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The Standard-based *f*-ratio Quantitative X-Ray Microanalysis Method for a Field Emission SEM

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A scanning electron microscope (SEM) / energy dispersive spectroscopy (EDS) system has become one general-purpose instrument for quantitative X-ray microanalysis due to its simple and fast operation. With the *k*-ratio method, the standard-based EDS analysis can achieve an accuracy comparable to the electron probe microanalyzer (EPMA) / wavelength-dispersive X-ray spectroscope (WDS), however, standard samples are required for every acquisition and this increases the workload of the quantification [1]. Moreover, the *k*-ratio method requires the samples to be analyzed at an EDS operating condition identical to the standards, which is difficult to be achieved in a field emission SEM, particularly for the electron beam current. Thus, to eliminate the influence of beam current, a quantification method, which is performed based on the routine EDS technique, was developed for a field emission SEM [2].

The quantification method is referred to as the *f*-ratio method and it can be defined using the equation below in a multi-element system,

$$f_i = \frac{I_i}{I_i + \sum_{j \neq i} \Lambda_{i-j} I_j}$$

where I_i represents the net X-ray intensity of element *i*, and Λ_{i-j} represents the calibration factor between any two elements in the unknown sample. The calibration factors are calculated through a combination of the experimental EDS acquisition and the Monte Carlo simulation on a standard sample with known composition. The *f*-ratio method works by varying the composition of the studied system of the unknown sample in the simulations. The theoretical intensities of each constituent with different weight fractions are simulated, and the calibrated *f*-ratios are computed with the calibration factors. In this way, the relationships between the calibrated *f*-ratios of all the constituents and the composition of the studied systems are built. Thus, the composition of the unknown sample can be interpolated by a Python script with the experimental *f*-ratios measured by a routine EDS analysis. The use of the ratio of X-ray intensities from one spectrum enables to cancel the impacts of beam current, and this quantification method has been successfully applied on a multi-element system [3].

In this work, the *f*-ratio method was applied on a mineral kyanite (Al_2SiO_5) with a certificated composition of 33.30 wt.% Al, 17.33 wt.% Si, and 49.37 wt.% O. A set of certificated mineral standards, including albite ($NaAlSi_3O_8$), orthoclase ($KAlSi_3O_8$), and anorthite ($CaAl_2Si_2O_8$), were used to calculate the calibration factors between Al, Si, and O. All the simulations were performed with the MC X-ray software [4]. Figure 1 illustrates the *f*-ratio calibration curve of Al versus Al and Si weight fractions. Figure 2 shows a bar chart of the quantification results computed by the *f*-ratio method, the routine EDS standardless and standard analyses, and the standard-based analysis performed with the DTSA-II software [5,6]. The red dots represent the reference concentrations of the three constitute elements, and the red dash frames

indicates the ranges of their concentrations within an error of 5%. As shown in this figure, except for the EDS standardless analysis (orange bars), all other techniques provide the quantification results within the framed ranges of composition with a satisfying accuracy. Different from the standard-based EDS analysis and the DTSA-II software, the *f*-ratio method can work without measuring the beam current, and thus the whole quantification process is simplified.

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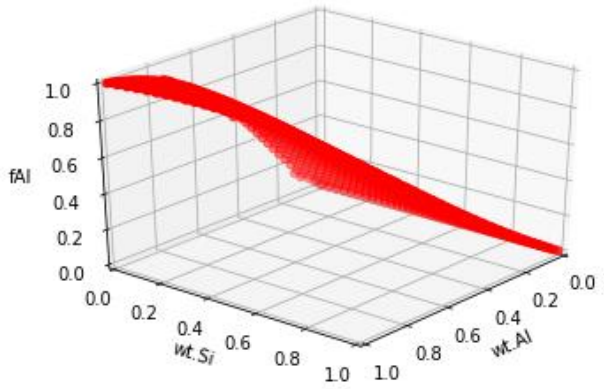

Figure 1. The *f*-ratio calibration curve of Al versus Al and Si weight fractions.

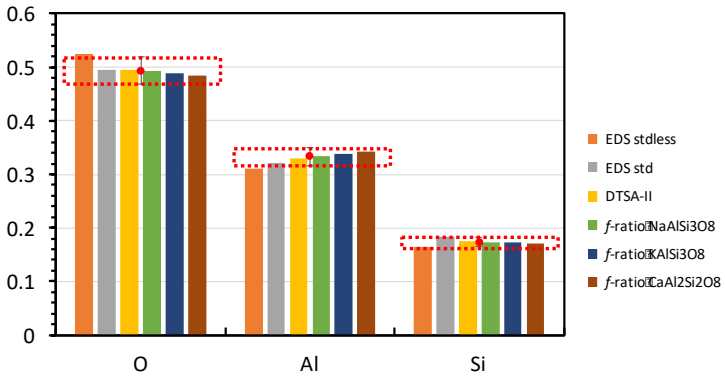

Figure 2. Quantification results of the mineral kyanite with different methods measured with a field emission SEM at an accelerating voltage of 15 kV.

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