

## Magnetic properties of silica coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles using X-ray magnetic circular Dichroism

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### 1 Introduction

Magnetite (Fe<sub>3</sub>O<sub>4</sub>) exhibits high chemical activity, high reactivity with atmospheric oxygen and rapid agglomeration leading to the reduced magnetism and trouble in grafting with other material [1]. In order to overcome such problem and to enhance the efficiency of Fe<sub>3</sub>O<sub>4</sub> as a magnetic beads, we are coating it with inert oxide silica, as it provides a kind of stability and prevents degradation of magnetic nanoparticles (MNPs) [1]. Silica is an ideal material for chemical coating of Fe<sub>3</sub>O<sub>4</sub>, due to its stability and versatility of surface modification. Fe<sub>3</sub>O<sub>4</sub> MNPs have received considerable attention due to its diverse method of synthesis, biocompatibility, non-toxicity, high field irreversibility and environment friendly in nature [2]. The ease of use of magnetic beads with surface coating makes them automation friendly and well suited for the range of applications including the sample separation for next generation sequencing, PCR, Protein purification, molecular and immuno diagnostics, magnetic activated cell sorting and also ease the challenges associated with the extraction of nucleic acid from different kind of sample[1-2]. These magnetic beads are nano-sized particles of Fe<sub>3</sub>O<sub>4</sub> which shows superparamagnetic behaviour with strong magnetic susceptibility, lack of remanent field, coercivity and the absence of hysteresis due to this nature they are intensively studied [1-2].

### 2 Experiment

The nanoparticles coated with silica were prepared by hydrolysis and the polycondensation of the tetraethylorthosilicate (TEOS) with the ethanol solution of TEOS being added to the stable suspension of citric acid coated nanoparticles. The effect of various parameters on the quality of coating were evaluated systematically. The quality of silica layer was depicted using electron microscopy and by performing leaching of the nanoparticles in HCl. While the surface reactivity was examined by grafting (3-aminopropyl) triethoxysilane (APS) onto the nanoparticles. The experimental condition for the coating procedure favours the heterogeneous nucleation of silica on the surface of the nanoparticles, enabled the preparation of very thin silica layers, less than 2nm thick. The decrease of silica layer's thickness to an almost homogeneous layer of Si-OH at the nanoparticles surfaces. The detailed samples

preparation is described somewhere else [2]. The X-ray absorption spectroscopy (XAS) and X-ray magnetic circular dichroism (XMCD) measurements were done at BL-16 of KEK-Photon Factory (PF), Japan. The XAS spectra were taken in the total electron yield (TEY) mode;

### 3 Results and Discussion

We have performed XAS measurements of Silica coated Fe<sub>3</sub>O<sub>4</sub> NPs in the magnetic field  $\pm 3T$ . Here,  $m^+$  and  $m^-$  represent left and right circularly polarized light, respectively which is shown in Fig 1 (a). Fig 1 (b) showed the XMCD spectrum which is the difference of XAS spectra with positive and negative helicity of circular polarized light. The XAS and XMCD spectrum show two main peaks which arise due to spin-orbit interaction. These two peaks are named as  $2p_{3/2}$  ( $L_3$  edge) and  $2p_{1/2}$  ( $L_2$  edge). In the  $2p_{3/2}$  edge of the XMCD spectrum shows three peaks which are at 708.5, 709.6 and 710.3 eV and represent  $O_h$  (Fe<sup>2+</sup>),  $T_d$  (Fe<sup>3+</sup>), and  $O_h$  (Fe<sup>3+</sup>), respectively. The Fe ions are in the mixed state (3+ and 2+ states). In Fe<sub>3</sub>O<sub>4</sub> NPs both sublattices at  $O_h$  sites (Fe<sup>3+</sup> and Fe<sup>2+</sup>) were found to be antiferromagnetically coupled to Fe<sup>3+</sup> ( $T_d$  sites), observing from the opposite signs of the XMCD signals. To calculate magnetic moment, we have used sum rule which suggests that spin magnetic moment ( $m_{spin}$ ) was in the range of  $2.2-2\mu_B/Fe$  and orbital magnetic moment ( $m_{orb}$ )  $0.4-0.3\mu_B/Fe$  in the magnetic field 3T-0.5T. From the data it is clear that ferromagnetism exists in the sample.

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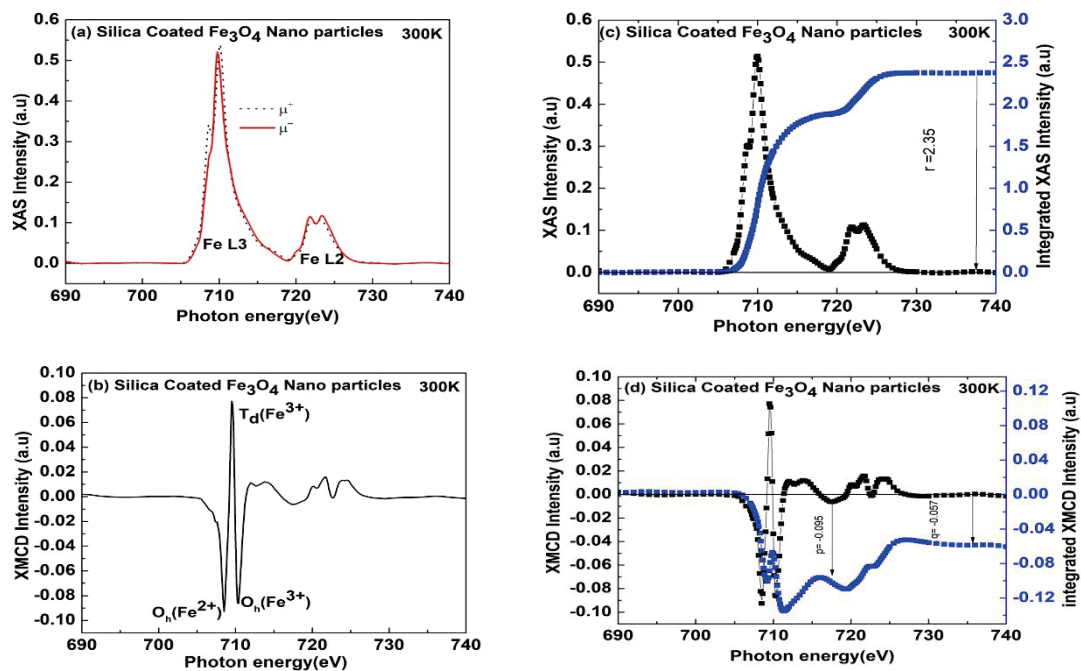


FIG. 1. Fe  $L_{2,3}$ -edge of Silica coated  $\text{Fe}_3\text{O}_4$  magnetic nanoparticles (MNPs) taken in the TEY mode at  $T = 300$  K and  $H = \pm 3$  T. (a) XAS. (b) XMCD spectra (c) integrated XAS and (d) integrated XMCD of silica coated  $\text{Fe}_3\text{O}_4$  MNPs

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