

DESY Summer Student Report 2015

Qualitative Study Pair Distribution Function of Magnetic Paper



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Abstract

The aims of this project are to characterize the structure and to study the high quality of pair distribution function of magnetic papers. The magnetic papers were synthesized at various iron salt weight. Diffraction measuring experiments were performed at the P02.1 beamline.



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1 Introduction and scientific background

Magnetic paper is a composite material consists of the cellulose and magnetic compound. Various researches were done with the aim to improve its magnetic remanence and tensile strength. One of the possibilities how to improve the tensile strength is using nata de coco as cellulose source [1]. The magnetic remanence can be tuned by tuning the iron salt concentration for synthesis magnetic compound [2][3].

The magnetic paper in this research was synthesized from nata de coco as paper source due to its free lignin and non-wood origin. The magnetic compound was studied by synthesizing of magnetic papers at different weight of iron salt precursors. Based on the stoichiometry, increasing weight of iron salt precursor was expected to increase the magnetic compound intensity.

One of the best instruments for characterization of material structure is XRD. High-quality XRD data of magnetic compounds were obtained at Hard X-Ray diffraction beam-line P02.1 at PETRA III synchrotron in DESY-Hamburg. From high quality XRD data, the high quality pair distribution function were obtained.

2 Goals of the research projects

Main goals of the proposed project can be formulated as follow:

- characterization of the magnetic compound incorporated into the magnetic paper
- to study the effect of increasing weight of iron salt precursor on the diffraction pattern
- to study the Pair Distribution Function from different resolution source

3 Experimental part

3.1 Sample synthesis

The magnetic paper was synthesized by in-situ magnetic compound synthesis inside cellulose. This method was started by immersing nata de coco hydrogel as paper source into a sodium hydroxide (as a base solution). The procedure followed by removing base hydrogel from base solution and then reacting with iron solution (from $\text{FeCl}_3 \bullet 6\text{H}_2\text{O}$: $\text{Fe}_2\text{SO}_4 \bullet 7\text{H}_2\text{O}$ with weight ratio 1:1). The reaction took place in closed beaker glass with additional heating at 100 °C. The synthesized magnetic gel was crushed into the paper pulp and dried on Teflon pan at 65 °C. The dried paper was washed with distilled water and dried. By described procedure the magnetic paper was cleaned and ready to be measured. In this experiment the weight of total iron salts was varied with the aim to see the influence on its diffraction pattern.

3.2 Diffraction experiment

The X-ray experiment on rolled magnetic papers were carried out at Hard X-Ray Diffraction Beamline P02.1 at PETRA III synchrotron in DESY, Hamburg-Germany. This beamline has fixed energy of 60 keV which correspond to the wavelength 0.20747\AA . The beamsize during the experiment was $0.5 \times 0.5 \text{ mm}^2$. The sample to detector distance (SDD) was roughly 228 and 1028 mm, and then the accurate distance was estimated by the measuring of standard material for powder diffraction CeO_2 . The 2 D diffraction patterns were recorded using Perkin Elmer XRD 1621 detector. The scematic diagram is shown in Figure 1.

