Chemical characterization of geomaterials at a nanoscale with TEM-EDS: a comparison between the Standardless, Cliff & Lorimer and Absorption correction quantification methods

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Transmission electron microscopy (TEM) is a well-established technique for the study of geomaterials from the microscale down to the atomic scale. Other than structural and microstructural information, chemical composition at a comparable scale can be obtained. Basically, energy dispersive X-ray spectroscopy (EDS) is the most widespread analytical method installed in TEMs because it is easy to use, fast and of relatively low acquisition cost. In TEM-EDS microanalysis, the A (absorption) and F (secondary fluorescence) corrections are generally omitted because the sample is so thin (tens of nm), and therefore the interaction volume so small, that they are negligible. However, as demonstrated in this work, this is not always the case. Here we describe a method for the calibration of the TEM-EDS system and the application of the absorption correction method (ACM) (van Cappellen & Doukhan, 1994) to the analyses. The results are then compared with those obtained with the standardless method (STL) (Williams & Goldstein, 1991) and the Cliff-Lorimer approximation (CLA) (Cliff & Lorimer, 1975). For the purpose, cordierite, johannsenite, fayalite, spinel, biotite and antigorite, previously characterized with electron micropobe, have been tested. The results show that in all cases the ACM and the CLA give results superior than the STL method. However, whenever one has to deal with dense minerals and/or to work in thick areas of the sample, the ACM gives more consistent results. For instance, with the CLA, the relative abundances of Fe and Si in fayalite (a relatively dense mineral) diverge with increasing thickness, showing a positive slope for Fe and a negative slope for Si – which is an indication of uncorrected absorption – whereas the results for ACM show flat trends for both elements, i.e. their relative abundances with varying thickness are constant. This behaviour is only slightly noticed for Mg and Fe in cordierite (a relatively light mineral) and is not observed at all in antigorite. Furthermore, the possibility to rule out the contribution of absorption to any detected chemical difference, makes the ACM o more confident method for the study of nanoscale zoning, as evidenced in the case of johannsenite, where chemical zoning involving Mn, Fe and Mg were detected.

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