magnetic nanoparticles in the presence of none, 1% or 5% plasma for 8 min and separated with our regime. The retained cells and the flow-through cells were quantified. In accordance with the results from healthy volunteers leukocytes from the patients samples showed a considerable reduction in binding of nanoparticles already after addition of 1% plasma. In the presence of 5% plasma less than 20% of the applied cells were separable by MACS. 70% of the tumor cells could be depleted from the leukocyte fraction with a single incubation. In contrast to normal leukocytes the tumor cell fraction increased 2.5-fold in the presence of 5% plasma in comparison to the sample without supplementation of plasma.

We could show, that CMD-coated magnetic nanoparticles could be used to separate tumor cells from human peripheral blood leukocytes. This effect could be increased by the addition of human plasma in a concentration-dependant manner. Our approach is promising with regard to the future elimination of disseminated tumor cells from the peripheral blood of cancer patients.

This work was supported by the DFG-Priority program 1104, grant CL 202/1-1

Dispersion of *in vitro* biocompatible magnetic fluids in mice's blood: a Raman analysis

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Magnetic nanoparticles can be engineered by surface-coating them with special molecules to interact with or bind to a biological structure, thereby providing a controllable means of targeting specific biological sites. More specifically, magnetic nanoparticles surface coated with organic molecules can be dispersed as a stable colloid in physiological medium, named biocompatible magnetic fluids (BMFs), which may be used in several applications [1]. The drawback that mostly concerns the wide use of BMFs in new technologies is the possible adverse effect of foreign particles on the organism. Particularly, the mechanism of interaction between the surface-coated magnetic nanoparticle and blood components, for instance, is still not clearly elucidated. Raman spectroscopy has a huge potential to work as an informative probe for the assessment of hemoglobin conformation changes in blood [2]. Micro-Raman Spectroscopy was used to investigate the effects produced by the dispersion of *in vitro* BMFs in healthy mice's blood. Fresh blood aliquots, taken from each mouse, were mixed with carboxymethyldextran coated (CMDMF), and citrate-coated (CiMF) magnetite-based magnetic fluids, resulting in two series of blood-doped samples, and in three different concentrations from $1x10^{12}$ up to $5x10^{15}$ particles/cm^3. The Raman spectra in the 1200-1700cm^{-1} region show the presence of bands typical of the core-size band region (1500-1650 cm-1) and pyrrole ring stretching region. From the data recorded using blood of ten mice, the ${\nu_{10}b_{1g}}^{oxi}/{\nu_{10}b_{1g}}^{deoxi}$ and ${\nu_{19}a_{2g}}^{oxi}/{\nu_{19}a_{2g}}^{deoxi}$ ratios were measured for the reference blood and for each blood-doped samples. The normalized values for the ${\nu_{19}a_{2g}}^{oxi}/{\nu_{19}a_{2g}}^{deoxi}$ ratio were 2.43 ± 0.37 and 1.82 ± 0.25 for BMFs surface coated with citrate and carboxymethyldextran, respectively. For the ${\nu_{10}b_{1g}}^{oxi}/{\nu_{10}b_{1g}}^{deoxi}$ ratio the values were 2.87 ± 0.59 and 2.50 ± 0.56 for CiMF and CMDMF, respectively. The Raman data will be discussed taking into account the level of the oxygen bounded to the hemoglobin species (oxyhemoglobin and deoxyhemoglobin) and the different types of nanoparticle surface coating.

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Monitoring of antigen/antibody interactions by magneto-optical relaxation measurements

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Commonly used immunoassays for determining antigen/antibody interactions (ELISA, RIA, FIA) are associated with time-consuming washing steps to eliminate unbound material. In contrast, the monitoring of biological binding reactions by magneto-optical relaxation of ferrofluids (MORFF) enables measurements without washing steps [1].

For the first time an antibody/antigen-system with clinical relevance was tested. Therefore, the interactions between eotaxin, a cc-chemokine with diverse functions in allergic reactions [2], and its polyclonal antibody anti-eotaxin were investigated by magneto-optical relaxation measurements.

Magnetic nanoparticles (DDM128, Meito Sangyo; MNP) were initially functionalized with streptavidin. The biotinylated antibody as well as the biotinylated antigen were conjugated with streptavidin-MNP applying the streptavidin-biotin-binding-system. Either the pure antigen or the antigen conjugated MNP were added to the antibody-MNP in different amounts. Binding reactions resulting in an increase of the particles diameter were detected by measuring the relaxation of the optical birefringence signal occurring when a pulsed magnetic field is applied to the ferrofluid.

Experimental relaxation data are evaluated by means of different fitting functions. Assuming a simple exponential decay resulting from a monodisperse size distribution an increasing relaxation time is found with onward reaction time. The addition of eotaxin to anti-eotaxin conjugated MNP in different amounts yields to an enlargement in the particles hydrodynamic diameter from 95 nm to 110 nm within 180 min, whereas preparing eotaxin conjugated MNP and anti-eotaxin-MNP in equal shares also generates an increase in particle size. Starting from 82 nm the aggregated MNP achieve an average diameter of 86 nm within 180 min.

Furthermore, a bi-exponential fitting function is introduced for evaluating the concentrations of bound and unbound particles in the liquid assay. Subsequently, rate constants are calculated from the received concentrations.

In addition, these results are compared with those obtained from surface plasmon resonance biosensor analysis, a standard tool for biomolecular interaction analysis.

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Synthesis and cellular uptake of differently charged magnetoliposomes

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For enhancing contrast in Magnetic Resonance Imaging [MRI], superparamagnetic iron oxide based colloids covered with (synthetic) polymers are widely used. As an interesting alternative, a few years ago we designed so-called magnetoliposomes [MLs].[1] MLs consist of nanometer-sized magnetite cores which are coated with a bilayer of biocompatible, zwitterionic and/or anionic phospholipid molecules.
In the present work we describe the synthesis of MLs whose coating is partly composed of postively charged lipids. In practice, dimyristoylphosphatidylcholine [DMPC] MLs were first prepared[1] and then incubated with DMPC/dioleoyl-tetramethylammonium-propane [DOTAP] (50/50, molar ratio) vesicles. After 24 h the desired DMPC-DOTAP MLs were isolated by high-gradient magnetophoresis. The kinetics of DMPC for DOTAP exchange were monitored by measuring the decrease in phosphate content in the ML coat, the increase in the electrophoretic mobility of the ML structures and also by the time-dependent decrease in DOTAP content in the eluates as deduced from gas-liquid chromatography analyses.
Next, the ML's uptake by 3T3 murine fibroblasts was investigated. For comparison the behavior of zwitterionic DMPC and anionic DMPC-dimyristoylphosphatidylglycerol [DMPG] (90/10, molar ratio) MLs was also checked. Iron uptake was qualitatively assessed by optical microscopy after Prussian Blue staining. In the experimental conditions applied, it appears that the zwitterionic and anionic ML types were moderately taken up, whereas a dense packing was observed with cationic MLs. Further, irrespective of the ML type used, the blue colour was evenly scattered over the entire cytoplasmatic zone and not concentrated at the cell periphery, which favours a real internalization of the iron oxide particles rather than a simple adsorption at the cell surface. More detailed data on the uptake event were gathered from the transmission electron micrographs which clearly showed localization of the dark iron oxide particles within lysosomal structures. Overall, the present work highlights the superior features of cationic MLs to magnetically tag biological cells.
Supported by SBO project nr IWT/30238 to MDC

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Magnetic particle distribution in biomedical applications – examination by X-ray microtomography

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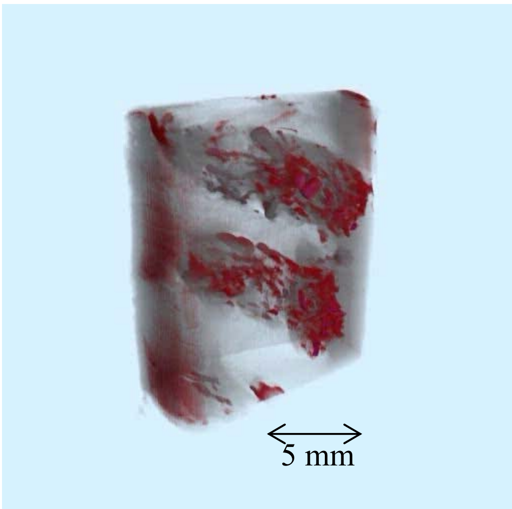
In biomedical application of ferrofluids the resulting distribution of the magnetic nano-particles is a crucial parameter for the effect of the therapeutic approach. The biodis-tribution is usually determined by histological cuts of the investigated specimen, a technique which provides only a very local information about the overall distribution of the magnetic material e.g. in a tumor. Here we will introduce X-ray microtomography as a tool for a 3-dimensional analysis of the distribution of magnetic nanoparticles in biological applications. We will present the basic features of the method as well as first results on mice and rabbit tumor tissue. The figure below shows the capabilities of a common cone-beam tomography setup. At spatial resolution of 80 µm it is e.g. possible to visualise the vessels of a rabbit tumor which have been enriched with magnetic nanoparticles. The position and size of the vessels can be analysed by means of 3D image processing methods [1].
Using a synchrotron as X-ray radiation source the spatial resolution can be significantly enhanced. In this context we additionally will introduce a new experimental setup at HASYLAB/DESY which will allow high spatial resolutions down to 2µm. Furthermore, fast scanning times down to a few seconds will allow serial- as well as in-vivo examinations.



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POSTER

Metallic and bimetallic magnetic fluids. Water based metallic magnetic fluids

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The preparation methods for magnetic fluids (MF) via peptisation of metallic or bimetallic magnetic nanoparticles (Fe, Co, Ni and Fe/Co) in different carrier liquids were investigated by using suitable surfactants [1-3]. The nanoparticles were synthesized by thermolysis of the metal carbonyls in the presence of aluminum alkyls [1,3]. The peptisation of the metallic particles in nonpolar media such as toluene, kerosene, vacuum /and silicon oils was achieved by using oleic- and lauric acid, AOT (Na-dioctylsulfosuccinate), LP-4 (fatty-acid-condensation-polymer), and Korantin SH as the surfactants. The resulting magnetic fluids show high magnetic properties at low metal concentrations.
Water-based metallic magnetic fluids have a high potential for a number of technical and biomedical applications. We have investigated different methods for the preparation of metallic nanopaticles in oder to form water-based MF. Bi- or polylayers around the particles, are formed by transfering the metallic particles from nonpolar media into water by suitable surfactants. Alternativly, the particles surface may be modified. We prepared water-based Co /and Fe MF by applying oleic acid or Korantin SH in combination with Na-oleate or an excess of Korantin SH in oder to form polylayers. In addition, tetramethylammonium hydroxide, sodium dodecylsulfonate, dodecylamin, OP-10 (polyoxiethylen-nonyl-phenyl-ester), BRIJ-35, TWEEN, betaines AMPHOLYT JB 131 polysoaps, L-Cystein, and sugar derivatives can be applaied. The best results were obtained with oleic acid, Korantin SH, AMPHOLYT JB 131 as well as with polysoaps.
Recently, water based MF were also obtained with L-Cystein. This opens the number of potential biomedical applications.

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Ferrit-Ferrofluide auf Basis von perfluorierten Polyethern

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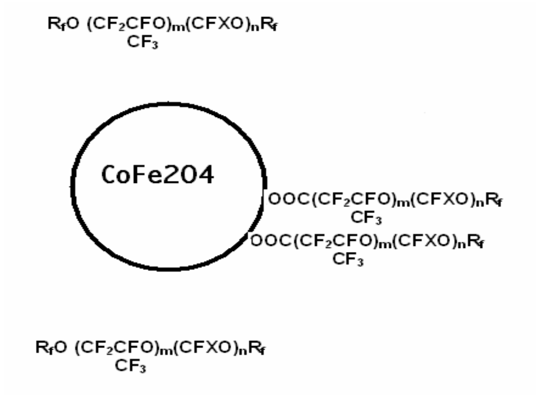
Motivation: Fe_3O_4/γ-Fe_2O_3 und $CoFe_2O_4$ Magnetflüssigkeiten auf Basis von perfluorierten Polyethern (PFPE) wurden entwickelt. Weder die PFPE noch die PFPE-Stabilisatoren enthalten H-Atome. Sie sind weder in Wasser noch in organischen Flüssigkeiten löslich und gegenüber aggressiven Oxidationsmitteln und Mineralsäuren inert. Die $CoFe_2O_4$ PFPE-MF hatten eine relative Magnetisierung in der Größenordnung von 1.2. Diese Eigenschaften sind sowohl für den technischen Einsatz als auch für die Neutronenstreuung von Vorteil.
Ergebnisse: Das entwickelte Herstellverfahren ging von elektrostatisch stabilisierten Ferrithydrosolen aus, wobei die positiv geladenen Ferritteilchen mit PFPE-Monocarbonsäuresalz-Solubilisaten in Kontakt gebracht wurden, was zur Bildung einer PFPE-Moncarbonsäure-Adsorptionsschicht führte, nachgewiesen durch die Bestimmung von Adsorptionsisothermen.
Die erhaltenen Kern-Hüllteilchen ließen sich in technischen PFPE-Lösungsmitteln (Fombline unterschiedlichen Polymerisationsgrades) dispergieren und als PFPE-Magnetflüssigkeiten (s. Abb.1) herstellen, wobei Sättigungspolarisationen bis zu 40mT erreicht wurden.
Physikalisch-chemische Daten zur Herstellung und zu den Eigenschaften der PFPE-MF werden im Detail präsentiert



$R_f = CF_3, C_2F_5, C_3F_7$; $X = F, CF_3$

Abb.1 Schema eines $CoFe_2O_4$-Nanoteilchens mit einer Hülle aus PFPE-Monocarbonsäuresalz, dispergiert in einer PFPE-Flüssigkeit

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Preparation and properties of magnetic silica-ferrite composite colloids

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We describe the preparation and properties of a new type of magnetic composite particles with tunable magnetic interactions as a colloidal model system for dipole-dipole interactions. Magnetic particles of the ferrites maghemite (γ-Fe_2O_4) or cobalt ferrite ($CoFe_2O_4$) can be covalently bound to the surface of colloidal silica spheres via a thiol-functionalized silane coupling agent. When these particles are exposed to a magnetic field, a large magnetic moment is induced in the composite particles due to alignment of individual dipole moments of the ferrite particles. The strength of these induced dipole moments result in magnetic interactions at much larger inter-particle distances than between individual ferrite particles, which makes it practically possible to grow a layer of silica (SiO_2) around the particles without screening the magnetic interactions. Varying the thickness of the silica layer and dispersing the particles in in a refractive index-matching solvent mixture makes it possible to tune the particle interactions. Results will be presented from optical- and electron microscopy studies of silica coated composite particles.

Preparation of water based dispersions of magnetic iron oxide nanoparticles in the mean diameter range of 15 to 30 nm

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With decreasing particle size magnetic particles undergo a transition from stable ferromagnetic to superparamagnetic behaviour. While typically superparamagnetic colloids – i.e. ferrofluids with mean magnetic core diameter in the order of 10 nm – were intensively investigated in the past there are rarely investigations on the transition range to stable ferromagnetic properties. On the other hand, magnetic behaviour of larger multidomain particles in a size range above about 50 nm is well documented and understood. Though the transition range should be rather narrow for so-called monodisperse suspensions, in reality one has size distributions which lead to remarkably broadening. In addition, with increasing core diameter there are increasing difficulties with the colloidal stability of the dispersions.
While for typical ferrofluids several preparation routes are well established, there are only few attempts known in the diameter range 15 to 30 nm. In the present paper a modified method for the synthesis of particles based on a slow oxidation of Fe(OH)2 hydrosol in this diameter range is described. The water based dispersions were stabilised by covering these particles with a coating of citrate or carboxyl dextran.
The dependence of magnetic properties on the morphologic properties was investigated. Mean particle size and the morphology were investigated by XRD and TEM. The size distribution was determined by TEM and PCS. Magnetic characterisation was done by vibrating sample magnetometry. The dependence of hysteresis losses on field amplitude is determined from measured minor loops. For comparison, specific loss power is determined by calorimetric measurements.

