ESRF	**Experiment title:** Hard X-ray RIXS-MCD investigation of Core@Shell@Shell ferrofluids	**Experiment number**: HC-2265
Beamline: ID 26	**Date of experiment**: from: 27-01-2016 to: 02-02-2016 and from: 27-07-2016 to: 29-07-2016 (compensation for shifts lost in the first session)	**Date of report**: 06-03-2018
Shifts: 18	**Local contact(s)**: Mauro Rovezzi	*Received at ESRF:*

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Report:

The goal of this proposal was to study magnetic couplings and local anisotropies of $MnFe_2O_4$@$CoFe_2O_4$@$NiFe_2O_4$ nanospinel ferrofluids (*i.e.*, nanoparticles dispersed in liquid heptane). The originality of these particles stems from the fact that a core of soft magnet ($MnFe_2O_4$) is surrounded by a first shell of hard magnet ($CoFe_2O_4$) covered by another soft magnet shell ($NiFe_2O_4$), which is likely to produce original magnetic properties in this trimagnetic coupled nano-object.

Our initial plan was *(i)* to measure RIXS and RIXS-MCD data at the different metal edges (Mn, Co and Ni), together with element selective magnetization curves, both in the liquid and frozen phases of the ferrofluid, and *(ii)* to compare the results with the data measured on the core@shell $MnFe_2O_4$@$CoFe_2O_4$ precursors and the $NiFe_2O_4$ bare nanoparticles.

In order to preserve particle-particle and solvent-particle interactions in the ferrofluids, we have used the dedicated liquid cell developped in 2014 in collaboration with the Sample Environement group of the ESRF and that we had used in previous experiments (see report of HE-1266). This special sample holder meets the different experimental constraints of RIXS-MCD measurements: *(i)* the sample is in liquid phase at room temperature, *(ii)* the magnetic field (1.5 T) is generated by an electromagnet with only 2 cm between the poles, *(iii)* incident beam is at 45° with respect to sample surface, *(iv)* detection is measured at 90°

on the spectrometer, *(v)* room and low temperature measurements can be done using the same setup in the Helium cooled cryostat.

The circular polarization is generated using a diamond quarter wave plate that has to be inserted in the optical hutch and that is removed after the RIXS-MCD experiment. When the beamtime started on January 27th, the quarter wave plate was not yet installed. Despite the strong commitment of the beamline staff, setting-up the quarter wave plate and doing the commissioning took much more time than expected, and we could start our experiments only on January 29th in the afternoon. Consequently, we did not have time to perform all the measurements that were planned initially: in particular, we could only perform measurements at the Mn and Co edges on the core@shell $MnFe_2O_4$@$CoFe_2O_4$ precursors (*i.e.*, RIXS / RIXS-MCD spectra and element specific magnetization curves, in the liquid and frozen phases, at different tempeartures) but unfortunately there was no time for the core@shell@shell sample. We were granted three extra days of beamtime in July 2016, compensating for the shifts lost in January. Nevertheless, since setting up the RIXS-MCD experiment with the electromagnet and the cryostat requires approximately one full day, we realized that there would not be enough shifts to complete our initial plan. Therefore we decided to focus our study on the comparison of the core@shell $MnFe_2O_4$@$CoFe_2O_4$ ferrofluid with the $MnFe_2O_4$ and $CoFe_2O_4$ references. Thus, we devoted the July shifts to the measurement of these two reference ferofluids (respectively at the Mn and Co edges), in the same experimental conditions as for the $MnFe_2O_4$@$CoFe_2O_4$ measured in January. These measurements were successful, which allowed us to obtain a complete set of data on this bimagnetic core@shell ferrofluid.

The results were published in 2017 in Advanced Materias Interfaces:

N. Daffé, M. Sikora, M. Rovezzi, N. Bouldi, V. Gavrilov, S. Neveu, F. Choueikani, Ph. Ohresser, V. Dupuis, D. Taverna, A. Gloter, M.-A. Arrio, Ph. Sainctavit, and A. Juhin. *Nanoscale distribution of magnetic anisotropies in bimagnetic soft core-hard shell* $MnFe_2O_4$@$CoFe_2O_4$ *nanoparticles.* Advanced Materials Interfaces 4, 1700590 (2017).

Abstract of the paper :

The nanoscale distribution of magnetic anisotropies was measured in core@shell $MnFe_2O_4$@$CoFe_2O_4$ 7.0 nm particles using a combination of element selective magnetic spectroscopies with different probing depths. As this picture is not accessible by any other technique, emergent magnetic properties were revealed. The coercive field is not constant in a whole nanospinel. The very thin (0.5 nm) $CoFe_2O_4$ hard shell imposes a strong magnetic anisotropy to the otherwise very soft $MnFe_2O_4$ core: a large gradient in coercivity was measured inside the $MnFe_2O_4$ core with lower values close to the interface region, while the inner core presents a substantial coercive field (0.54 T) and a very high remnant magnetization (90% of the magnetization at saturation).