

## Non-destructive, rapid major element analysis of wet sediments by X-ray microscope, and its application to the Japan Sea sediments

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Understanding of millennial-scale large, abrupt climate changes during the Late Quaternary requires high-resolution quantitative chemical analysis of wet sediment cores. However, conventional XRF method is generally too time consuming and destructive to the samples. The X-ray analytical microscope (XGT) is now used for non-destructive, rapid, high-resolution, quantitative chemical analysis of dry rock samples under non-vacuum condition (Koshikawa, 2002MS). However, its application to quantitative analysis of wet sediments has not been attempted because of the difficulty to evaluate the effect of wet sediment surface and interstitial water.

To explore the possibility of quantitative analysis of wet samples by XGT, the influence of 1) the sample surface conditions and 2) the interstitial water on XRF intensity must be evaluated. In this study, we first examined surface drying effect of wet samples on XRF intensities. The wet samples left in desiccator showed the formation of  $\text{FeSO}_4$  as a result of oxidation of pyrite and many cracks on sample surfaces after 1 and 4 hours. The damaged sample surface conditions caused poor reproducibility of the XRF intensities measured by XGT. The drying effect can be avoided by covering the wet samples with polyethylene-terephthalate (Mylar) film. Although, the XRF intensities decreased by the thin film of water between the wet sample surface and Mylar film, their influence on the XRF intensities could be evaluated for the wet samples, which were covered with Mylar film after the excess water on the surface was removed by leaving the sample in desiccator for 15 to 30 minutes. We also examined the effect of interstitial water on XRF intensities. XRF intensities of major elements decreased with the increase in the water content for all the elements. The relation between the relative XRF intensities (rXRF), which is defined as the XRF intensity of wet samples normalized by that of dry samples, and the water content shows clear negative correlations that basically agrees with the theoretical relationships. The results indicated that correction on the effect of interstitial water is need for XRF intensities of wet samples. The transmitted X-ray intensity index (TXI) can also be measured on the sample by XGT. The TXI and the water content showed clear correlation. These results suggest that the XRF intensities of major elements measured on wet samples by XGT could be corrected for the interstitial water effect, using the water content estimated from TXI based on the relationship between TXI and the rXRF.

We tested the utility of this method by comparing the element concentration calculated from corrected XRF intensities of wet sediment core measured by XGT with major element concentrations measured by ordinary XRF method. The studied core MD01-2407 was taken from Oki-ridge, SW Japan Sea and is composed of silty clay which is calcareous and/or siliceous to various extents.  $20 \times 3 \times 0.68$ cm size slab samples that are continuously sampled from the core were covered with Mylar film after leaving in desiccator for 30 minutes, and  $1.25 \times 1.25$ cm area was measured with 1.25cm steps by XGT. The water content was estimated from TXI for each area. The XRF intensities of each element were correct for the effect of interstitial water using the water content versus rXRF relation. The results showed that the corrected XRF intensities of Al, Si, K, Ca, Ti and Fe agreed well with major element concentration of selected sub samples measured by conventional XRF method. The Ca element concentration showed the large, abrupt, millennial-scale changes. The other element concentrations showed the large millennial-scale changes and clear negative correlations with Ca. These results indicated the applicability of this method to rapid, high-resolution quantitative chemical analysis of wet sediment core samples.