Size fractionation in a phase-separated colloidal fluid

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Phase separation of a polydisperse colloidal dispersion implies size fractionation.[1] An application of this effect is given by size-selective purification procedures associated with the colloidal synthesis of so-called monodisperse nanoparticles, which self-assemble into superlattices.[2-4] We used electron microscopy to determine detailed particle size distributions of coexisting colloidal fluid phases containing highly polydisperse iron oxide nanoparticles with a log-normal distribution. Analysis of about 10000 particles per phase yields the first five statistical moments of the distributions.[5] Within experimental error, the interdependence of the statistical moments is in quantitative agreement with the "universal law of fractionation" proposed by Evans, Fairhurst, and Poon[6-8], even though the theory was derived in the limit of slight polydispersity. This suggests that the theory is applicable to a wide range of colloidal systems, since colloidal synthesis often automatically leads to particles with a broad log-normal distribution, comparable to the system studied here.

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Monodisperse ellipsoidal core-shell colloids with adjustable aspect ratio

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We report on the preparation of monodisperse hematite-silica core-shell ellipsoids with aspect ratio's ranging from spindles to nearly spheres. Hematite cores were prepared following the procedure of Ocaña at al. [1] The cores were grafted with poly-(vinylpyrrolidone) (PVP)[2] in order to improve the stability of the system during the silica coating, which is obtained by addition of tetraethoxysilane (TES) in a seeded growth process. The use of tetramethylammonium hydroxide instead of ammonia, as base for the hydrolysis and polymerization of TES, facilitates continuous growth, without problems due to ammonia evaporation. The low polydispersity and the high yield, compared to other preparative methods, make this process suitable for preparing colloidal systems which can be used, for instance, to study random packing and glasses of ellipsoids and (when labelled) rotational diffusion of ellipsoids as function of aspect ratio.

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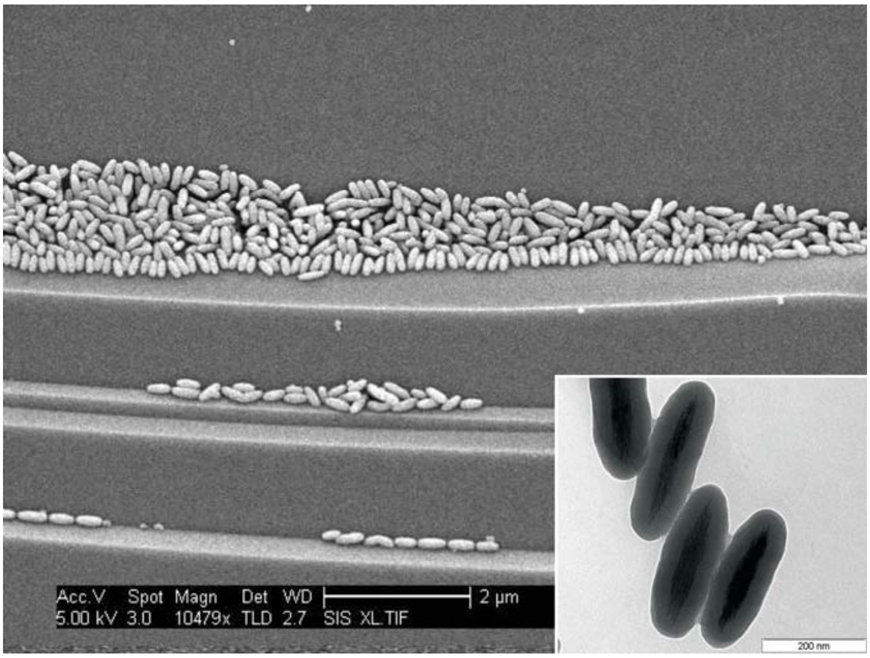

Figure. Hematite ellipsoids coated with a 37 nm thick silica shell.

Erzeugung und Eigenschaften ferromagnetischer nanoskaliger Metallpulver hergestellt durch Laserverdampfung

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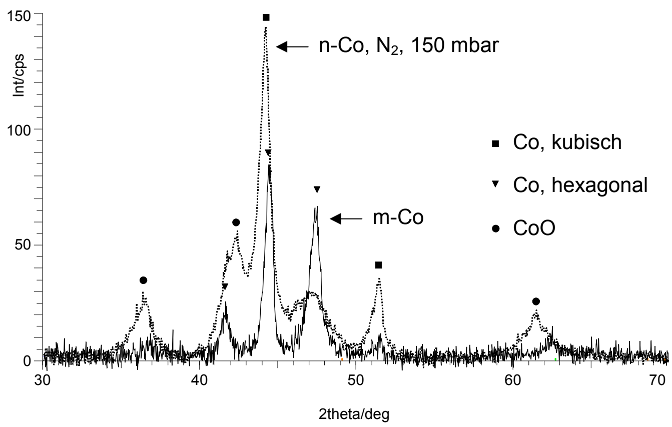
Die Herstellung ferromagnetischer nanoskaliger Metallpulver für Magnetofluide durch die Verdampfung von entsprechenden Metallen mittels eines gepulsten Nd:YAG-Lasers stellt eine interessante Alternative zu den bewährten chemischen Methoden dar. Sie bietet unter anderem den Vorteil, dass die chemische und strukturelle Zusammensetzung der Pulverteilchen durch die Zusammensetzung des Targets und die Wahl der Verdampfungsbedingungen beeinflusst werden können.
Aufbauend auf den Ergebnissen und Erfahrungen, die bei der Herstellung magnetischer Nanopulver durch Verdampfung von eisenreichen FeSi- bzw. FeCo-Targets [1] gesammelt werden konnten, liegt der Schwerpunkt der aktuellen Untersuchungen auf der Erzeugung und Untersuchung nanoskaliger Pulver durch die Laserverdampfung reiner Co- bzw. Fe-Targets sowie eisenreicher FeCr- und FeAl-Targets. Für unterschiedliche Verdampfungsbedingungen wie Art und Druck des Aggregationsgases wurden durch Röntgendiffraktometrie die Phasenzusammensetzung der entstandenen nanoskaligen Pulver bestimmt. So konnte z.B. festgestellt werden, dass es bei der Verdampfung der Co-Targets zu einer Phasenumwandlung des hexagonal strukturierten mikroskaligen Ausgangspulvers kommt. Im Nanopulver wird als Hauptbestandteil die kubische Phase des Co neben Anteilen von CoO nachgewiesen. Überprüft wird dieses Ergebnis durch XAS-Messungen.



Die Sättigungsmagnetisierungen einiger hergestellter Pulver wurden durch VSM-Messungen untersucht.

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Magnetic Polymer Brushes: Towards tailor-made stabilization of magnetic fluids by surface-initiated polymerization

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Like common colloidal dispersions, ferrofluids need to be stabilized in order to prevent phase separation by sedimentation and agglomeration of the magnetic particles. Known procedures include electrostatic and steric mechanisms by adjustment of surface charge, ionic strength and/or concentration of surface active substances like tensides or block copolymers. Many systems are based on dynamic processes and show insufficient stability upon dilution or concentration.
The preparation of magnetic particle-cored polymeric brushes by surface-initiated polymerization is a new approach to stabilize the magnetic particles by a tailor-made polymeric shell. We report the synthesis of dilution-stable ferrofluids by polymerizing from a surface-immobilized initiator, using suitable polymerization mechanisms like ring-opening polymerization (ROP)[1] and atom transfer radical polymerization (ATRP, see fig. 1). This grafting-from approach results in particles carrying end-attached polymer arms with a high grafting density.

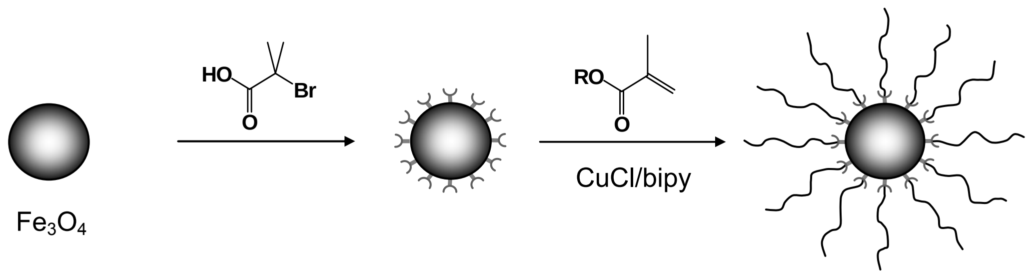

Fig. 1: Synthesis of magnetic polymer brushes by surface-initiated ATRP of (2-methyoxyethyl)methacrylate (R = O CH$_3$) on magnetite (Fe_3O_4) nanoparticles.

The dispensability of the hybrid particles is determined mainly by the solution properties of the polymer arms and can be adjusted to fit the dispersion medium by the choice of (co)monomer and molecular weight. This leads to a variety of possible carrier fluids. We implemented magnetic fluids with different polymeric shell materials based on the carriers toluene, chloroform and dimethylformamide.
Thermoreversible ferrofluids can be designed by the combination of a polymer/medium system exhibiting critical solution behavior. The new materials can be useful for the design of model ferrofluids with adjustable hydrodynamic radius and for the development of magnetically activated delivery systems.

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Synthesis of rodlike magnetic particles for application in ferrogels with magnetic anisotropy

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Gels are cross-linked polymer chains which are swelled by a fluid. If the gel is filled by a ferrofluid it will become sensitive to an external magnetic field and is called a ferrogel [1]. It has been demonstrated that such ferrogels exhibit a significant change in shape under the influence of an inhomogeneous magnetic field [1,2]. Under the influence of a homogeneous magnetic field, a small change in the gel's elastic behaviour has been observed, which was attributed to the formation of particle chains [3]. The magnetic properties of these chains are controlled by the uniaxial shape anisotropy. The objective of the present study is to tailor the morphology of magnetic nanoparticles toward a high aspect ratio in order to enhance the effect of shape/magnetic anisotropy. The synthesis of such particles which is used in the present study is separated into two steps [4]. In the first step, β-FeOOH particles with an ellipsoidal shape are precipitated from an aqueous solution of $FeCl_3$ by forced hydrolysis. In the second step, the β-FeOOH particles are dehydrated to γ-Fe_2O_3 by a heat treatment. In this study, we examined the influence of hydrolysis time on the particle size, characterized by transmission electron microscopy. The conversion from β-FeOOH to γ-Fe_2O_3 is accompanied by an increase in the saturation magnetization. However, TEM studies revealed that the needle-shaped FeOOH particles disintegrated during this conversion into very fine nanoparticles. This destruction of the shape anisotropic particles was prevented by coating the FeOOH–aggregates with a silica layer, based on the Stöber-synthesis [4,5]. Preliminary results on the magnetic properties of ferrogels, filled with ellipsoidal particles will be presented.

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Preparation and characterization of some water ferrofluids stabilized with citric acid

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Water ferrofluids, generally intended for bio-medical applications, may have various coating molecules that make them stable and compatible with the biological liquids. Ferrofluids containing iron oxide particles have been prepared by a controlled co-precipitation method, using citric acid as stabilizer. The ferrophase from the ferrofluids prepared by us was obtained by chemical precipitation from ferric ($FeCl_3$) and ferrous salts ($FeSO_4$ or $FeCl_2$) in alkali medium (ammonia hydroxide). The temperature was kept around 80 ^{0}C in all the five situations (three samples prepared using the ferrous sulphate and two samples prepared using only iron chlorides). Citric acid that has been used to stabilize the ferrophase particles (about 5% volume), represented about 10% volume. The differences between the preparation protocol stages were given by the ferrophase washing, namely the total amount of washing water and the stirring time. Physical tests have been performed in order to reveal their microstructural and rheological features. Transmission electron microscopy was the main investigation method for the assessing of the ferrophase particle size. The dimensional distribution of the ferrophase physical diameter was comparatively presented using the box-plot statistical method. Infrared absorption spectra have been recorded aiming to get some information on the ferrofluid composition. The ferrofluid density (picnometric method), viscosity (capillary method) and surface tension (stalagmometric method) have been measured using standard methods. The pH measurements were carried out with universal indicative paper (Merck). The ferrofluid macroscopic homogeneity was obvious by the visual inspection of the sample vials when submitted to the action of a relatively weak magnetic field (provided by the magnetic stirrer).

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Phase transitions in an Ising model with impurities

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Using the power of actual algebraic processors and the method presented in a series of earlier papers we get the exact partition function L(T) in the Generalized Two Dimensional Ising Model[1,2]. We analyze an Ising model with spins arranged in unitary cells with twenty four different parameters of interaction.
First studying the locus of phase transitions we obtain magnetic regions. In order to determine the magnetic states that correspond to the different magnetic zones that are found, we calculate the twenty four correlation functions between spins. We show that some magnetic configurations can exist in finite domains and others that are privative of infinite domains.

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Pattern formation on viscous ferrofluids

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This work deals with surface instabilities on viscous ferrofluids. In the course of the experiments, the existence of the anomalous dispersion branch of parametrically driven surface waves has been demonstrated for the first time. The experimental data are compared to numerical results. The algorithm, implemented to perform a linear stability analysis, is introduced and explained. Further experiments investigate the pattern formation in the presence of a homogenous magnetic field. Its strength is varied from values that are below the threshold for the Rosensweig instability to values well above this threshold. The patterns, resulting from the competition of Faraday and Rosensweig modes has not been examined up to now. Within this parameter region the existence of a harmonic response with respect to time has been shown for filling levels, that disallow this harmonic response for non-magnetizable fluids. The patterns, appearing in this region, have been seen in two- or three frequency experiments only for non-magnetizable fluids, are described within the framework of amplitude equations, as far as possible in this strong nonlinear realm.

Dynamic versus arrested dipolar structures

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Recently, cryogenic transmission electron microscopy was used to demonstrate the presence of chain-like dipolar structures in two types of ferrofluid, with metallic iron nanoparticles [1] and with large magnetite nanoparticles [2]. Here, the question that will be addressed is whether the observed structures result from a dynamic equilibrium between chains of different lengths or whether, to the contrary, the structures are arrested. The answer is given by frequency-dependent measurements of the complex magnetic susceptibility[3,4]. It will be shown that such measurements as a function of concentration can distinguish unambiguously between dynamic and arrested dipolar structures.

