

# Speciation of Mg-containing Materials by X-ray Absorption Spectroscopy at Mg K-edge

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## 1 Introduction

Magnesium (Mg) is one of the most abundant elements on the earth. The natural abundance of Mg makes it not only a primary component of minerals but also a versatile material with a range of technological applications. The material importance of Mg both in natural science and industry is supported by reliable characterisation of materials. X-ray absorption spectroscopy (XAS) is a unique and powerful analytical tool for materials characterisation. This study aims at characterising a range of Mg-containing materials by XAS in combination with factor analysis (FA), a multivariate analysis method to extract pure matrices from the mixture. More concretely, this study attempts to obtain spectral information of pure Mg-containing components from spectral mixture by combining XAS and FA.

## 2 Experiment

X-ray absorption spectra were collected on the beamline BL-16A, the Photon Factory, KEK. An incident X-ray beam was monochromatised with a variable-included-angle varied-line-spacing plane grating monochromator. The spectra were collected at Mg K-edge (1.305 keV) with a fluorescence mode using a silicon drift detector (SiriusSD, RAYSPEC). The measurement was performed at an ambient temperature (~298 K). Samples for XAS measurement were the pure powders of Mg oxide (MgO), Mg hydroxide (Mg(OH)<sub>2</sub>), and Mg fluoride (MgF<sub>2</sub>). All the samples were purchased from FUJIFILM Wako Pure Chemicals and characterised by powder X-ray diffraction prior to the measurement. The sample powder was ground, spread on a carbon tape, and transferred into a vacuum sample chamber for the measurement. The collected spectra were treated by a standard procedure [1] using a dedicated software WinXAS (Version 3.1) [2]. FA on the collected spectra was performed using a program code ITFA [3]. The procedure of FA on XAS data was detailed in [4].

## 3 Results and Discussion

Shown in Fig. 1 are the X-ray absorption near edge structure (XANES) spectra of the three pure samples (i.e. MgO, Mg(OH)<sub>2</sub>, and MgF<sub>2</sub>). The edge positions and shapes of these spectra are well comparable to those reported in literature [5, 6], indicating that the obtained

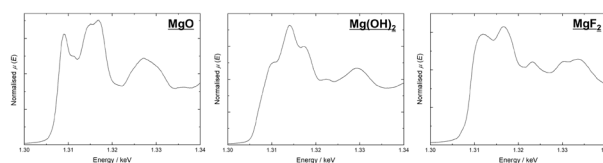


Fig. 1: Mg K-edge XANES spectra of MgO, Mg(OH)<sub>2</sub>, and MgF<sub>2</sub>.

spectra can be used as the reference spectra of the corresponding Mg compounds. The three reference compounds confirmed by XANES (Fig. 1) were further mixed with arbitrary mixing ratios. Five mixed samples were prepared in total and the collected XANES spectra are given in Fig. 2-(a). The spectra were analysed with FA to extract principal components that compose the spectral mixture (i.e. the five spectra in Fig. 2-(a)). The results suggest that the series of the spectra are reasonably interpreted with three principal factors, which is consistent with the fact that the samples were prepared by mixing three compounds. Given this result, the spectra were further treated with FA by assuming three principal factors. The FA-reproduced XANES spectra for the three principal factors and their fraction variations are given in Figs. 2-(b) and -(c), respectively. The XANES spectra of Principal factors 1 and 2 (black and dark-grey data in Fig. 2-(b)) are well comparable to the spectra of Mg(OH)<sub>2</sub> and MgF<sub>2</sub> in Fig. 1, respectively. This indicates that Principal factors 1 and 2 correspond to Mg(OH)<sub>2</sub> and MgF<sub>2</sub>, respectively. Given the fact that the five samples were prepared by mixing MgO, Mg(OH)<sub>2</sub>, and MgF<sub>2</sub>, Principal factor 3 should correspond to MgO, which is not the case. The FA-reproduced XANES spectrum of Principal factor 3 (grey data in Fig. 2-(b)) is inconsistent with the spectrum of MgO (Fig. 1), but it would appear to be rather Mg(OH)<sub>2</sub>. In fact, MgO is transformed into Mg(OH)<sub>2</sub>-like oxyhydroxide compounds by mixing with Mg(OH)<sub>2</sub> and by absorbing ambient moisture [7]. Additionally, the fraction variation of Principal factor 3 (Fig. 2-(c)) is comparable to the ratios of the original MgO in the mixed samples. These results indicate that the combination of XANES and FA can be a powerful tool to investigate the chemical transformation of Mg-containing compounds without reference data. The ongoing study attempts to apply FA to the extended X-ray absorption fine structure (EXAFS) spectra to obtain

