Low-temperature glassy state of maghemite nanoparticles in magnetic beads

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Magnetic beads *i.e.* polymer capsules of micrometer or sub-micrometer sizes that contain magnetic nanoparticles have recently been used for a variety of biotechnology and medical applications. Most of these applications (including the most popular magnetic separation) benefit from high concentration of magnetic nanoparticles (iron oxide in particular). Additionally, a large range of surface functionalizations allows easy conjugation with target biomolecules and their manipulation.

The magnetic fluid studied in this work was a water based carrier containing 3% volume fraction of the beads with the diameter of ~200 nm [1]. The capsules contained maghemite nanoparticles covered by an oleic acid surfactant. The γ-Fe_2O_3 particles were extremely confined inside the spheres, constituting ~50% of their volume fraction. Measurements of the frequency dependence of the complex magnetic susceptibility, performed at room temperature, have already indicated that the relaxation of this colloid is due to Néel relaxation of the inner maghemite particles and not to Brownian relaxation of the magnetic spheres in water based carrier [1].

In this work the ferrofluid in its frozen state (*i.e.* after solidification of the solvent) was investigated by *dc*-magnetization and *ac*-susceptibility measurements. The results demonstrate that at temperatures below the freezing point of the carrier liquid the sample exhibits a typical superparamagnetic behaviour of individual particles (with zero coercivity and remanence and magnetization curves that can be described by a Langevin function with log-normal distribution of particle diameters). However on further cooling, the relaxation of the ferrofluid cannot be attributed to a blocking process of single particles and polydispersity. The static and dynamic susceptibility measurements indicate that at low temperatures the sample exhibits a glass-like behaviour. Cooling of the sample in a strong magnetic field (5 T) from a temperature above the melting point of the carrier liquid causes an ordering of particles enhancing the susceptibility and changing the relaxation profile of particles. The results indicate that these changes do not arise from a topological reordering of particles confined in capsules but from magnetic ordering induced by freezing in the field, which remain stable when the field is removed. The induced magnetic ordering affects the moment dynamics up to around 200 K, above which a fast transition to the superparamagnetic state occurs.

The glassy state of the frozen suspension results from the aggregated structure of almost close packed maghemite particles in the capsules. Strong dipolar interactions together with randomness in the particle positions and orientations of anisotropy axes, lead to the spin frustration and cooperative spin-glass like dynamics within the magnetic beads.

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