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Relaxationsverhalten $Co_xFe_{3-x}O_4$ basierender Ferrofluide

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Kobaltferrit ist ein hartmagnetisches Material, und auf Kobaltferrit basierte Ferrofluide zeigen Brownsches Relaxationsverhalten[1)]. Im Gegensatz dazu ist Magnetit ein weichmagnetisches Material und relaxiert bei Teilchendurch-messern bis 15 nm bevorzugt nach dem Néelschen Mechanismus.
Eine Serie von $Co_xFe_{3-x}O_4$ Mischferriten (mit x = 0, 0.1,..., 1) wurde mittels der Präzipitationsmethode hergestellt und anschließend mit Oleoylsarcosinsäure in verschiedenen Lösemitteln stabilisiert. Durch Messen und Berechnen der komplexen Suszeptibilität konnte bereits bei einem Kobaltgehalt von x = 0.1 überwiegend Brownsches Verhalten festgestellt werden.
Die komplexe magnetische Suezeptiblität mehrerer Ferrofluide wurde konzentrationsabhängig gemessen. Die Anfangssuszeptibilität nimmt, wie erwartet, mit kleiner werdender Konzentration ab. Das Maximum des Verlustpeakes verschiebt sich zu niedrigeren Frequenzen mit steigender Konzentration.
Ebenso beobachteten wir den Einfluss eines statischen Magnetfeldes auf die magnetische Suszeptibilität. Dabei nimmt die Anfangssuszeptibilität mit wachsendem Magnetfeld ab (Abb. 1 links). Die Lage des Verlustpeakes verschiebt sich deutlich zu höheren Frequenzen mit wachsendem Feld, d.h. es zeigt sich eine scheinbare Abnahme der Teilchengröße. Die relative Abnahme der Anfangssuszeptibilität zeigten gute quantitative Übereinstimmungen mit Magnetisierungsmessungen (Abb. 1 rechts).

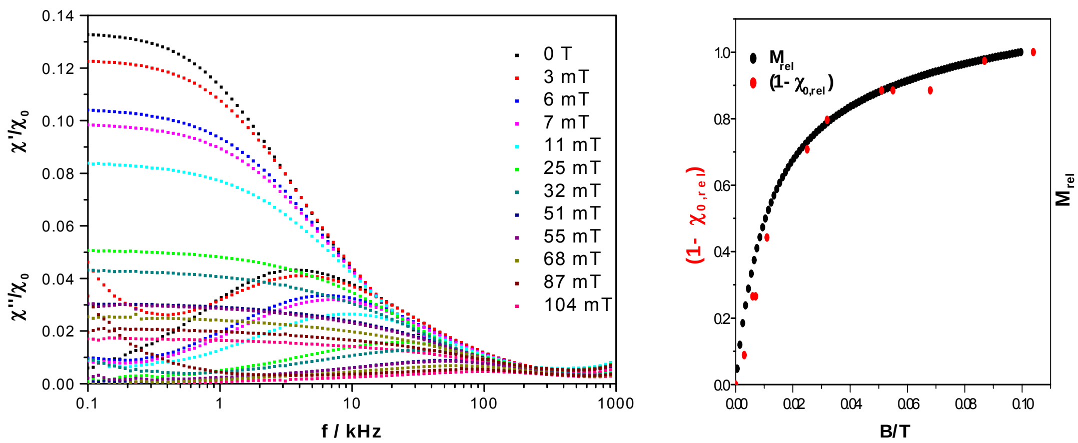

Abb. 1 Links: Komplexe magnetische Suszeptibilität von Kobaltferrit mit Korantin in Cyclohexan als Funktion eines äußeren statischen Magnetfeldes. Rechts: Relative Abnahme der Anfangssuszeptibilität in Abhängigkeit des magnetisches Feldes verglichen mit der Magnetisierungskurve.

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Magnetoviscoelasticity modifies pattern formation in ferrofluids

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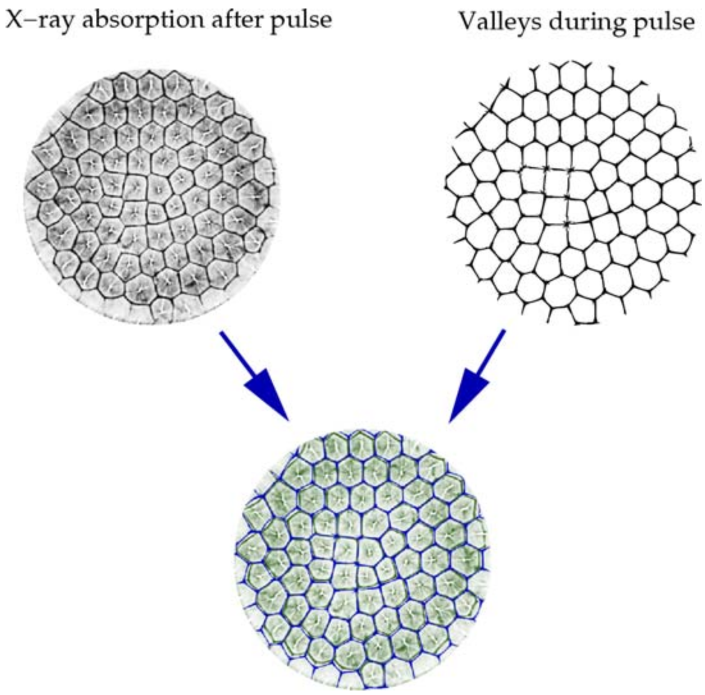

An inverse ferrofluid is a suspension of non-magnetic particles in a conventional ferrofluid, and has been first investigated by Skjeltorp [1]. The particles can be treated as magnetic "holes", the magnetic moments of which point into the opposite direction of the external magnetic field. The Rosensweig instability of an inverse ferrofluid is investigated and compared to the pattern formation of the base fluid. Due to chain formation and magneto-visco-elastic effects [2] the qualitative behaviour changes drastically, in that hysteresis is introduced, which is not present in the base fluid. By the use of X-rays, complete surface profiles can be recorded [3] and phase separation of the inverse ferrofluid in gravitational and magnetic gradient fields are observed. In combination with the Rosensweig instability this gives rise to a memory effect. Eventually, the voronoi tesselation is utilized as a tool to visualize ordering phenomena of the Rosensweig pattern.

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Driving ferrofluidic patterns by alternating magnetic fields

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A surface of magnetic fluid (MF) subjected to a normal field is becoming unstable when a certain threshold B_c of the magnetic induction is surpassed [1]. Previously we have investigated the wavenumber of maximal growth of this instability [2], and their oscillatory decay [3]. The more general case is a field, which is tilted towards the normal of the MF-surface. This has important consequences for the stability of the liquid layer. Now the well known hexagonal lattice of Rosensweig cusps, is replaced by liquid ridges (see Figure), which appear as primary pattern [4,5,6]. The ridges are oriented parallel to the tangential component of the magnetic field, i.e. the tangential component stabilizes the fluid surface in this direction.

In our contribution we drive these liquid ridges by alternating magnetic fields. We investigate the stability of the pattern under variation of driving amplitude and frequency.

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Ferrofluids dispersed in paramagnetic metallomesogens: magnetic and optic properties

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Paramagnetic metallomesogens are liquid crystals containing a paramagnetic metal ion mostly inside the central complex unit. Those compounds, in particular rare earth metallomesogens, show very interesting magnetic properties namely high magnetic anisotropy. Interestingly, the magnetic anisotropy can be stabilized in a supercooled glassy state at room temperature.
By doping metallomesogens with ferrofluids the magnetic anisotropy can be increased dramatically.
In this report we are presenting results of magnetic measurements in DC and AC magnetic field within the liquid crystalline temperature range and at lower temperature down to 4 K. Furthermore, results of magnetization measurement will be discussed.
Optical CD measurements under applying an external magnetic field were done. The MCD spectra are depending on concentration, wavelength and temperature.

SANS investigation of inter-particle correlations in concentrated Fe_3O_4 ferrofluids

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Recent observation of local hexagonal structures in some Co-based concentrated ferrofluids (FF) [1] stimulates similar small angle scattering experiments of polarised neutrons (SANSPOL) on magnetite based FF. Since the saturation magnetisation of a single Fe_3O_4 particle is lower than for Co one can increase the magnetic coupling only by using very small nonmagnetic shells and high magnetic particle concentrations. For this requirements samples of Fe_3O_4-FF dissolved in petroleum or water with very high concentrations up to 30 vol. % have been synthesised using, respectively laurin-acid (L) or laurin-acid-malipal (LM) as surfactants. From the SANSPOL measurements on samples diluted with deuterated solvents we extracted the parameters of a core-shell model of the particles. The shell thickness corresponds to a monolayer of L-surfactants in petroleum and to a bi-layer of LM in water. These parameters were used to model the form-factor contribution of the scattering in the concentrated samples (see fig. 1). For the concentrated samples we observe inter-particle correlations induced by an external magnetic field which lead clearly to non-isotropic SANS scattering pattern. The observed anisotropic structure factor has a lower symmetry than the hexagonal one observed in Co- FF [1] but can not be explained by a simple isotropic dense-packing of the particles .

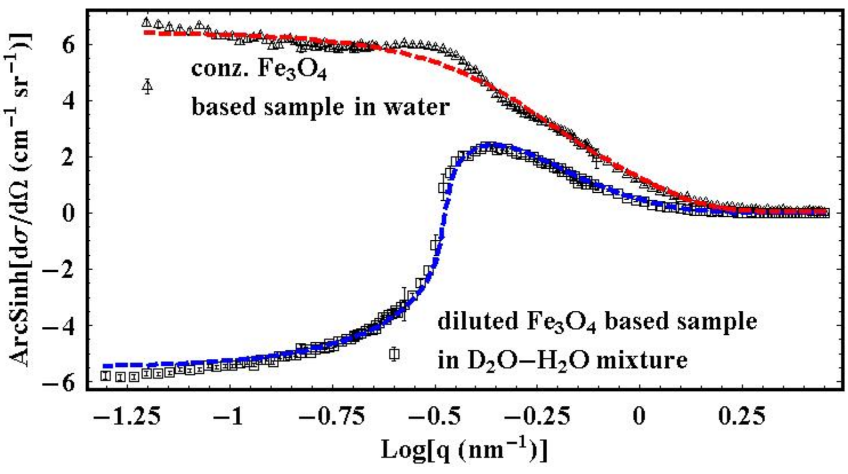

Fig. 1 SANSPOL intensity differences (I^+- I^-) for a Fe_3O_4 FF. Single particle information was extracted from the diluted case (solid line: fit to core-shell model) and used to model the form factor contributions in the high concentrated sample (dashed line). The deviation from the observed curve (triangles) results from a field induced structure factor.

Work supported by DFG project Wi 1151/2-2 (SPP1104)

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Pattern selection in colloidal solutions in the Rayleigh-Bénard system

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When investigating the convection of ferrofluids between two parallel plates heated from below (Rayleigh-Bénard convection) the binary nature of these fluids has to be taken into account[1)] to understand the bifurcation behaviour. Being colloidal liquids, ferrofluids are different from the molecular mixtures studied normally. In comparison they exhibit a much slower concentration diffusion and a much stronger negative or positive Soret effect; the relevant parameters differ by about 3 orders of magnitude.[2)]

Using the Galerkin method we calculated the fixed points of several 2D and 3D convection structures known to appear in molecular liquid mixtures within a multi mode model and tested their stability. Our main results are that for a positive Soret effect that was found for sterically stabilised particles square patterns are the stable form of convection at small temperature differences and roll patterns at higher Rayleigh numbers. These structures were already found in experiments.[3)] In between, at about the critical Rayleigh number R_c of the pure fluid mediating crossroll patterns can be found.

For the case of a negative Soret effect that is relevant for ionically stabilised ferrofluids the backwards bifurcating stationary roll solutions known from molecular mixtures are present with a saddle at very small convection amplitudes. Travelling waves however do not exist at moderate temperature differences for fluid parameters typical for ferrofluids.

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Evidence for oxide coating in air stable Co-ferrofluids

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We investigated a new series of air stable cobalt ferrofluid samples prepared by thermolysis of $Co_2(CO)_8$ in the presence of $Al(C_2H_5)_3$ and subsequent smooth oxidation [1]. EXAFS and other studies [2] have suggested the presence of Co-O groups. The Co particles were coated by technical surfactants Koranthin SH. Earlier Small Angle Neutron Scattering experiments on ferrofluids prepared without this smooth oxidation step showed a complex composition [3].

The unknown ferrofluid structure and size distribution as well as the magnetic nanostructure are subject of evaluation. After the smooth oxidation we performed Small Angle Neutron Scattering studies using polarised neutrons (SANSPOL) [4] with a wavelength λ=0.6nm. A horizontal magnetic field up to 1.0T and perpendicular to the incoming neutrons was applied on the samples. The advantage of the usage of polarised neutrons is the ability to sensitively distinguish between the chemical and magnetic nanostructure. This allowed an exact calculation of the structural parameters in the fitting procedure.

Different samples in toluene and kerosene were investigated. All samples were solved in none-deuterated solvents. In such a case the Korantin SH has no scattering contrast and only other compounds of the ferrofluid are visible. The analysis of the scattering intensities perpendicular to the applied magnetic field gives evidences for a nonmagnetic layer on the surface of the cobalt-core. The layer's thickness is independent of the solvent used ranging between 1.2 nm and 1.5 nm. The scattering length density of the nonmagnetic layer could be precisely extracted from this core-shell model. It was found to correspond closely to the high value expected for cobalt-oxide. This results shows unambiguously that smooth oxidation consist on the formation of an non-magnetic oxide layer on the Co-core protecting the particles from further oxidation.

Work supported by DFG project Wi 1151/2-2 in the frame of priority program SPP1104.

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Zero-field dipolar structure formation in colloidal dispersions of synthetic magnetite

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We present the first direct observation of zero-field dipolar chain formation in colloidal dispersions of synthetic magnetite (Fe_3O_4) with a single magnetic domain (Figure 1). Cryogenic transmission electron microscopy images directly illustrate that the length of the dipolar structures depends on the size --and therefore on the magnetic dipole moment-- of the particles. An important feature of our magnetite fluid is the possibility to study the dipolar structures quantitatively as a function of particle size on a single-particle level by analysis of the cryo-TEM images. The analysis yields radial distribution functions, the nearest-neighbor probability, and the chain length distribution. The obtained chain length distribution is in line with a dynamic equilibrium between chains of different lengths and an interaction parameter can be extracted. This parameter agrees with the theoretical value that can be calculated from the known dipole moment of the particles. Concentration-dependent dynamic magnetic susceptibility measurements confirm that the dipolar chain formation is a dynamic-equilibrium process. The presented chemical system[1] enables us to test theories[2] for dipolar fluids that predict anisotropic aggregation in zero field if the dipolar interactions are strong enough compared to thermal energy.

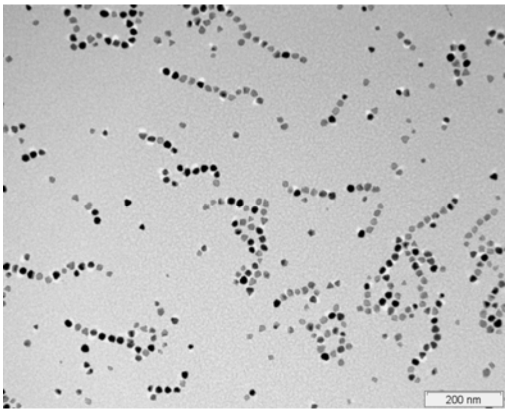

Figure 1. Typical in situ cryo-TEM images (zero field) of vitrified magnetite dispersions.

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Dynamic nature of dipolar chains in colloidal magnetite dispersions demonstrated using magnetic susceptibility measurements

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Magnetite (Fe_3O_4) forms the basis of most dispersions studied in the field of magnetic fluids and magnetic colloids. When the magnetic interactions between the particles are sufficiently strong compared to thermal energy, theory predicts anisotropic aggregation in zero field [1]. Recently we have shown that synthetic magnetite colloids can be prepared sufficiently large to exhibit rich dipolar structure formation in zero field.[2] Here, we demonstrate the dynamic nature of the structures using frequency-dependent measurements of the magnetic susceptibility.