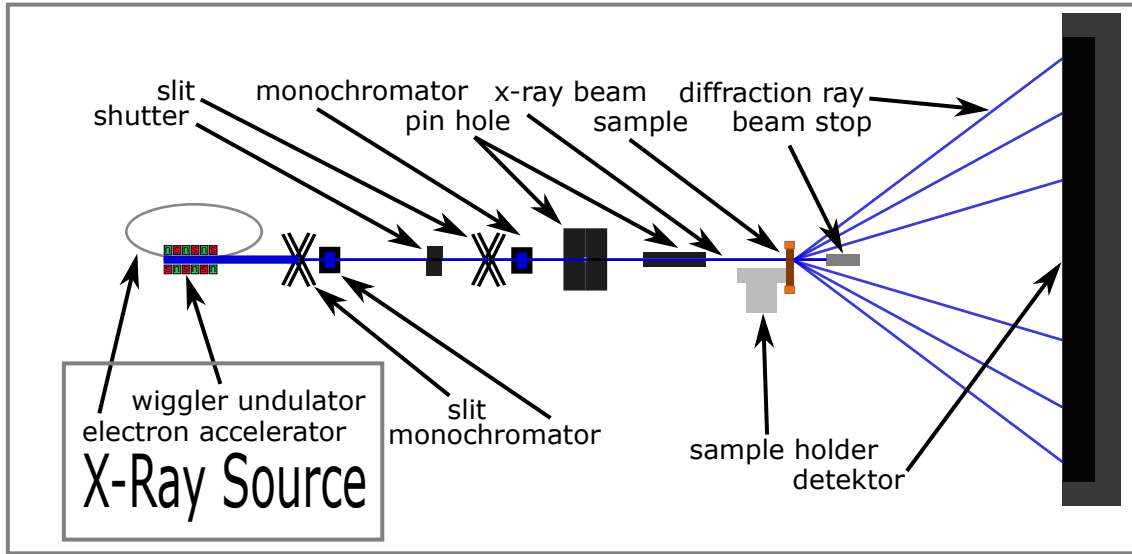


Figure 1: Schematic image of Hard X-Ray Powder Diffraction Beamline setup at PETRA III synchrotron in DESY

3.3 Data processing

The 2D data from detector were integrated to the q-space using the software package FIT2D [4]. The maghemite [8] diffraction pattern for comparison was generated by PowderCell for Windows Version 2.4 [5]. The final images was obtained by Python 2.7 [6] [7].

4 Result and discussion

The magnetic papers were measured by High-Resolution Powder Diffraction. The diffraction patterns for identification magnetic phase were taken by sample to detector distance at 1028 mm. The diffraction patterns are shown in figure 3. The figures show that magnetic paper were made contained many crystalline compound, so the diffraction pattern was treated qualitatively to identify the maghemite phase incorporated in magnetic paper.

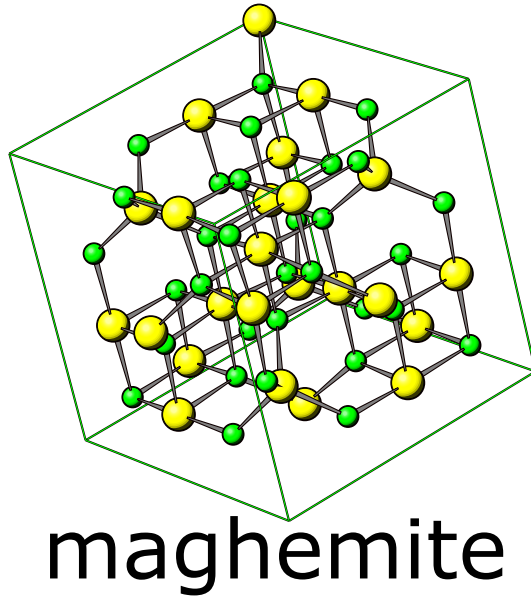


Figure 2: The 3D crystall structure represent of maghemite [8]. The green atom is oxygen and the yellow atom is iron

4.1 Identification of magnetic phase

The diffraction pattern from sample Fe02, Fe04, and Fe06 in figure 3 show that the sample is more fit with maghemite. In Figure 3, the increasing peak at 2.50 and 4.26 in sample Fe02 to Fe06 indicated that the maghemite phase in the magnetic paper was increasing. The increasing peak lead to conclude that increasing weight of iron precursor was increasing the maghemite phase in the magnetic paper.

The diffraction pattern from sample Fe08, Fe10, and Fe12 in figure 3 show that the sample is slightly fit with maghemite. However, the decreasing peak at 2.50 and 4.26 in sample Fe08 to Fe12 indicated that the maghemite phase in the magnetic paper was decreasing. The decreasing peak lead to conclude that increasing weight of iron precursor was decreasing the maghemite phase in the magnetic paper. The other crystalline compounds were also formed in this iron precursor weight range, but the compound couldn't be identified.

