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CHARACTERISATION OF PURE AND MIXED SULPHUR COMPOUNDS BY X-RAY EMISSION SPECTROSCOPY

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ABSTRACT

Sulphur is one of the most important pollutants in the atmosphere, being one of the main elements responsible for acid rain. Since its chemical reactivity depends on the oxidation state, the chemical speciation of sulphur compounds is a clue for the study of environmental contamination[1-3]. To this purpose, Kα and Kβ X-ray emission spectra of several sulphur compounds were studied.
Sulphur spectrum was observed to depend on the oxidation state; for example, in the case of sodium sulphite and sodium sulphate, where the sulphur oxidation state is 4 and 6, respectively. The spectra used in the present work were measured with a scanning electron microscope LEO 1450VP equipped with a wavelength dispersive spectrometer.
Because of the low statistics, the better sensitivity of Kβ peak with the oxidation state could not be used with the required accuracy. Instead, Kα peaks were introduced as a tool for chemical speciation. In order to characterise mixtures with different sulphur compounds, binary samples with different fractions of Na_2SO_3 and Na_2SO_4 were prepared, and the modifications on the spectrum were studied as a function of the S^{IV} and S^{VI} concentrations.
The experimental spectra $I(E)$ were processed obtaining for each mixture i the parameter α_i which minimises a function $\chi^2=\Sigma\{I_i(E)-[\alpha_i\ I_A(E)+(1-\alpha_i)I_B(E)]\}^2$, where the sum is over all the spectral channels analised and the subindexes A and B refer to each of the two compounds. A calibration curve was obtained by plotting the concentration C_A of one of the compounds in the sample as a function of α for all the mixtures prepared. In addition, the dependence of the Kα peak position with the concentration of one of the compounds in the sample was studied. The calibration curve obtained in this second way can be used complementarily to the first method.
Finally, the curves were tested with an "unknown" sample (i.e., a sample not used to obtain the calibration curves). The results show that this method can be used for chemical speciation in this kind of samples and could be extended to different mixtures.

Keywords: X-ray spectroscopy; sulphur oxidation states; electron probe microanalysis

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