Dynamic susceptibility spectra of dispersions containing 16 nm magnetite colloids reveal that the magnetic moments are free to rotate inside the particles, which is called N/'eel relaxation. The rotation of the magnetic moments occurs at a characteristic frequency above 100 kHz. Spectra of dispersions containing larger magnetite particles (21 nm) show a characteristic frequency of less then 100 Hz, which is well below the frequency corresponding to the rotational diffusion of single particles (a few kHz). The low characteristic frequency is due to the Brownian rotation of large dipolar structures in which the magnetic moments are fixed. Concentration-dependent measurements indicate a dynamic equilibrium between chain-like dipolar structures of different lengths, because the structures break up upon dilution.

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Comparing measurements of the growth rate of the Rosensweig instability with theoretical predictions

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The surface of magnetic fluids subjected to a normal magnetic field is becoming unstable when a certain threshold of the magnetic induction is surpassed and the initially flat surface exhibits a stationary array of peaks (Rosensweig or normal field instability). Up to now there exists only a thjeoretical prediction of the behaviour of the growth rate of this surface instability [1]. Therefore we have performed time resolved measurements of the amplitude and their relaxation with the help of a linear array of 32 Hall sensors which are placed directly under the ferrofluid. Sensors situated under a ridge (trough) detect higher (lower) values of the local magnetic induction, respectively. By calibrating the magnetic signal with normed reliefs of the ferrofluid surface the height of the liquid surface can be measured as well. With a time resolution of the sensors of about a half millisecond the maximal growth rate of the modes of the Rosensweig instability can be measured and are compared with the theoretical predictions.

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Measuring millihertz to megahertz magnetic susceptibilities of weakly dipolar fluids

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Most complex magnetic susceptibility measurements have until now been performed at frequencies above 100 Hz on ferrofluids that have a susceptibility of order 1.[1] We present an improved mutual inductance set-up that can also measure at far lower frequencies on much weaker magnetic systems. This makes it possible to study rotational diffusion in colloidal fluids containing large particles or structures with multiple magnetic domains.

Frequency-dependent measurements of the magnetic susceptibility are often carried out with mutually inducing coils. An alternating electrical current in a primary coil generates an alternating magnetic field, which in turn creates an alternating voltage in a concentric secondary coil. This voltage is affected by introducing a sample at the center of the coils, an effect from which the magnetic susceptibility of the sample can be calculated.[2] One major difficulty in measuring weak susceptibilities in this way is the background signal already present without the sample. To improve sensitivity, one must try to subtract the background before voltages are measured by the electronic equipment. Another difficulty is that the signal-to-noise ratio collapses with decreasing frequency. We will present a combination of measures with which we aim to bring the background signal down to levels corresponding to the diamagnetic susceptibility of water.

Several fluid systems lend themselves for study with the presented set-up. Ferrofluids with susceptibilities of order 1 could be diluted by several orders of magnitude to examine changes in dipolar coupling between the magnetic nanoparticles. Vibrational modes in extensive gel-like networks of magnetic nanoparticles could be accessed. Local motion in liquid crystals of weakly magnetic anisotropic oxide particles could be probed. Moreover, the rotational diffusion of large composite magnetic particles similar to those used in biomedical research could be examined *in situ* in an aqueous environment. All such investigations have now been initiated in our laboratory.

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2) B.H. Erné, K. Butter, B.W.M. Kuipers, G.J. Vroege, *Langmuir* **19**, 8218 (2003).

Investigation of the relaxation behavior of magnetite nanoparticles for magnetorelaxometry

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Magnetorelaxometry (MRX) is a very promising analytical tool for the specific detection of biomolecules, such as bacteria, antibodies and proteins. In MRX the target molecules are specifically coupled to superparamagnetic nanoparticles. The method relies on the analysis of the decay of the magnetic signal of the nanoparticles after turning off a magnetizing magnetic field. Our setup is based on fluxgate magnetometers which – in contrast to SQUID magnetometers as used by other groups (e.g., [1]) – are sensors that measure absolute magnetic fields and not just field changes as a SQUID. In addition, the fluxgates are arranged with their sensitive axis perpendicularly to the magnetizing field which is provided by a Helmholtz coil. This allows one to measure the magnetic signal from the nanoparticles sample even when the magnetic field, which is of the order of 1 mT, is on.
We investigated the relaxation behavior of freeze-dried magnetite (Fe_3O_4) nanoparticles provided by various manufacturers as a function of the amplitude of the magnetizing field and of the magnetization time. In freeze-dried magnetic nanoparticles the Brownian relaxation is inhibited so that the net magnetic signal of the nanoparticles can only relax via the Néel mechanism. It is found that the generally used fit function $B_r(t)=B_N\ln(1+t_{mag}/t)$ [2] can only be applied for a very limited parameter range to describe the measured relaxation curves. The experimental data are analyzed in the framework of the magnetic moment superposition model which is also applied to describe M(H) curves measured on the same samples. Special emphasis is placed on the influence of the Néel relaxation time, both in zero magnetic field and in the presence of a magnetic field. Furthermore, we studied the magnetic signal of the sample just before switching off the magnetizing field. It is shown that this provides information on the Néel relaxation time in a magnetic field.
The results are discussed with regard to two aspects: How can one deduce the content of bound and unbound magnetic nanoparticles from MRX data and can MRX measurements be used to characterize magnetic nanoparticle samples?
This work is supported by the Deutsche Forschungsgemeinschaft via SFB 578.

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Examination of the magnetisation dynamics of rotating ferrofluids

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We investigate the magnetisation of a ferrofluid cylinder rotating as a rigid body with angular velocity Ω parallel to the cylinder axis. A d.c. magnetic field is applied normal to the cylinder axis. The rotation of the fluid leads to a non-equilibrium situation, whereby the ferrofluid magnetisation, **M**, and the magnetic field, **H**, are no longer parallel to one another.

The resultant transverse component of the magnetisation, My, is measured as a function of external field and fluid viscosity, for fields of up to 90kA/m. The Ω-dependence of My is measured in a range from a few Hz up to frequencies well above the inverse Brownian relaxation time, $1/\tau_B$.

Experimental results show ferrofluid relaxational behaviour in solid-body flow to be an order of magnitude slower than would be expected, and we offer for the first time a microscopic explanation for this phenomenon. Results are compared to the polydisperse expression for a Debye relaxor, where we see a good, quantitative agreement between experiment and theory.

Pattern formation in the Faraday instability on a ferrofluid in a vertical magnetic field

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The results of the recent experiments on the Faraday instability (i.e. parametrical generation of standing waves by vertical vibrations) on a ferrofluid in a vertical magnetic field will be presented and compared with the predictions of the weakly non-linear analysis. The purpose of the work is to study the pattern formation in the system where two mechanisms exist, each of which can alone generate the Faraday or the Rosensweig instability respectively.

The experiments are performed using a dish with diameter 30 cm, which is placed between a pair of the Helmholtz coils and is connected to a shaker. The deformations of the free surface are detected by the light reflected from the areas inclined by a certain angle. The reflected light is captured by the CCD camera connected to computer where the Fourier transform can be done.

The pattern formation is studied in the wide range of the parameters including the region, where both the Faraday and the Rosensweig instabilities can occur. Squares, hexagons, and superlattices are observed using a single-frequency driving. A qualitative discrepancy between the linear theory and the experimental data has been found. The wave number of the observed pattern is much smaller than the predicted value. Though this shift is consistent with the dependence of the wave number of the maximal growth on the acceleration amplitude, the non-linear analysis should give a deeper insight into this interesting behaviour of the system.

In the theoretical analysis, a horizontally unbounded ferrofluid layer of a finite depth is considered. The perturbation expansion of the whole ferrohydrodynamic set of equations is performed using the standard technique, in the way suggested by Chen and Viňals [1]. The principal extensions are the magnetic part of the problem and the account of the finite depth of the ferrofluid. The following particular cases are considered: i) the Faraday instability at a vertical magnetic field weaker than the critical field for the Rosensweig instability. In the case, where the primary response is subharmonic, the slow varying spatial scales are introduced to address the mentioned unexpected experimental results. This leads to a solvability condition at the second order even in the case of the subharmonic primary response. This solvability condition is independent on any introduced planform and predicts a weak decrease of the wave number at the parameters of interest. ii) If the magnetic field is above the critical field of the Rosensweig instability, there are two independent supercriticality parameters, which describe the mechanical and magnetic driving.

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Structural anisotropy of charge-stabilized ferrofluids under an external field investigated by Brownian dynamics

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Aqueous ferrofluids stability is often ensured by interparticle electrostatic repulsions. The interparticle repulsion has to be strong enough to prevent phase separation even under high magnetic fields required for practical applications. Furthermore, as the microstructure of the suspension plays a crucial role, the anisotropy of the local structure of such repulsive aqueous magnetic nanoparticles suspensions under an external magnetic field has already been explored with Small Angle Neutron Scattering (SANS) experiments[1,2]. Unexpectedly, when considering the structure factor, the highest correlation peak is found in the direction perpendicular to the magnetic field, the parallel direction being therefore the more disordered.

In order to shed some light on this phenomenon, Brownian dynamics simulations of charge-stabilized ferrofluids, with and without an external magnetic field have been carried out. The colloidal suspension is considered as a collection of spherical particles, bearing point dipoles at their centers and subjected to translational and rotational Brownian motions[3,4]. The overall repulsive isotropic interactions between particles, governed by electrostatic repulsions, are taken into account by a one-component effective pair interaction potential. The potential parameters are chosen in order that computed structure factors are close to zero field experimental SANS data. Structure factors are computed from pair correlation functions with and without an applied magnetic field. In the latter case, the microstructure of the repulsive ferrofluid is found to be anisotropic. This rather simple description of the suspension allows us to account for the main experimental features: an increase of the structure factor is observed in the direction perpendicular to the field whereas the correlation is altered in the parallel direction.

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Quasielastic light scattering investigation on magnetite-based ferrofluids

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As part of our laser-optical investigations on the magnetoviscous effect in ferrofluids we study the formation of structures in the bulk phase by Quasi-Elastic Light-Scattering (QELS). This standard method for the non-invasive determination of diffusion coefficients in colloidal systems can be most easily used in highly diluted dispersions. Yet it can also be applied to highly concentrated systems by using backscattering geometries, especially in combination with fiber-optic detection (FOQELS). We use a light scattering set-up with a classical backscattering geometry as well as FOQELS for our studies.

In the present study QELS-experiments have been performed on magnetite-based ferrofluids with water and alkanes as dispersion media. Especially we investigate the influence of particle concentration and of an external magnetic field (up to $H = 20$ kA/m) on the apparent diffusion coefficient systematically. We will present first preliminary results.

Magnetoviscous effect in Co based ferrofluids analysed by small angle neutron scattering

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One of the most important features of ferrofluids is the possibility to influence their properties, by means of moderate magnetic fields. In the presence of these fields, ferrofluids show an increase of their viscosity, the so called magnetoviscous effect. The magnitude of the effect depends on the concentration of the particles that are large enough to form structures within the fluid. In order to explain the microscopic mechanisms of the magnetoviscous effect a model based on numerical and experimental data has been established. According to this model, chain formation of magnetic particles with strong particle-particle interaction as well as structure destruction by means of shear influence are the essential processes for the understanding of the magnetoviscous phenomena.

To prove the correlation between the macroscopical behaviour and the microstructure, measurements using small angle neutron scattering technique (SANS) have been performed. For this procedure a special rheometer was designed. The most important feature of the rheometer is that it allows SANS measurements in the same geometry and under the same conditions, i.e. magnetic field strengths and shear rates, as used in the rheological experiments.

The scattering experiments were performed for different cobalt based ferrofluids, supplied by N. Matoussevitch and H. Bönnemann (Forschungszentrum Karlsruhe). The coupling parameter as well as the concentration of the magnetic material was changed, in order to facilitate formation of various structures and therefore different magnitude of the magnetoviscous effect.

Depending on the parameters of the investigated ferrofluids, strong dependency of the scattering patterns on the magnetic field strength and shear rate were observed. These results are in a good agreement with the rheological data and with the qualitative model developed to explain the mechanism of the magnetoviscous effect.

After a brief description of the experimental setup, the presentation will contain the current results of the investigation of the microstructure using SANS.

These results will be compared with rheological data concerning measurements of the magnetoviscous effect as well as with the existent numerical simulations.

The obtained experimental results will offer a starting point for theoretical approaches, leading to a detailed understanding of the magnetoviscous effect and opening the way to new technologically useful applications.

Low-frequency spectroscopy and cluster analysis of magnetic fluids

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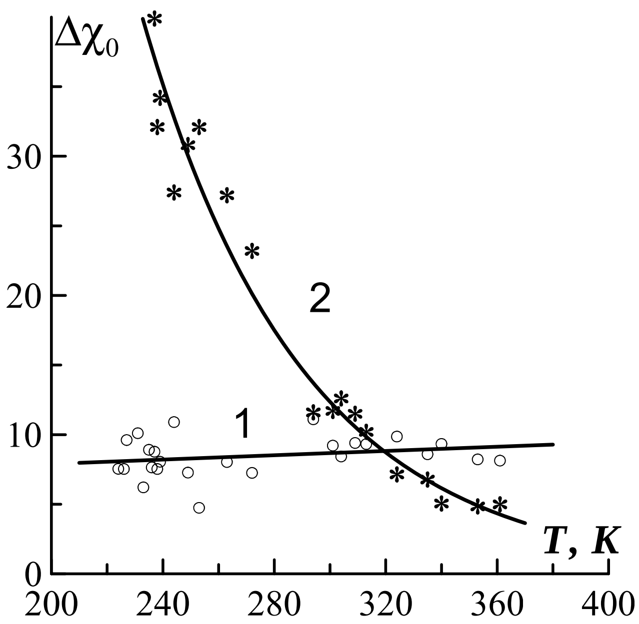
A new method for determination of sizes and concentrations of aggregates (clusters) in magnetic fluids is proposed. The method is based on the series expansion of the low-frequency spectrum of dynamic susceptibility in terms of Debye functions. The obtained series expansion coefficients are used to determine the concentration and specific sizes of particles and aggregates. The maximal size of single-domain particles in magnetite-based fluids is not more than 30 nm and, therefore, micro-objects of sizes more than 30 nm are considered as aggregates. Fraction averaging is carried out within each group particles and clusters. The proposed method was applied to magnetic fluids with high level of interparticle interactions. The dynamic susceptibility was measured with the aid of an mutual inductance bridge (10 Hz -100 kHz and 230 - 360 K). Spectral amplitudes were calculated by the iterative procedure for 14 fractions different by the relaxation time of the magnetic moment. Experiments indicated that in the magnetic fluid there is an abundant number of clusters with non-compensated magnetic moment and of characteristic size 50-60 nm exceeding the mean size of separate particles by 3-4 times. The role of temperature, coagulants (alcohol) and free oleic acid was studied thoroughly.



The contribution of the separate particles (curve 1) and aggregates (2) to the equlibrium susceptibility vs temperature.