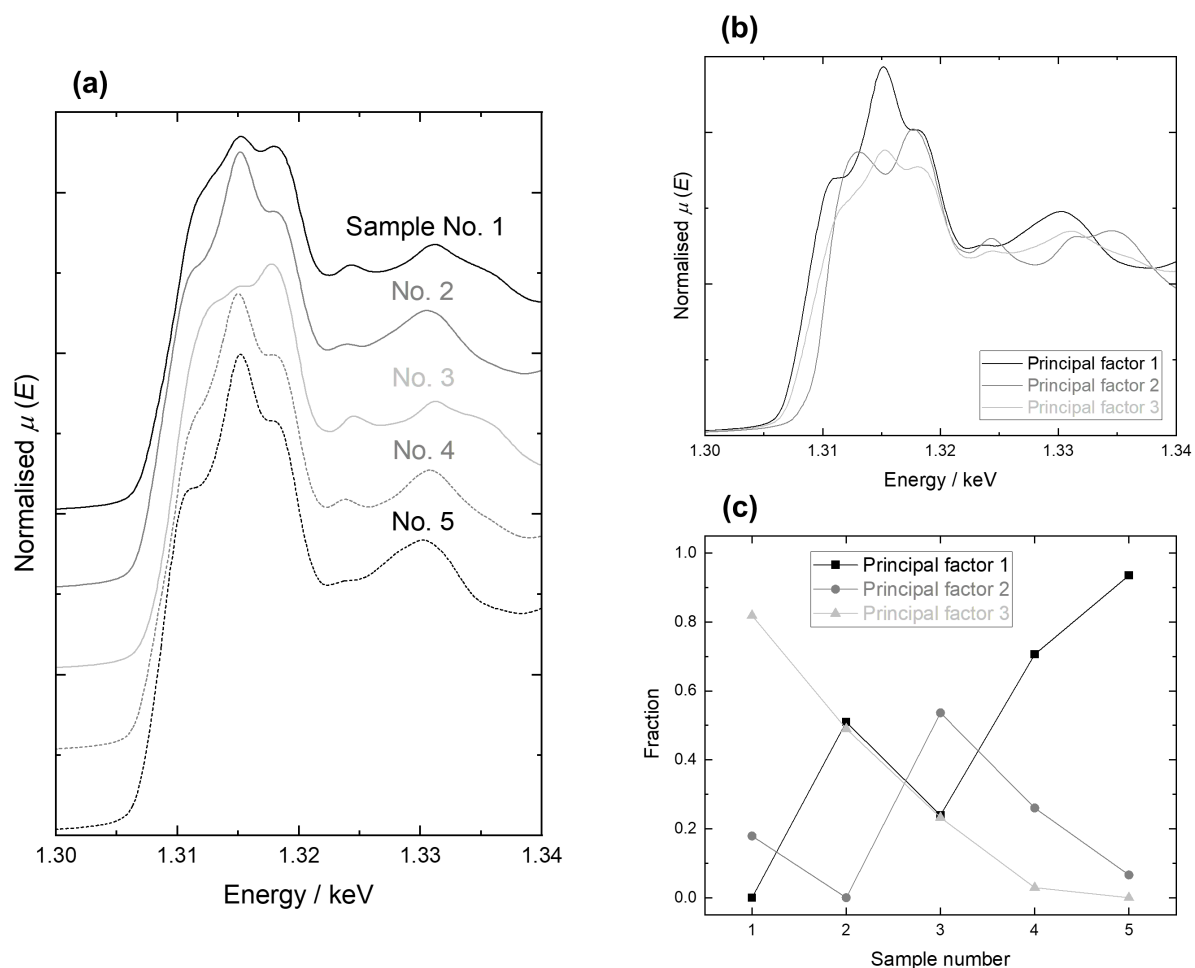


Fig. 2: (a) Mg K-edge XANES spectra of five mixtures composed of MgO, Mg(OH)<sub>2</sub>, and MgF<sub>2</sub> with different mixing ratios, (b) XANES spectra of three principal factors calculated by FA based on the five spectra in (a), and (c) fraction variation of the three principal factors in the five spectra.

comprehensive information on the five mixture samples by combining XANES and EXAFS.

#### Acknowledgement

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#### References

- [1] D. C. Koningsberger, R. Prins, *X-ray Absorption: Principles, Applications, Techniques of EXAFS, SEXAFS and XANES*, John Wiley & Sons (1988).
- [2] T. Ressler, *J. Synchrotron Rad.* **5**, 118 (1998).
- [3] A. Roßberg, T. Reich, G. Bernhard, *Anal. Bioanal. Chem.* **376**, 631 (2003).
- [4] A. Ikeda-Ohno, *Bunseki Kagaku* **73**, 147 (2024).
- [5] K. Nakanishi, T. Ohta, *J. Phys.: Condens. Matter* **21**, 104214 (2009).
- [6] T. Yoshimura, Y. Tamenori, N. Iwasaki, H. Hasegawa, A. Suzuki, H. Kawahara, *J. Synchrotron Rad.* **20**, 734 (2013).

[7] W. Feitknecht, H. Braun, *Helv. Chim. Acta* **50**, 2040 (1967).

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