The maghemite synthesize in magnetic paper could be predicted. The reaction was following equation: $8 \text{Fe}_{(\text{aq})}^{2+} + 2 \text{Fe}_{(\text{aq})}^{3+} + 11 \text{OH}_{(\text{aq})}^{-} + 2 \text{O}_2 \longrightarrow 5 \text{Fe}_2\text{O}_{3(\text{s})} + 11 \text{H}_{(\text{aq})}^{+}$ and was valid up to sample Fe06. Above sample Fe06, the other reactions were happened and it couldn't be identified.

4.2 Study of magnetic paper using pair distribution function

The magnetic compound identification in the previous subsection is important to obtain high quality pair distribution function in magnetic paper. However, the high quality pair distribution function will be discussed qualitatively due to unidentified peak and

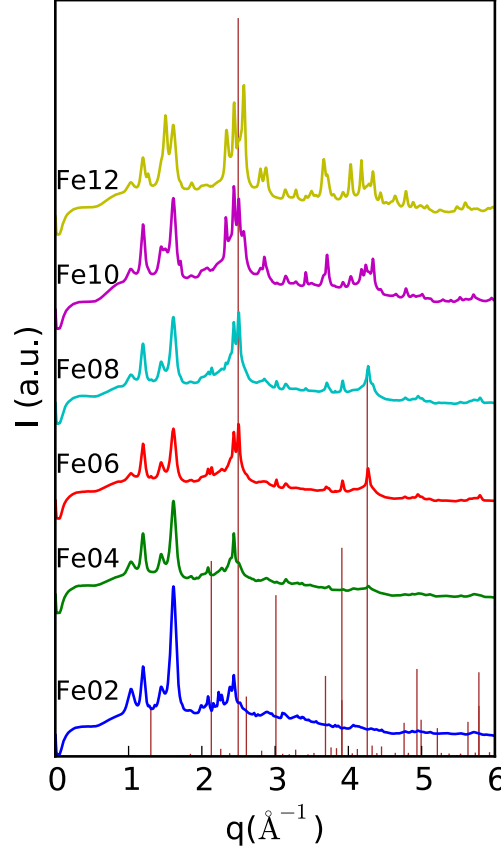


Figure 3: XRD patterns of magnetic paper were obtained from iron salt weight of 0.2 g (Fe02) to 1.2 g (Fe12) with 0.2 g step. The vertical line with brown color in left column and with black color in right column belong to maghemite [8].

elemental composition in magnetic paper. In this section, the study will be focused on the comparison of the diffraction.

The Fe06 sample is chosen as studied material for the comparison of combination and 288 mm SDD due to high content of maghemite. The combination SDD is obtained by joining the multiplied intensity from 1028 mm diffraction pattern as head with the 228 mm as the tail.

The comparison of the structure factor at top left in figure 4 shows that the peak pattern are different in the q below of 10 \AA^{-1} . The peak resolution from combination SDD is better than 228 mm. As a sample at the top right in figure 4, the peak at 2.11 and 2.47 \AA^{-1} in 288 SDD are splitted into two peaks respectively in combination SDD.

The comparison of the pair distribution function at bottom left in figure 4 shows that the last oscillation pattern is different in the tail of distance domain. The last oscillation pattern in combination SDD is longer than in 228 mm SDD. The long tail from combination SDD gives roughly information of the crystall size. However, there is no significant

