Sampling protocol, preparation scheme and error evaluation for SEM-EDS quantitative analysis of asbestos in ophiolitic rocks

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The quantification of asbestos in ophiolitic rocks is of particular importance in the management of soil and rock excavated in civil works and materials from quarry exploitation.

In Italy, a well-described quantitative method is currently available taking advantage of the high resolution of scanning electron microscopy (SEM) and the mineral discrimination provided by energy dispersion spectroscopy (EDS) (Italian Ministerial Decree of 06/09/1994). The method provides a limit of detection of ca. 4-10 ppm and delivers quantitative results for asbestos content higher than 100 ppm. Conversely, a guide for on-field sampling and laboratory sample milling / preparation is still required, to correctly define the quantities of materials of variable geometric dimensions and weight to be sampled following a representative approach. The development of a proper sampling protocol will define the minimum volume of material that is required to correctly represent an asbestos-bearing soil/rock.

The work aims to introduce a structured composite sampling and processing protocol, to reduce data variability and increase sample representativeness for a specified volume of material under investigation.

The protocol is designed to obtain one single aliquot for SEM-EDS quantitative analysis (ca. 10 g) that has all the constituents in the same proportion with a known grade of accuracy and to minimize sample preparation time.

Variability in measured asbestos concentration in ophiolitic rocks between discrete samples is due primarily to the texture of rocks and heterogeneity in the distribution of asbestos. To consider the heterogeneous distribution of asbestos, a simulation of size distribution of the material after laboratory size reduction (crushing and grinding) as a function of operating parameters was obtained. It was studied the influence of some parameters, specifically linked to ophiolitic rocks, such as: particles shape factor, granulometric factor, mineralogical factor, asbestos liberation factor, and maximum particle size on the representativeness of the subsamples.
The methodology provides reasonably unbiased, reproducible estimates of the mean concentration of asbestos in the specified volume of material.