Liquid to solid transition in inverse ferrofluids

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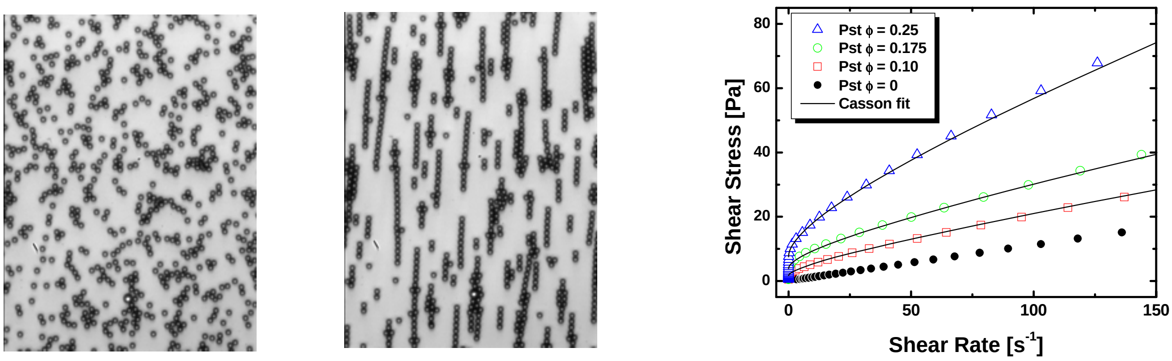
By disperging microsized polystyrene particles in ferrofluid an ideal magneto-rheological model fluid can be created [1]. Because polystyrene particles are available, which are practically monodisperse (σ=0.04), this hybrid fluid allows to controll particle size and polydispersity, in advantage to the common ferro- and magneto-rheological fluids, which are polydisperse. The nonmagnetic polystyrene particles create a hole in the ferrofluid, which appear to posess a magnetic moment corresponding to the amount and suszeptibility of the displaced fluid. Due to the dipolar interactions of the holes, chain formation sets in (see Fig. a,b) and the inverse fluid undergoes a transition from liquid to solid like behaviour.



a) b) c)

We investigate this transition by measureing the yield stress for different volume fractions Φ (see Fig.c) and magnetic fields. Moreover we are recording the storage modulus G´ and the loss modulus G´´ versus the magnetic field for different volume fraction, particle size, and particle size distribution. Our results show that for a monodisperse fluid the liquid to solid transition is more pronounced than for a polydisperse one. A related effect has recently been found in MD simulations for standard ferrofluids [2].

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On field-induced anisotropy in ferrofluids: effective axis ratios of particles and clusters

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We have investigated the field-induced dielectric anisotropy of ferrofluids at various concentrations and magnetic field strengths. Dielectric measurements were performed in a frequency range from 5 Hz to 1 GHz. An effective-medium analysis allows us to determine effective axis ratios of the respective polarized units. At high frequencies these are mainly single particles whereas at low frequencies also clusters contribute. The effective axis ratios reflect both the shape anisotropy and the orientation of the respective units. The data is compared to results from SAXS experiments[1)] and to MC-simulations[2)], that take the polydispersity of particles into account.

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Thermodiffusion and diffusion in ferrofluids under the influence of magnetic fields

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Investigations were made to analyse the thermodiffusion process (Soret effect) of magnetic particles in a ferrofluid under the influence of a magnetic field. The so called magnetic Soret effect was theoretically predicted to be two to three orders of magnitude smaller than the conventional Soret effect. In contrast, former experiments have qualitatively shown that the magnetic influence on the thermodiffusion process is much higher than the theoretical predictions for the magnetic Soret effect. However in those experiments the influence of buoyancy and magnetic driven convection disturbed the measurement significantly. Thus it is still an open question how strong the influence of a magnetic field on thermodiffusion can be and how this influence can be explained theoretically. Therefore an experimental setup was developed which minimizes parsitic effects, simplifying the analysis of the experimental results. These results provide quantitative measures of the magnetic field dependence of the Soret effect in suspensions of magnetic nanoparticles. If this influence is taken as a magnetic Soret effect it is shown that it can even be higher than the conventional one and that its strength as well as its direction depend on the magnetic field strength and its relative alignment to the temperature gradient in the fluid.

Additional investigations were made to determine the diffusion coefficient of magnetic nanoparticles in a Ferrofluid under the influence of a magnetic field. This “magnetic diffusion effect” was also predicted to be a function of the strength as well as the direction of the field relative to the concentration gradient. In former experiments the diffusion coefficient could only be measured by optical methods u to medium field strength.

With the thermodiffusion column mentioned above it is possible to determine also the diffusion coefficient even for very strong magnetic fields with high accuracy.

The results obtained by using this experimental setup provide quantitative measures of the magnetic field dependence of the diffusion coefficient in suspensions of magnetic nanoparticles. It is shown, that the magnetic diffusion coefficient depends on the magnetic field strength and its relative alignment to the concentration gradient of the particles in the fluid.

In the poster we will present the theoretical model, the experimental setup as well as actual results.

Direct measurement of subfemtonewton dipolar forces in suspensions of magnetic particles

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Colloidal core-shell particles consisting of cobalt ferrite as magnetic core and silica as shell interact both electrostatically induced by surface charges of the silica shell and via a magnetic dipole-dipole potential. Compared to conventional ferrofluids, these particles are much more defined in terms of polydispersity and self organize to liquid-like or even crystalline structures [1-3].
The magnetic pair interaction in dilute suspensions with a volume fraction in the range of ϕ=0.03 is for this system as low as $10^{-2}\, kT$
for neighboring particles. Without the presence of an external field, the orientation of the magnetic moments is random and, as consequence, the resulting magnetic interaction vanishes. In the presence of an external field, however, the moments align parallel to the field leading to a magnetic attraction in field direction anda magnetic repulsion perpendicular to the field direction. Hereby, a structural distortion of the short range, liquid-like order can be observed by means of Small Angle X-ray Scattering (SAXS).
For small magnetic interactions, the resulting anisotropic structure factors can theoretically be described via the Rescaled Mean Sphere Approximation (RMSA) for a magnetic Yukawa interaction. The analytic solution for the structure factor depends on the number density, the effective charge of the particles, the Debye screening length and, finally, the magnetic moments. The magnetic interaction parameter is obtained from twodimensional analysis of the scattering pattern. Hereby, magnetic forces in the range of $10^{-16} N$ can be measured thanks to the extremely soft, screened Coulomb interaction in concurrence to the dipole interactions and the outstanding experimental accuracy of synchrotron SAXS .

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Rosensweig Instability in Ferrogels

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Ferrogels are chemically cross-linked polymer networks that are generated using a ferrofluid as a component. As was shown in many publications [1,2], there exists a coupling between the elastic and magnetic degrees of freedom allowing to control the mechanical behavior by applying external magnetic fields. A hydrodynamic description of isotropic ferrogels has been given recently [3].
If a magnetic field is applied normal to a free surface of a ferrofluid, an instability of the surface occurs above a certain threshold resulting in a periodic, stationary structure of spikes [4]. The threshold field depends on the surface tension and gravity, but is independent of the viscosity of the ferrofluid. However, for a ferrogel the elasticity does influence the threshold, but not the critical wavelength at onset [5]. We discuss also the possible influence of the viscosity of the ferrogel on the instability. In addition, we investigate the Rosensweig instability for the case of a uniaxial gel with frozen-in magnetization [6], based on recent hydrodynamic equations for such a system [7].

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Thermal convection in colloidal suspensions with negative separation ratio

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Thermal convection in colloidal suspensions of nanosized particles is investigated. Representative examples for such materials are ferrofluids, but since we do not imply any external magnetic field, the description applies to nonmagnetic suspensions as well. With the grain size being large on molecular length scales, the particle mobility is extremely small, allowing to disregard the concentration dynamics in most cases. However, due to the pronounced Soret effect of these materials in combination with a considerable solutal expansion, this cannot be done when thermal convection is under consideration [1]. Here we consider the case when the separation ratio (the Soret coefficient) is negative. This case reveals a much richer variety of phenomena than that of positive separation ratio. In particular, for heating from below we find a linear oscillatory instability, whose amplitude, however, relaxes to zero on the long turn and is, thus transient only and, at higher Rayleigh numbers, a finite amplitude stationary instability coexistent with the linearly stable convection-free state. By heating from above short-length-scale convective structures occur, whose wavelength depends on the Rayleigh number [2].

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Experimental research of the magnetic fluid comverter

V. M. Polunin, A. G. Besedin, V. M. Paukov, M. V. Chistyakov

The article deals with the experimental research of the converter of sound oscillations into electromagnetic the active element of which is the magnetic fluid. The opportunity of functioning of magnetic fluid converter in the field of frequencies adjoining to the bottom border of an ultrasonic range (10 – 65 kHz) is shown - on the acoustic-magnetic effect basis.

In the field of high frequencies (10 – 65 кHz) research was made on the acoustic-magnetic effect basis (AME) [1,2]. The diagram of an experiment on studying AME in a rotating magnetic field is shown in Fig.1.

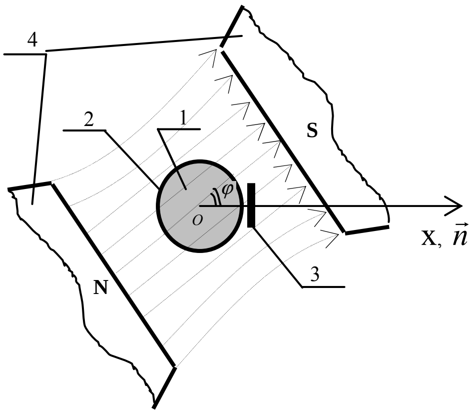

Fig. 1. The diagram of experiment

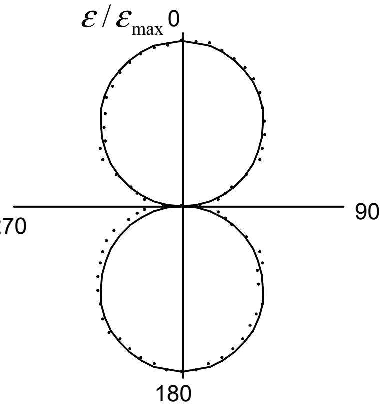

Fig. 2. Dependence of relative amplitudes AME on angle φ

Dependence of amplitude induced by EMF on the angle φ in relative units is presented in Fig.2. The thin line shows the graphic $\cos\varphi$.Thus, during only one revolution of a magnet the amplitude, following the change of $\cos\varphi$, accepts the maximum value twice and is twice equal to zero. Fig.3 presents curve 1 – experimentally received dependence of the cross component to the tube of magnetic intensity upon distance along the axis; curve 2 – dependence EMF of an induction ε on the distance measured along the axis of the tube.

The dependence of amplitude EMF of an induction ε_0 on amplitude of a voltage of variable EMF going to piesoelement U is shown in Fig.4.

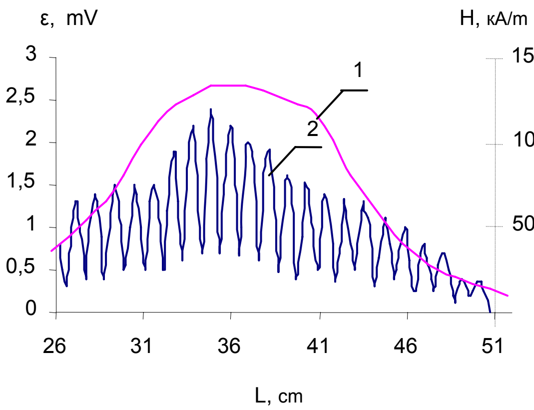

Fig.3. Dependences $H(L)$ – curve 1and $\varepsilon(L)$ – curve 2.

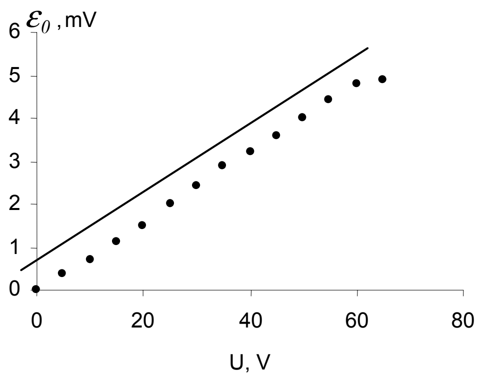

Fig.4. Dependence $\varepsilon_0(U)$.

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Experimental research of the magnetic fluid membrane

V. M. Polunin, J. J. Kameneva, V.V. Kovarda

In the field of low frequencies (20 – 200 Hz) the research has been made with the use of magnetic fluid membranes (MFM).

The magnetic field in borders of the drop contour (dotted line) is mainly directed along the axis of the annular magnet, i.e. the axial component of field H_z is prevailing; in the radial direction small growth H_z is observed; the radial component of field H_r is absent in plane $z = 0$ and tends to increase in the vicinity of the axis. The approach of the “low-magnetic” environment and marked features of geometry of the magnetic field in a zone of an arrangement of the crosspiece, testifying a determining role in ponderomotive elasticity formation of the axial component of magnetic field H_z, are used for calculation of ponderomotive elasticity factor.

In the methodical attitude one of the most important questions is the establishment of borders of a dynamic range. An experiment with magnetic fluid membrane (MFM) has been made to find the latter. Magnetic fluid crosspiece blocks the section of the tube being the neck of the glass flask in volume 0,5 *l*. At rise of the flask on height Δz above a support and its fixation in this position by soft pressing, the crosspiece is displaced concerning the position of equilibration on δz, that is

$$\delta z = \frac{k_g}{k_g + k_p} \Delta z, \qquad (1)$$

here k_g – elasticity of the gas cavity, k_p – elasticity of ponderomotive type.

At sharp returning the flask in a starting position due to inertness the crosspiece appears to be displaced concerning the position of equilibration, it predetermines the development of the oscillatory process. At the moment of passage of position of equilibration by the crosspiece maximum value EMF– ε_m is fixed. Sharp moving of the flask is achieved under the influence of the impact. MF used in mechanical engineering, representing the colloid solution of one-domain particles of magnetite Fe_3O_4 in kerosene (MF-1 and MF-2) are applied. Physical parameters of magnetic colloids are shown in Table1:

Sample	ρ, kg/m^3	η_s, Pa·s	M_s, kA/m	χ
MF-1	1294	$3{,}2\cdot10^{-3}$	52±1	6,2
MF-2	1499	$8{,}1\cdot10^{-3}$	60±1	7,5

Here: ρ – is density MF, χ – an initial magnetizability, η_s – colloid static shift viscosity. The listed parameters were defined by standard methods.

On Fig. 1 the dependence $\varepsilon_m(\Delta z)$ received for MFM on the basis of colloid MF-2 is shown. Under the conditions of the given experiment the height of the cargo falling h' =20,3 mm. Temperature is T=21 ± 0,5°C. Linear approximation is executed with the use of the program MS Excel. At $\Delta z \geq 3{,}5$ mm for MF-2 and $\Delta z \geq 4{,}5$ mm for MF-1 backlog of dependence $\varepsilon_m(\Delta z)$ from the linear is observed. Let’s term device β – a tangent of an angle of an inclination of an approximated line as sensitivity (to displacement), and value of first oscillation’s amplitude at $\Delta z=0$ as the initial impulse ε_{m0}. In Table 2 values β and ε_{m0}, received from the Fig. 1. Dependence $\varepsilon_m(\Delta z)$ experiment with various height of the cargo falling h' are shown.

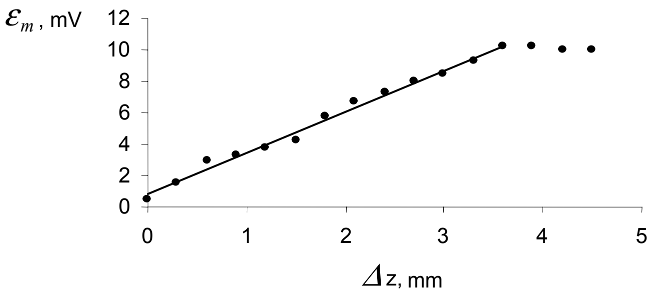

Table 2

Colloid	h', mm	β, mV/mm	ε_{m0}, mV	Colloid	h',mm	ε_m/h', mV/mm	ε_{m0}, mV
MF-1	9,0	4,64	0,5	MF-2	10,8	2,53	0,75
	14,6	4,88	0,5		20,3	2,62	0,50
	19,4	5,32	0,5				

The parameter β increases almost in 2 times if to use colloid MF-1 instead of more concentrated colloid MF-2. It is possible to assume, that the specified result is caused by the negative role of viscous friction’s forces due to which the amplitude of initial displacement of the crosspiece from the position of equilibrium at the moment of drawing of impact is decreased.