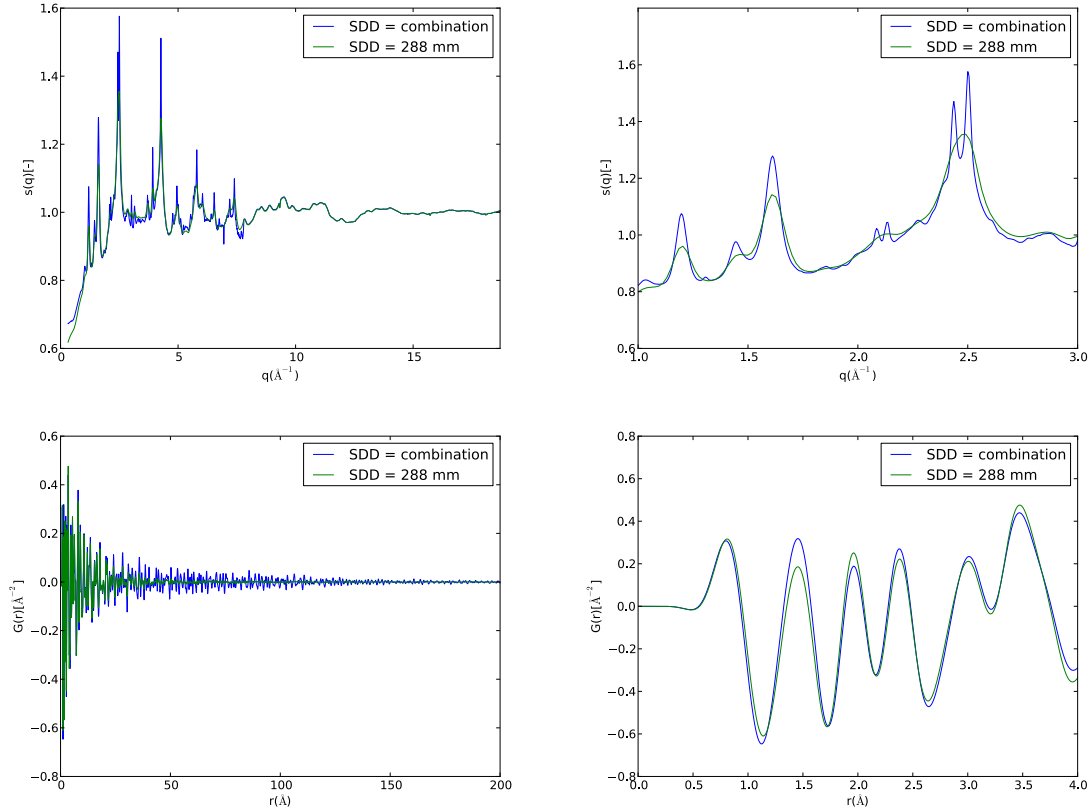


Figure 4: Comparison of combination and 288 mm SDD for structure factor (top) and pair distribution function (bottom)

different pattern in the short distance domain between the combination and 228 mm SDD as presented at bottom right in figure 4.

5 Conclusions

By Hard X-ray Diffraction experiment, the magnetic compound of magnetic papers were identified qualitatively. The magnetic compound was dominated by maghemite. In this experiment, increasing weight of iron precursor up to sample Fe06 were increasing the maghemite phase in magnetic papers. However, the increasing weight above Fe06 lead to another synthesis reaction. In the other words, the best magnetic paper based on the maghemite compound was Fe06. Furthermore, the pair distribution function from combination SDD gives more information about atomic structure in magnetic paper.

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References

- [1] Zheng, J. Yang, W. Zheng, X. Wang, C. Xiang, L. Tang, W. Zhang, S. Chen and H. Wang, *Mater. Sci. Eng.C*, 33 (2013) 2407 - 2412.
- [2] C. Katepetch and R. Rujiravanit, *Carbohydr. Polym.*, 86 (2011) 162 - 170.
- [3] E. Sourty, *Master Thesis: Characterization of Magnetic Nanocomposites Based on Cellulosic Membranes*, McGill University, Quebec, 1997.
- [4] A. Hammersley, [http : //www.esrf.eu/computing/scientific/FIT2D/](http://www.esrf.eu/computing/scientific/FIT2D/)
- [5] W. Kraus and G. Nolze, [http : //www.ccp14.ac.uk/tutorial/powdcell/](http://www.ccp14.ac.uk/tutorial/powdcell/)
- [6] [http : //www.activestate.com/activepython](http://www.activestate.com/activepython)
- [7] J. Hunter, [http : //matplotlib.org/](http://matplotlib.org/)
- [8] Shmakov, A.N., Kryukova, G.N., Tsybulya, S.V., Chuvilin, A.L., and Solov'eva, L.P., ICSD 79196