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EAXFS study of Mn and Ni doped maghemite nanoparticles

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Keywords: EXAFS, maghemite, nanoparticles

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Superparamagneticnanoparticles are a group of materials whichhave attracted much attention of researchers in recent years. The reason of that is a wide range of applications, e.g. in medicineas MRI contrast agents or in magnetic hyperthermia, which is very promising for development of modern cancer therapy. They can be used in combination of a targeted delivery of nanoparticle agents, e.g. using monoclonal antibodies, and the Magnetic Resonance Imaging (MRI) detection of localization of these agents. Thus, nanoparticle materials that could play such a dual role are sought.Among them SPIONs (Superparamagnetic Iron Oxide Nanoparticles) are very promising candidates.

For the improvement of theirdetectability e.g. with X-Ray Fluorescence (XRF) and/or XANES (X-ray Absorption Near Edge Structure) spectromicroscopy, the material has been modified by replacement of iron in 10% by manganese and in 10% by nickel. The nanoparticles coated with cationic derivative of chitosan (CCh) were obtained using coprecipitation method. The proper amounts of Fe, Mn and Ni salts were dissolved in aqueous solution of CCh. Nanoparticles were formed upon the addition of ammonia. Finally, the precipitated nanoparticles, were purified by magnetic filtration.

The crystallographic structure of the material obtained has been determined with X-ray diffraction (XRD) and the diffractogram is presented in the Fig. 1.

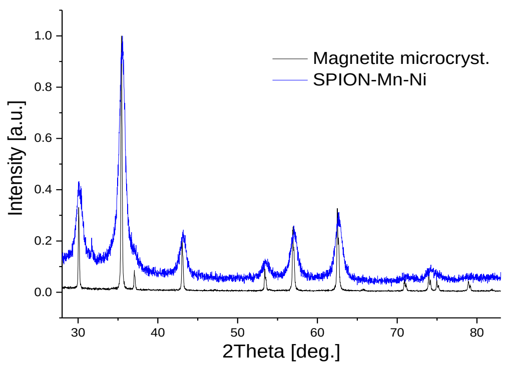

Figure 3. X-Ray diffraction pattern of Mn and Ni doped SPION nanoparticles together with that of microcrystalline magnetite.

It shows, that the material has the same crystallographic structure as magnetite, i.e. of inversed spinel. A large broadening of the diffraction peaks, comparing with magnetite, indicates a nanometric size of the crystallites of the material. The Williamson-Hall analysis of the widths of the peaks provided the information on the density of defects and the average crystallite size, which was estimated at 9 nm.

The Moessbauer measurements carried out at room temperature resulted in a magnetically split, relaxational spectrum, indicating a superparamagnetic character of the nanoparticles. This was also proven by the measurements of a DC magnetic susceptibility and magnetization curves.

The X-ray Absorption measurements were taken at the Swiss Light Source, PSI, Switzerland,at the SuperXAS beamline in the fluorescence mode at room temperature. The EXAFS functions $\chi(R)$ obtained by Fourier transformation of the oscillations above the Fe, Mn and Ni K-edges are shown in the Fig.2.

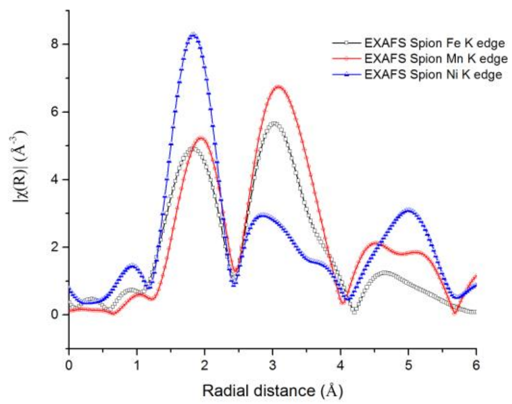

Figure 4. EXAFS functions $\chi(R)$ for the Fe, Mn and Ni K-edges.

The EXAFS functions $\chi(R)$ show a much higher intensity ratio of the 1^{st} neighbor peak (oxygen) to the second neighbor peak (metal) for Ni. This possibly indicates location of Ni at the tetrahedral sites having cation vacancies, appearing at the octahedral sites, in the second neighbor shell. The results are compared with the simulated XAS data and discussed in terms of their relation to the local structure and magnetic properties of the materials.

Acknowledgments: Financial support from National Science Centre, Poland, Project No. 2012/07/B/ST8/03109 is acknowledged. Work partly supported by the Ministry of Science and Higher Education (MNiSW), Poland.

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