Ferrofluids simulation using Brownian dynamics: the role of hydrodynamic interactions

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Ferrofluids simulation is a topic of increasing interest as the computers speed allows the study of realistic representative systems. The numerical techniques are the Brownian Dyanmics and the Monte-Carlo ones, and take into account the magnetic dipolar interaction and the particle-to-particle interactions [1]. But most of these simulations neglect the hydrodynamic interactions (HI): when a particle is moving inside the solvent, it creates in its neighbourhood a flow, which induces a perturbation in the displacement of the other particles.

The present work studies the influence of these hydrodynamic interactions on the predictions of physical properties of a ferrofluid subjected to a flow, namely the anisotropic diffusion, or the viscosity. These HI are calculated in Brownian Dynamics simulations from the Rotne-Prager tensor, whereas fast Cholesky decomposition is used to get the correlated random displacement of the moving objects, which allows a significative number of particles to be simulated [2]. The system is subjected to a shear flow (employing Lees-Edwards boundary conditions). Simulations have been performed for on- and off-equilibrium, for different values of shear rates and dipolar interactions. The results are compared to experimental and other numerical data to point out the role of hydrodynamic interactions in ferrofluids systems.

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Simulation of thermomagnetic convection in a ferrofluid under micro-gravity conditions

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A 2-D computational simulation has been undertaken to study the flow patterns of themomagnetic convection in a ferrofluid under microgravity conditions in the region of the onset of fluid instabilities. The simulation has been designed to complement the drop tower experiments of Odenbach (1) and the linear stability analysis of Zebib (2) in order to clarify the conditions and mechanisms of the phenomena. The analysis of Zebib predicts a n=4 wave number for the 2-D azimuthal flow pattern which corresponds well with the observations of Odenbach. Zebib also predicts a value of the Magnetic Rayleigh number for the onset of fluid instabilities although he notes that there are other competing modes in the vicinity. The simulations show that although there is a significant n=4 flow pattern in the region predicted by Zebib, the dominant process of heat transfer in this region is conduction. The simulations indicate the existence of a well defined transition to thermomagnetic flow occurring at a higher than predicted value of the Magnetic Rayleigh number, which is marked by change to the characteristic temperature distribution observed by Odenbach Figure 1.

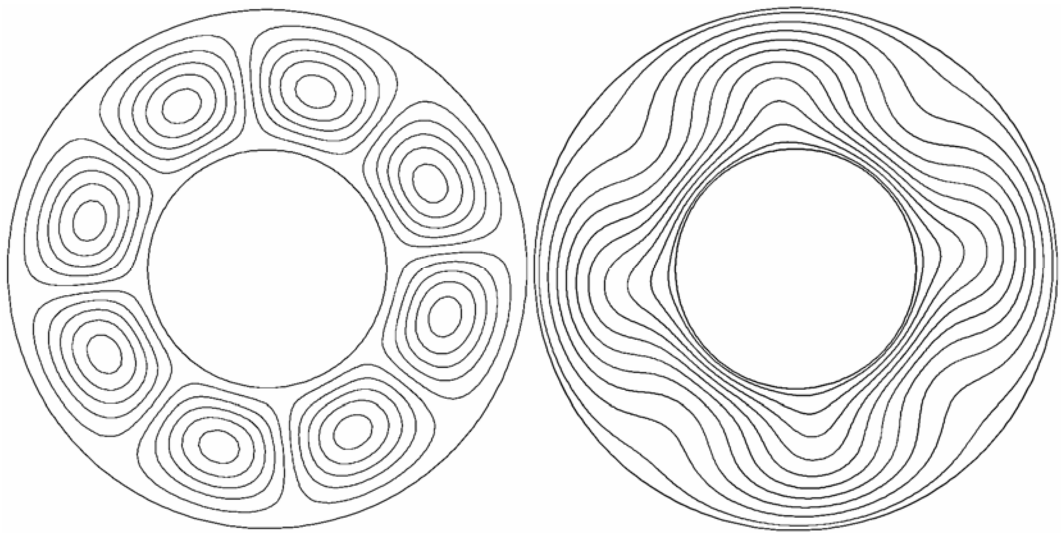

Figure 1: Mode n=4 flow Streamlines and Temperature Contours.

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Theoretical investigation of ferrofluid properties with fractal aggregates

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Imbalance of attraction and repulsive interparticle interactions leads to formation of various aggregates in ferrofluids. The structure of these aggregates is determined by physical and chemical properties of the system. Fractal aggregates may arise in ferrofluids [1] due to the action of molecular forces in a way similar to classical mechanism of colloidal coagulation. As far as the physicochemical reasons of ferroparticle coagulation are concerned, there could be a strong van der Waals attraction, as well as deformation or destruction of surface sterical layers and low values of interparticle repulsive electrostatic barrier in ionic ferrofluids. The presence of aggregates and their internal structure influence greatly properties of ferrofluids [2]. This paper is devoted to the theoretical study of fractal clusters, which may arise in the ferrofluids, and to the determination of effective magnetic permeability of ferrofluids with fractal aggregates.

The aggregation of the ferroparticles can be initiated by two ways. The first way of aggregation caused by the presence of specific nuclei in the system. The second mechanism take place in the system without specific centers of nucleation. In the first case, the part of nucleation centers can play, for example, the large particles. As a result, aggregate fractal dimension has been found analytically. Also, the concentration of dispersed particles and the aggregate size as functions of time have been found. In the second case, the aggregates appear as a result of occasional meeting of some ferroparticles. The developed theoretical model takes into account the aggregate size distribution. Finally, the concentration of dispersed particles as function of time and fractal dimension have been found numerically.

In an external magnetic field an effective permeability of a ferrofluid with fractal aggregates $\overline{\mu}$ has been obtained. This value has been compared with the one of aggregated ferrofluid with drop-like microstructures $\overline{\mu}_\Sigma$. As a result, for the same aggregate sizes, the value $\overline{\mu}_\Sigma$ has been found larger then $\overline{\mu}$. The difference has been proved to increase with the growth of aggregate.

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Gravity level influence on a horizontal ferrofluid layer submitted to an inclined strong magnetic field and to lateral heating

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For strong magnetic fields, three different characteristic inclinations of the magnetic field are considered:

A] When the magnetic field is parallel to the layer, the magnetic field does not change the classical Birikh flow of a Newtonian fluid.

B] If the magnetic field is applied normally to the layer, the magnetic field has a limited influence since it does not change very much the shape of the velocity and temperature profiles.

C] For an inclined magnetic field, the solution is expressed as a power series in terms of a small dimensionless parameter measuring the ratio of variation of the magnetization across the layer to the magnitude of the external field i) When the gravitational Rayleigh number is larger than the magnetic Rayleigh one, the zero-order approximation does not differ much from the Birikh classical solution The magnetic field influences slightly the first-order approximation to reduce the maximum velocity and the temperature deviation from the steady-state distribution. ii) When Ra is smaller than Ra_m, which is a very likely solution in microgravity, the solution can be approximated by the classical Birikh solution multiplied by a factor independent upon the layer width, $I = 1 + \frac{Ra_m}{Ra} \sin\phi \cos\phi$, where ϕ is the angle of the magnetic field inclination. The inclination then plays a major role since it van lead to a pure conductive solution at a critical inclination or revert the direction of flow..

We are now developing the stability study of that reference steady state

Polydispersity influence upon magnetic properties of aggregated ferrofluids

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The presence of chains, formed due to the magnetic dipole-dipole interaction, leads to the strong deviation of magnetization curve from the Langevine curve for ideal superparamagnetic gaz. Voluminous literature is devoted to the study of interpartical correlation influence upon ferrofluid magnetic properties, Refs. [1-3], for example. In paper [2] the chain input into magnetization and initial susceptibility was estimated, nearly the same estimations were obtained in computer simulations in Ref. [3]. However the majority of theoretical and computer investigation in this field deals with model monodisperse ferrofluids, whereas it is well known that the polydispersity is an inescapable feature of any real magnetic fluid on the one hand, and influences greatly the chain properties on the other.
The present paper is aimed at the comparison of the bidisperse ferrofluid computer simulation results (Ref. [4]) with the theoretical model (bidisperse ferrofluid with chains) predictions (Ref. [5]). In the above mentioned paper [4] the analysis of magnetization curves for the fluids with different large particle concentrations was carried out in particular. This analysis resulted in the strong dependence of the magnetization in low fields on the concentration of large particles. Thus, when the large particle concentration is small the magnetization behavior is close to the one given by the modified mean field approach, but the higher is the portion of large particles the more noticeable is the growth of the initial susceptibility. The latter couldn't be described in terms of homogeneous ferrocolloids. An attempt to describe this effect in the frame of large particle chains only didn't give us quantitatively good agreement with simulation results, although it gave the opportunity to study the magnetization dependence not only upon the chain length, but also on the chain concentration. On the basis of the model developed in Ref. [2] its bidisperse generalization was built with the help of model from Ref. [5], and the magnetic curves were calculated for the parameters from Ref. [4]. The theoretical results appeared to be in a good agreement with the ones of computer simulations (Ref. [4]).
The research was carried out under the financial support of Russian Federation President Grant № MD-336.2003.02, RFBR Grant № 04-02-16078a, RFBR-DFG Grant № 03-02-04001, INTAS № 03-51-6064, REC-005(EK-005-X1) Grants, also it was supported by CRDF and Russian Ministry of Education and Science.

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Concerning the dependence of equilibrium ferrofluid drop shapes on uniform magnetic field

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The main objective is the numerical modeling of equilibrium shapes of an axisymmetric ferrofluid drop subjected to a uniform applied magnetic field. The mathematical model consists of a coupled system of the Maxwell's equations for the magnetic field distribution and the Young-Laplace equation for the free surface shape [1]. The Maxwell's equations are formulated in the domain with an *a-priory* unknown fluid-air interface.
For solving the Maxwell's equations, reformulated in terms of a scalar potential, the coupling of the collocation boundary element method (BEM) and the finite element method (FEM) [2] is used. The free surface is parametrised with respect to the arc length and a finite-difference scheme is applied for computing equilibrium drop shapes as solutions of the Young-Laplace equation on the interface [3]. The coupling between Maxwell's and Young-Laplace equations is realised by the iterative procedure.
We reviewed previously done numerical studies of the problem in [4,5] and developed a new numerical strategy. Applying this strategy we recovered the hysteresis effect for the drop deformation, which could be established experimentally [6]. The transition from the shapes with rounded ends to the equilibrium shapes with conical interfaces was observed numerically. The dependence of equilibrium shapes on magnetic field was investigated in a wide range of field intensities. The nonmonotone behaviour of the drop elongation versus the increasing magnetic field was detected numerically.

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Periodically forced ferrofluid pendulum

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We investigate the dynamics of the response of a torsional pendulum containing a ferrofluid that is forced periodically. A homogeneous magnetic field is applied perpendicular to the pendulum axis.

Magnetization of rotating ferrofluids – comparison with theoretical models

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We consider a ferrofluid, that is rotating with constant rotation frequency $\mathbf{\Omega} = \Omega\mathbf{e}_z$ as a rigid body. A homogeneous magnetic field $\mathbf{H}_0 = H_0\mathbf{e}_x$ is applied perpendicular to the rotation axis $\mathbf{e}_z$. This causes a nonequilibrium situation. Therein the magnetization $\mathbf{M}$ and the internal magnetic field $\mathbf{H}$ are constant in time and homogeneous within the ferrofluid. According to the Maxwell equations they are related to each other via $\mathbf{H} = \mathbf{H}_0 - N\mathbf{M}$. However, $\mathbf{H}$ and $\mathbf{M}$ are not parallel to each other and their directions differ from that of the applied field $\mathbf{H}_0$. We have analyzed several different theoretical models including some that take into account the polydispersity of the ferrofluid. The model results are compared with experimental data.

Solitary waves on ferrofluid surfaces

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The interaction between hydrodynamic and magnetic properties in ferrofluids offers a variety of Particular nonlinear phenomena. A well known nonlinear effect oh hydrodynamics are solitons. They are characterized due to astonishing properties, e. g. non-dissolving of wave pachets, stable to collisions with each other etc.
We demonstrate, it is possible to get an equation with solitary wave solutions in Ferrofluids in case of suitable external magnetic fields. In particular a cylindrical setup is investigated in detail and we demonstrate, how to map the hydrodynamic problem to the Korteweg-de-Vries equation [1,2]. Additionally we show, on conditions where Rosensweig instabilities appears, the existence of the Korteweg-de-Vries equation is excluded.

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Surface tension of magnetic fluids in mean spherical approximation

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With the appearance of magnetic fluids (MFs) in mid 60ies, the problem of phase transition has been the subject of heated discussion in the literature. However, some fundamental aspects of the problem are still an open question. At present the most comprehensively studied effect is the phase transition of the first kind—stratification of MF into weakly and highly concentrate phases under the action of magnetodipole inter–particle interactions [1, 2]. In this case, the concentrated phase is represented by the so-called drop aggregates with characteristic dimensions of a few tens of mkm. In the absence of external fields the aggregate takes the spherical shape due to the existence of the surface tension at the interface between the drop aggregate and colloidal solution. A nearly spherical shape is also preserved in a weak magnetic field. However, despite a great body of experimental data [3–5] the evaluation of the surface tension coefficient even in the framework of the mean field model (MFM) is a challenging task, which has been accomplished quite recently [6, 7]. The calculations of this kind in the context of the microscopic mean spherical aproximation (MSA) [8] are still to be done.

Therefore in the present investigation the evaluation of the surface tension coefficient and its dependence on the particle density, temperature for a plane and spherical interface between weakly and highly concentrate phases of MF was made in square-gradient approximation for MSA of dipole-dipole interaction. Calculation of the density profile in the "liquid–vapour" transient layer, the influence factor, the surface tension coefficient were made as functions of temperature, concentration and magnetic field. These results are possible to present as functions of "liquid–vapour" phases physical measured parameters: magnetic susceptibility, density and compressibility. That opens possibility to use these expressions for description of polydisperse magnetic fluids. The theoretical results agree well with experimental estimations [3–5].

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Derivation of thermomagnetic coefficients from thermodynamics of irreversible processes

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Well established and experimentally verified expressions, providing the thermomagnetic coefficients, exist (1,2). Thermomagnetophoretic effect is sometimes also considered as a magnetic Soret effect (3) that adds to the thermophoretic Soret effect acting on dispersions. It seemed interesting to derive such expression from the general formalism of Thermodynamics of Irreversible Processes. Although no new results are expected, this approach should allow to quantitatively distinguish the two effects.
To obtain such general relations, it is necessary to consider also polarised systems, as performed for electrical effects in (4). To do so, the chemical forces in polarized systems are provided, and the local mechanical equilibrium approximation is used to eliminate the pressure from the momentum balance equation. Such approach has been demonstrated to be valid in other colloidal configurations (5).
In order to be able to compare the results, isotropic systems are considered and following Rosensweig's approach, the molar magnetisation is linked to the magnetisation of colloids trough the Langevin model for diluted ferrofluids. When applied to spherical particles, the expressions obtained for the thermomagnetic coefficient differs from the classical expression (1) by a multiplicative factor $(1+1/\chi)$. The underlying physical explanation linked to this difference is not clear for the moment. More elaborated model from TIP should be considered.

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Cylindrical magnetic fluid layer in a traveling magnetic field

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The possibility of creating a viscous magnetic fluid flow by means of a magnetic field is investigated. This effect can be used in designing autonomous mobile robots without a hard cover. Such robots can be employed in clinical practice and biological investigations. In [1], the flow of a viscous fluid layer due to an undulation of bounding impermeable walls was considered. The undulatory perturbations of the wall surfaces and the fluid velocity at the wall were assumed to be given. The average flow-rate in the layer was calculated for the boundary moving as a sinusoidal travelling wave and the boundary velocity perpendicular to the unperturbed boundaries. In [2], an analogous flow was considered taking into account the influence of the adjacent layer of another viscous fluid. The undulatory perturbation of the interface between the two viscous fluid layers was given. For the boundary perturbed in the form of a sinusoidal travelling wave, the average flow-rate in the layer was calculated. In [3], the behavior of a magnetic fluid film on a rotating horizontal disk in a nonuniform magnetic field was studied. It was shown that the magnetic field affects the film shape considerably, for example, turning on the field leads to the formation of a moving layer thickness jump. In [4], the motion of a magnetic fluid layer on a plane in a travelling magnetic field was investigated experimentally. A dependence of the surface velocity on the magnetic field amplitude and frequency and the layer thickness was revealed. In [5,6], the theoretical analysis was performed within the framework of the ideal fluid model. In the present study, the motion of an incompressible magnetic fluid layer on a cylinder, produced by a nonuniform axisymmetrical magnetic field, is analyzed within the framework of the viscous fluid model. The strength of the magnetic field producing the travelling sinusoidal wave, and the velocities, including those at the surface, are to be found. The dependence of the magnitude of the magnetic field on the wavelength is analyzed and the average flow-rate is calculated.
This work is supported by Deutsche Forschungsgemeinschaft (DFG, ZI 540/7-1).

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Ferrofluid controlled by ac-fields

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Ferrofluid filled in a circular channel, which is exposed to an external magnetic field as shown in Fig. 1, is set in motion.

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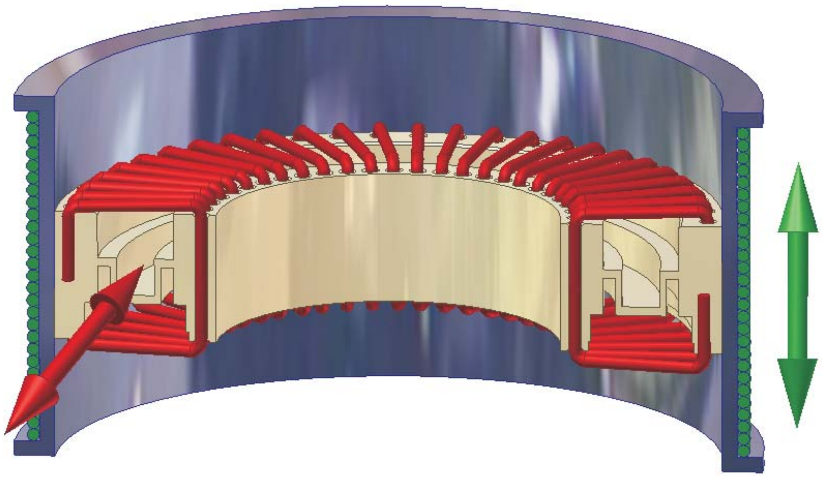

FIG. 1: Experimental setup of the magnetic pump. The arrows indicate the direction of the oscillating magnetic fields provided by the coils.

Considering this effect and its theoretical description [1], led us to the conclusion that another controlling mechanism of ferrofluid should work in the same manner with droplets.

Thus we arranged an experimental setup where magnetic liquid droplets floating on the surface of another non-permeable liquid move linearly in a rotating field. The velocity of the droplets is changed depending on the applied ac-field strength and frequency as well as the size of the droplets (making a size seperation possible). With some modifications to the field geometry the droplets can be forced to make any 2-dimensional movement on the surface plane.

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Multidimensional magnetofluidic positioning systems

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The ability of ferrofluids for being controllable with applied magnetic fields enables their use as active medium in technical applications. Their main advantage is the use of electromagnets for varying the magnetic fields in a wide range. Investigations within the field of magnetofluidic positioning systems enabled the development of prototypes of controlled multidimensional systems. Thereby, the general underlying physical effects are levitation, displacing, coupled motion, and magnetically stresses. The force for the motion of a levitating paramagnetic actor is generated by the different permeability of the ferrofluid and of the actor. One-axis magnetofluidic positioning systems are characterized by a high positioning accuracy, a high load capacity and an inherent distinctive compliance as a protection against overload [1, 2, 3]. Their disadvantage is the dependence of the high positioning accuracy on the fixed position of the experimental set-up and on the active direction. This means that each deviation could lead to a significant decrease of accuracy. One technical solution for solving this problem is the opposite arrangement of combined controlled coils. These resulting new positioning systems can be used for gripping applications of sensitive objects at a set preload. Due to the resulting low forces in miniaturized types of positioning systems the sealing of the actor shaft requires low friction coefficients. This can be realized by using non-contact seals with permanent magnets. Thereby, the overlapping of simultaneous existing magnetic flux lines has to be considered for avoiding leakages. Actual results of preliminary investigations for sealing the actor shaft with different permanent magnets, their arrangement, the gap widths and used ferrofluids as parameters will be presented at the workshop.

Acknowledgement
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Distribution von mitoxantron nach magnetischem drug targeting in vivo und in vitro

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Eine lokale Chemotherapie hat neben der Reduktion unerwünschter Nebenwirkungen das Ziel, die Wirksamkeit des verwendeten Chemotherapeutikums gegenüber der systemischen (regulären) Therapie zu verstärken. Eine Form der lokalen Chemotherapie ist das Magnetische Drug Targeting (MDT). Dabei wird das Chemotherapeutikum an magnetische Nanopartikel gebunden und mit einem externen Magnetfeld in ein bestimmtes Körperkompartiment (i.e. Tumor) gesteuert. In vorangegangenen Studien wurde das Magnetische Drug Targeting bei Kaninchen mit einem VX2-Plattenepithelkarzinom angewendet und mit der systemischen Applikation verglichen [1]. HPLC-Untersuchungen haben gezeigt, dass mit Mitoxantron-gekoppelten magnetischen Nanopartikeln und intraarteriellem Magnetischen Drug Targeting unter Verwendung von nur 50 % bzw. 20 % der regulären systemischen Dosierung in der Tumorregion Tumor und umgebendes Gewebe ≤ 1 cm) 62mal mehr Mitoxantron angereichert werden kann als nach alleiniger systemischer Applikation [2]. Die zelltoxische Wirkung des Mitoxantrons beruht darauf, dass es in der im Zellkern befindlichen DNA interkaliert und Strangbrüche verursacht. Mittels Fluoreszenz-Photometrie konnten wir das Mitoxantron in der DNA qualitativ nachweisen.

DFG SPP 1104 (AL552/2-2)

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The ferrofluid influence on the bacteria fluorescence

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The experimental investigation was intended to reveal the response of some pathogen bacteria to low concentration ferrofluid supply. The ferrophase was prepared according to [1] from ferrous and ferric salts reacting in ammonia hydroxide medium. The ferrophase precipitate (magnetite and maghemite) was coated with ammonium oleate (the physical diameter ranging between 4 and 25 nm with an average physical diameter of 10 nm) dispersed in water. The saturation magnetization was equal to 120 A/m while the ferrophase volume fraction was equal to 1.5%. The biological material was chosen as a fluorescent bacteria, namely *Pseudomonas aeruginosa*, ATCC collection culture. This bacteria is known as a producer of pyoverdine, an iron chelate belonging to the siderophore class [1]. The pyoverdine production is related to the lack of iron in the environment so these bacteria are iron scavengers and their sensitivity to the colloidal iron oxides have been reported in relation to its fluorescence intensity [2]. In this experiment the fluorescence of *P. aeruginosa* cultures (under the influence of various ferrofluid concentrations) was studied in time, by measuring the fluorescence intensity at 490 nm at 6-12-24-48 hours. The results suggested that two concurrent phenomena can explain the variation of the bacterial fluorescence in time for various ferrofluid concentrations: the bacterial population dynamics in limited culture medium and the internalization of the iron supplied by mean of ferrofluid. Peculiar behavior was recorded for relatively low ferrofluid concentration during early time measurement. Experimental procedure was repeated five times in the same controlled conditions. Statistical significance was assessed comparing the average values and standard deviations corresponding to ferrofluid samples and control ones by means of t-test considering the statistical significance of 0.05.

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Magnetic nanoparticles for biomedical heating applications

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Magnetic nanoparticles have attracted increasing interest for biomedical applications in the last years. In the present paper we consider the suitability of magnetic nanoparticles for heating purposes in bio-medicine. The heating effect of magnetic nanoparticles is caused by several mechanisms of energy dissipation if subjected to an alternating magnetic field. We are interested in two types of applications: First, heating of particles inserted into tumor tissue in order to damage tumor cells (hyperthermia) or to destroy them (thermoablation), and second, drug targeting by thermally mediated opening of a drug capsule at a predetermined site within the body (e.g. in the gastrointestinal tract). Since it is advantageous for both applications to use low amounts of particles one needs rather high specific heating power (SHP). Moreover, due to technical limitations of the magnetic field amplitude achieved at the target SHP has to reach high values even for relatively small field amplitude (below about 10 kA/m).
It was shown by previous investigations that SHP depends strongly on mean particle size as well as size distribution width. For the biocompatible magnetic iron oxides (magnetite and maghemite) a maximum of SHP was found for typical sizes larger than superparamagnetic particles but clearly below the size of multidomain particles. The highest value of SHP was found for magnetosomes (mean size: 39 nm) produced by bacteria. Nearly 1 kW/g was measured at AC-field parameters of 410 kHz and 10 kA/m. Unfortunately, those particles are available only in rather small amounts. Therefore, we have investigated chemically precipitated particles in a range of mean diameters of 10 nm up to about 100 nm. Powder preparation was done by two different methods; the mean particle size was determined by XRD, BET, SEM, TEM. Magnetically, the particles were characterized by measuring the hysteresis loss per cycle in dependence on field amplitude using a vibrating sample magnetometer. Since hysteresis losses in this range of particle size may be influenced by thermal relaxation processes (Néel or Brown type), SHP was determined calorimetrically as described previously. Results show that with decreasing particle size hysteresis loss underestimates SHP measured calorimetrically at high frequencies. The effect of measurement frequency on hysteresis loss is shown experimentally. Considering useful SHP for applications the chemically precipitated particles are inferior to magnetosomes. Results are discussed in the frame of known theoretical models of nanoparticle magnetism.

In vitro investigation of MNP-uptake by human tumour cells and their interaction with magnetic RF-fields

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It was predicted by Gilchrist et al. [1] more than 4 decades ago that the interaction of intracellular magnetic nanoparticles (MNP) with magnetic RF-fields may raise tissue temperature up to harmful values. Rabin calculated negligible effects due to intracellular single cell "hyperthermia" [2]. However, he introduced some questionable simplifications concerning the properties of biological tissue.
The aim of our investigations is to investigate in cell experiments whether intracellular magnetic hyperthermia is indeed impossible. We therefore have labelled adherently growing human tumour cells with biocompatible ferric oxide nanoparticles with and without modification of the particle coating. The modifications were due to (a) increasing the cellular uptake and (b) visualisation of the particles within the cells. The uptake was proven qualitatively using magnetic cell separation and Perl's staining as well as determined quantitatively by means of atomic absorption spectroscopy. Special interest was directed towards the intracellular localisation of the MNP. This was accomplished by means of confocal laser scanning microscopy. Furthermore, it was intended to optimise the specific heating efficiency of the nanoparticles used. Thus, different particle sizes were separated magnetically according to a modification of the method described by Rheinländer et al. [3]; the heating efficiencies of the fractions were examined with respect to dependence on either frequency or field amplitude. Moreover, the contribution of different relaxation mechanisms responsible for heating was investigated by means of freeze-drying. Therefore, a continuous tunable calorimetric device was designed [4].
The results show a strong interrelation between particle concentration and MNP uptake but only little influence of the investigated coatings. There is evidence for the localisation of MNP in late endosomes in the proximity of the cell nuclei, even though in selected cases fluorescent dye was accumulated in the nuclei. The specific heating power was increased by more than factor two due to the fractionation process. The frequency dependence within the investigated range was found nearly linear; the power law of field amplitude depends on the particle size. The results of calorimetric measurements are in fair accordance with theoretical predictions. At the current stage we observed in agreement with Rabin no significant proof of thermal damage by means of single cell hyperthermia.

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Application of functionalized magnetic beads to produce structured surfaces for contacting living cells

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Artificial magnetic nanoparticles of 20–100nm diameter are applied to investigate the influences of different surfaces on cell growth. It is possible to functionalize those particles by binding for example growth or differentiation factors to their surfaces by carboxylic groups. Permalloy structures in the micrometer range are brought to a substrate by e-beam lithography and by a lift-off process afterwards. An external magnetic field polarizes the Permalloy and allows the deposition of the magnetic beads from the liquid phase in a well defined manner. This method deliveres a helpful tool to produce functionalized surfaces with different chemical and topographical properties, because it is easy to vary the concentration of the allocated biomolecules by external magnetic fields and different particle concentrations. Another advantage is that structures can be obtained without difficult chemical techniques of binding biomolecules to a biocompatible surface.

Some immobilization modes of biologically active substances to fine magnetic particles

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One of the prerequisites for success of the application of drug targeting for the treatment of localized diseases is the development of an effective method to transport the drug to the target site in the organism. Biocompatible ferromagnetic particles have been used effectively as potential drug carriers since 1970. The targeting of drug-bearing magnetic particles to a specific part of the body has been achieved by several directions. The one solution is based on the use of a biocompatible polymers such as Polyvinyl alcohol (PVA), dextran, polyethyleneglycol or cyclodextrin [1]. The coating acts to shield the magnetic particle from surrounding environment and can also be functionalized by attaching carboxyl groups, biotin, avidin and so on. The second way consists in encapsulation of both a magnetic materials and a drug in various special matrix materials like albumin, polysaccharide or liposomes [2]. In the next direction, a drug can be grafted directly on the surface of a magnetic carrier without using polymeric binders or encapsulating materials to obtain magnetite-drug complex.
In the present work we give survey of the preparation and characterization of magnetic carriers for three different modes for coupling biologically active substances to magnetic particles, e.g. covalently bound proteins and enzymes to freshly prepared magnetite in the presence of carbodiimide (CDI), the encapsulation nanosized magnetic particles and drugs in lipid mixture dipalmitoyl-phosphatidylcholine (DPPC) and in biodegradable Poly D,L-lactide polymer (PLA), respectively. In case of direct coupling method the present findings clearly show that it is possible to bind proteins (such as Bovine Serum Albumine, Dispase, Chymotrypsine and Streptokinase) onto magnetic particles in the presence of CDI without the aid of a primary coating of freshly prepared magnetic particles. The encapsulation method was used to the study of transmembrane transport of dye Crystal Violet (model of hydrophilic drug) and the study showed the temperature dependence of membrane transport of dye from DPPC liposomes to magneliposomes. Finaly, a poorly water soluble drug Indomethacine (an anti-imflammatory agent) was successfully encapsulated in PLA magnetic nanospheres by spontaneous emulsification solvent diffusion method [3].

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Magnetic target liposomes – hollow nanoparticles for neutron capture NCT, imaging (MRI, PET) and photodynamic X-ray therapy PXT of cancer

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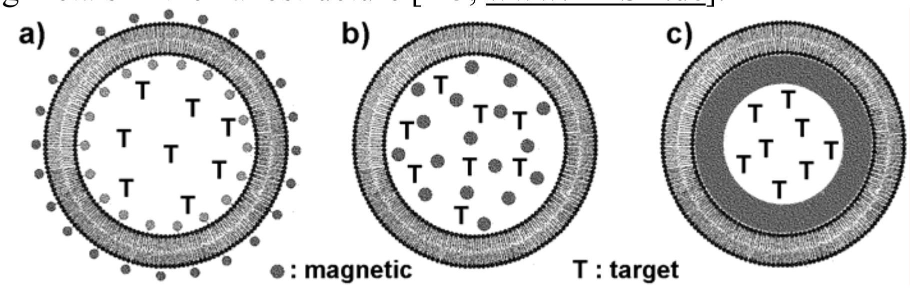
Liposomes are hollow lipid nanoparticles for biomedical applications by their immunotolerance and bio-degradability. While simple liposomes entrapping target material in the lumen have been used as drug carriers and early Neutron therapy applications, problems occurred due to the low bio-life time and local concentration (www.bnct.org). We have obtained a tremendous improvement of the method by introducing metals in the nanostructure [1-3, www.MPSD.de]:



As shown above, the magnetic metal compound was introduced in the metallo-liposomes by three methods: **a)** bound to the lipid layer as metal-head lipid, investigated at DESY-HASYLAB-B1 and ESRF-ID1 [1]; **b)** entrapped in the lumen of the liposomes as soluble complex or sub-nanoparticles; or **c)** in magnetic shell liposomes bearing a lipid - metal oxide double layer shell, as studied at ILL-D22 [2,3]. The structure during synthesis was investigated by ASAXS, time resolved Neutron scattering TR-SANS and electron microscopy TR-EM with our stopped-flow technique [3, 4, www.MPSD.de].

If the metal is applied as a magnetic compound, the method results in magnetic liposomes, which can be concentrated selectively in the body area of interest, e.g. a tumor. Further material can be co-entrapped for therapy, diagnostics, imaging, PET. We were successful in entrapping *water-soluble target* material in the lumen: drugs, metal chelates, Boric acid and (better) the Borates BGB, BBG and BBT [3]. This enables the magnetic liposomes for imaging (MRI, PET), neutron capture therapy (NCT @ B, Gd, ^{7}Li) and photodynamic therapy with Synchroton radiation, with Gd to Lu or cis-Platinum, sources of γ and Auger electrons. Thus magnetic target liposomes can supply a local radiotherapy with secondary radiation of very short range (< 30 µm), i.e. a few cell diameters.

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Evaluation and characterization of biocompatible ferromagnetic fluids for site-specific radiofrequency-induced hyperthermia by nuclear magnetic relaxation measurements

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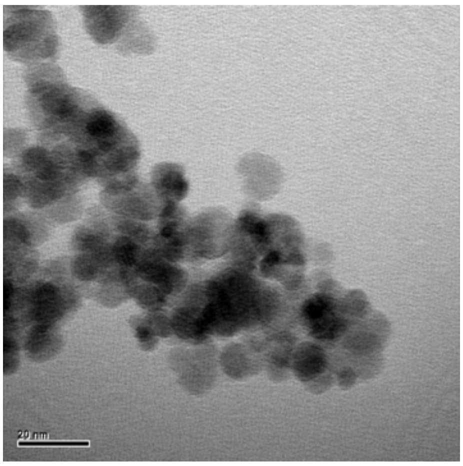
Abstract: Different biocompatible ferromagnetic fluids were prepared and evaluated for application in radiofrequency-induced hyperthermia. Different iron oxide particles with core size in a range from 10nm to 100nm and various polymer matrixes as coatings such as dextran, dextran with carboxylate groups, starch, starch with phosphate groups and Arabic acid in a range from 30nm to 100nm were studied. Dextran coated magnetic nanoparticles with a core size of 10nm (Figure 1) showed promising results concerning the induction of Hyperthermia. Nuclear magnetic relaxation measurement is a valuable tool for the characterization of these super-paramagnetic nanoparticles. T1 and T2 spin-lattice relaxation were been measured for the above samples using the spin echo techique. As the hydrodynamic diameter of the ferrofluids decreases so does the T1 value, an explanation could be that the energy tranfer is greater having smaller and monodisperse in size particles in the media. T2 measurents showed that these specific magnetic nanoparticles can be used also as contrast agents in MRI technique.

Figure 1: TEM image of dextran coated iron oxide (bar scale 20nm).



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Ferrofluid effect on peroxide level in technical vegetables

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The utilization of ferrofluids in life sciences is already a well-known area of research, especially for medical applications. The biological interest in the ferrofluid effect in living organisms (such as vegetables and microorganisms) represents also an important application field, mainly for biotechnological use. The present experimental investigation was focused on the study of peroxide level in young plants intended for technical use (the pumpkin). Water based ferrofluid, stabilized with citric acid was added in various concentrations (10 – 50 – 100 – 150 – 200 – 250 microL/L) in the culture medium of pumpkin plantlets in their early ontogenetic stages. The standard spectrophotometrical assay techniques have been utilized to reveal the activity of some enzymes involved in the biochemical processing of several peroxide substrates. The average values and standard deviations for the enzyme activity of catalase, peroxidase have been graphically represented for ferrofluid samples and control. The statistical signification was assessed by means of t-test for equal size data series corresponding to five repetitions for every control and ferrofluid samples. The discussion was carried out considering first the production of hydrogen peroxide (the substrate of catalase) under the ferrofluid stress, then the putative existence of orto-phenols and orto-quinones etc. that are known as the substrates of peroxidase (besides the hydrogen peroxide). The interpretation has taken into account the fact that the catalase is a good indicator of relatively high peroxide levels while the peroxidase, on the contrary, is able to reveal accurately the low peroxide concentrations. The correlation between the obtained experimental data series was interpreted considering the defense reaction within the young plant organism in the presence of the ferrofluid. The stimulatory effect as well as the inhibitory influence in certain ferrofluid concentrations was finally discussed considering the ferrofluid components.

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"Reactivation" of magnetic nanoparticles by reincubation with carboxymethyl dextrane

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Magnetic nanoparticles coated with carboxymethyl dextrane (CMD) can interact with living cells. This interaction can be modulated by incubation conditions, e.g. time of incubation, incubation buffer or the nature and the specific coating of the magnetic nanoparticles. In addition, the source (supplier, product charge) of the CMD coated nanoparticles influence the interaction with living cells. Another parameter, affecting the interaction is aging of the particles. During long-term storage the nanoparticles alter their ability to interact with tumor cells and leukocytes. We speculate, that this observation might be associated with the integrity of the carbohydrate core of the nanoparticles. Therefore we studied the influence of the CMD core on the differential interaction of magnetic nanoparticles with tumor cells and leukocytes.

Leukocytes were prepared by erythrocyte lysis from whole blood samples of healthy volunteers. The breast cancer cell line MCF-7 was kept under standard cell culture conditions. Magnetic nanoparticles from various suppliers and configurations were applied. Cells were inoculated with these magnetic nanoparticles for distinct time-periods and then separated with MACS. Cell numbers in the flow-through (negative fraction) and the retained fraction (positive fraction) were estimated.

After a storage of 3-5 months at 4 °C nanoparticles showed an increased labeling of living cells within a short time period, but failed to label tumor cells and leukocytes differentially as demonstrated previously. This might be due to a destruction of the carbohydrate core. Therefore, we incubated an aliquot of the nanoparticle samples with carboxymethyl dextran. The reincubated magnetic nanoparticles showed a delayed interaction with living cells comparable to the separation rates during the initial experiments. In detail, the leukocyte nanoparticle interaction was decreased and the tumor cell nanoparticle interaction was unchanged or slightly increased. Magnetic nanoparticles, which showed initially a clear difference in the number of separated tumor cells and leukocytes, exhibited an even more pronounced differential labeling after reincubation. Finally, we analyzed the functional connection between CMD and the nanoparticles. The above mentioned experiments were performed with newly CMD coated nanoparticles. Simple addition of CMD to the reaction buffer did not affect the labeling of the tumor cells or leukocytes with magnetic nanoparticles.

In conclusion, we could show, that a fully established functional CMD core is crucial for differential labelling of different cell types and thus periodical restoration of the CMD core is necessary for optimal cell separation results.

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The potential of the superparamagnetic nanoparticles for the radionuclides separation

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New and innovative methods are of great importance to devise technologies for the separation of radionuclides from nuclear waste or environmental samples. The purpose of this research is to develop a simple, cost-effective and fast separation procedure without production of large waste streams, which combine the simple magnetic separation method with the high selective liquid extraction.

The nano-structured magnetic particles have the high-surface-area and high-adsorption –capacity, which has received considerable attention in recent years in the fields of biomedicine, molecular biology, medical diagnostics, bioinorganic chemistry and catalysis. Using these particles to immobilize metal ions, specially radionuclides on their surfaces exists a high potential.

In the present work, the synthesis, characterization and applications of superparamagnetic polymer nanoparticles were studied for radionuclides separation. The magnetite (Fe_3O_4) with a mean diameter of 8nm was prepared by the coprecipitation of ferrous and ferric salts with NH_4OH. The superparamagnetic Fe_3O_4/polymer microspheres and with the incorporated extraction Tri-n-octylamine (TOA) were synthesized. The morphology and magnetic properties of the particles were examined by transmission electron microscopy (TEM), field emission scanning electron microscopy (SEM) and VSM magnetometer. FTIR infrared spectroscopy showed the changes of chemical composition of initial and incorporated extraction polymer microshperes.

On magnetic field control experiments of ferrofluid convection motion

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Magnetically driven convection in insulating media is actively investigated last years due to uses in the field of materials processing, e.g. crystal growth from protein solution and paramagnetic melts. However, for natural diamagnetic and paramagnetic materials the pondermotive force exerted by a typical magnet on the Earth is insignificant compared to gravity-induced buoyancy one. In that case the colloidal suspensions of small (10 nm) monodomain particles (ferrofluids), which have the susceptibility thousand times higher then ordinary fluids, are very convenient mediums for ground-based modeling of magnetic convection.

The most treatments of ferrofluid convection are taken into account only temperature induced driving buoyancy, magnetic and thermodiffusion mechanisms. On the other hand, our experiments shown that alongside with thermal mechanisms the gravity sedimentation effects can also play the important role in convection of ferrocolloids. The density gradients of solid phase arise due to the settling of magnetic particles and their aggregates in gravity field. Near the onset of convection the competitive action of thermal and concentration density gradients results in oscillatory and traveling wave, mostly spatiotemporally chaotic, regimes.

Experiments were performed to examine the influence of external homogeneous magnetic field on convection instability of mechanical equilibrium and flows in ferrofluid layer heated from one wide side and cooled from another in a range of inclination angles from $\alpha = 0^0$ (heating from below) to 180^0 (heating from above). Integral and local temperature sensors were used for measurement of heat transport across the layer. The visualization of flow patterns was provided by a temperature-sensitive liquid crystal sheet. It is shown that with the help of a magnetic field it is possible to control by the convection stability, intensity of the heat transfer and the form of convection motions. The interaction and the splitting of gravitational and magnetic convection mechanisms for the different orientations of the layer and the magnetic field were studied. A number of non-linear regimes of convection, including localized states and repeated long-wave transients from Rayleigh convection to pure conduction in the case of horizontal layer and to shear flow under inclination were observed.

Thus, the ferrofluid offers a new magnetic field controllable system for the study of so-called spatio-temporal chaos and also non-gravitational - magnetic and thermodiffusion -convection mechanisms.

The research described in this publication was made possible in part by Russian Foundation for Basic Research under grant 04-01-00586 and Award No PE-009-0 of the U.S. Civilian Research & Development Foundation for the Independent States of the Former Soviet Union.

Results in Specific Heat Capacity Determination of Selected Magnetic Fluids Using a Modified Heat Flow Calorimeter

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Magnetofluids are becoming more and more the basis of technical components and actuators. Prior to setting up such components, it is frequently important to know the specific heat capacity of these special suspensions. Therefore, LPA measured the specific heat capacity C_p as a function of the temperature ϑ and the induction flux density B of four different magnetic fluids using a heat flow calorimeter of constant mass. The investigated fluids were 1. MRF 266 (magneto-rheological fluid, mineral-oil-based), 2. MSG-W11 (ferrofluid, water-based), 3. APG 513A (ferrofluid, oil-based) and 4. MTV-MA 093-02 (Cobalt magnetic fluid, kerosene-based). The calorimeter was ready-made equipment with a modified bulb (see Figure 1a). For the measurements it was operated in the temperature range of 20 ... 50°C. As a standard reference material we used pure water. After calibration, the measured value of its specific heat capacity had a deviation of 0.5% from the value found in literature. The specific heat capacity data of the four magnetic fluids showed a slight temperature dependence. To magnetize the liquids we used a water-cooled electromagnet of variable pole pieces distance as shown in Figure 1b.

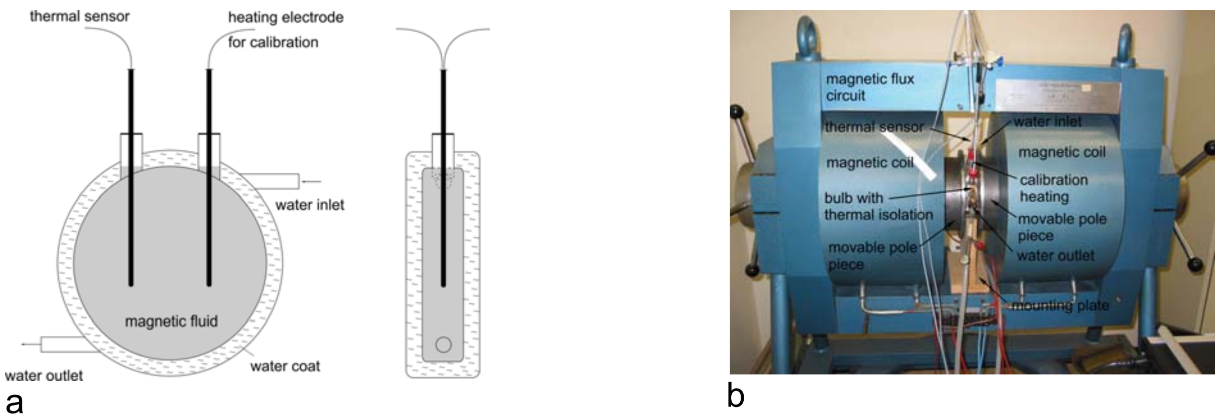

Fig. 1: **a** Special bulb for investigating the influence of magnetic fields on C_p. **b** Total arrangement for determination of the influence of magnetic fields on C_p of magnetofluids.

In the case of fluid 3, no influence of a magnetic field on the specific heat capacity was observed, while C_p of fluid 1 decreased to 84.7% under an applied magnetic field. In contrast, the C_p values for fluids 2 and 4 increased by up to 48.3% when a magnetic field was applied. The measured results are presented and an explanation for the behaviour is given [1].

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Temperature dependent Néel relaxation (TMRX) measurements for particle size distributions of maghemite nanoparticles

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A recently introduced method for the characterisation of magnetic nanoparticles based on the analysis of the temperature dependent Néel relaxation signal (TMRX) has been applied to characterise maghemite particles with different particle size distribution. The probes have been made using an improved magnetic fractionation method for a ferrofluid with a broad size distribution. The temperature range of the measurement set-up has been increased down to 4 K to detect even the smallest particles in the fractions. A mean magnetically relevant particle size has been derived from TMRX and low temperature coercivity measurements, and has been compared to a mean physical size determined by AFM investigations.

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