

A micro-analytical SEM-EDS method applied to the quantitative chemical compositions of fibrous amphiboles

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ABSTRACT. — In this work, a new micro-analytical SEM-method to perform quantitative chemical analyses of mineral fibers is described. The method makes it possible to correct the EDS results of fiber analyses, obtained by means of the conventional procedure (i.e., ZAF) utilizing, as compositional reference "standards," small fragments of massive crystals whose composition was previously determined by Electron Microprobe (EMPA). The method reduces the errors in evaluating the chemical composition of fibers due to two principal effects: "absent mass" and "reduced absorption" related to the small size of the fibers. The correct quantitative analyses of the fibers were obtained on the base of: a) the trends (i.e., regression equations) of the element concentrations in the "standard fragments" (as obtained by ZAF correction) versus the fragment sizes; b) the setting of correction factors through the ratio between the true element concentrations (as determined by EPMA in the massive crystal) and the apparent element concentrations revealed by the regression equations; and c) the evaluation of the correction factor errors through the residual dispersions (standard deviation σ) around the curves.

The method was applied for the first time on the fluoro-edenite amphibole fibers found in Biancavilla

area, to obtain quantitative chemical analyses. The unequivocal chemical characterization of this fibrous material is extremely important to verify their effective toxicity in environmental and health issues. The micro-analytical method was also applied to fibers of fluor-rich amphibole to confirm its validity on fibers with higher compositional variability.

RIASSUNTO. — È stato messo a punto un nuovo metodo microanalitico SEM-EDS finalizzato alla determinazione di analisi chimiche quantitative di fibre minerali. Il metodo permette di correggere i risultati delle microanalisi X mediante spettroscopia a dispersione di energia (EDS) ottenuti con le procedure convenzionali (p.e. metodo ZAF). La procedura si basa sull'uso di frammenti di campioni massivi, come standard di riferimento, delle stesse dimensioni delle fibre da analizzare, la cui composizione è esattamente nota.

Il metodo permette di evitare i grossolani errori dovuti ai cosiddetti effetti della "massa mancante" e del "ridotto assorbimento".

I risultati corretti nell'analisi quantitativa delle fibre vengono determinati considerando: a) l'andamento delle concentrazioni apparenti degli elementi (ottenute con la correzione ZAF) nei frammenti dello standard massivo in funzione delle dimensioni dei frammenti stessi; b) la valutazione dei fattori di correzione utilizzando i rapporti fra le concentrazioni effettive

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(note) degli elementi e le concentrazioni apparenti ottenute dalle curve di regressione; c) la valutazione dell'errore di misura sui fattori di correzione tramite la dispersione dei residui intorno alle curve (deviazione standard σ).

Il metodo è stato applicato per la prima volta sulle nuove fibre di anfibolo di fluoro-edenite di Biancavilla, allo scopo di ottenere analisi chimiche quantitative. La composizione chimica inequivocabile di dette fibre è indispensabile per una migliore interpretazione dei risultati sperimentali dei test biologici in corso e per definirne, quindi, l'effettiva tossicità.

Il metodo microanalitico qui proposto è stato inoltre verificato su fibre di anfibolo fluor-richerite al fine di confermarne la validità e la ripetitività anche su fibre con maggiore variabilità composizionale.

KEY WORDS: SEM-EDX micro-analysis, quantitative chemical analyses, EDS standardization, fibrous amphiboles.

INTRODUCTION

The determination of the chemical compositions of mineral fibers (asbestos or not) represents a complex analytical problem that is still unresolved. For the quantitative determinations of the fibrous minerals there are several analytical techniques that present advantages and disadvantages as summarized, for example, in Meeker *et al.* (2003), Gunter *et al.* (2003, 2007) and Gianfagna *et al.* (2007). While a Scanning Electron Microscopy (SEM) is useful for observing the morphology of mineral particles, the EDS systems commonly used on them can provide for only semiquantitative chemical data. The quantitative analysis of fibers and small particles (e.g., less than 5 microns) present many challenges.

In the analysis of larger particles, to calculate the matrix effects (i.e. ZAF method), both the sample and the standard are considered as infinitely thick in comparison with the penetration of the incident electron beam, assuming moreover that both have a smooth and often polished, regular surface (Goldstein *et al.*, 1981; Scott *et al.*, 1995).

Size and shape of fibrous or small particles cannot be controlled, therefore two important effects can drastically change the intensity of the characteristic X-rays generated by the sample

and invalidate the results obtained with routine correction procedures (Small, 2002).

The first effect ("absent mass") is related to the limited particle/fiber sizes and is strongly influenced by the average atomic number of the particle, which determines the probability of elastic scattering and the real volume of the electron beam diffusion inner (or outside) the same particle. Consequently, when a significant fraction of the incident beam goes outside the particle, and no ionization events and X-ray emissions are generated, the intensity of the characteristic X-ray lines cannot be corrected by the Z factor, which takes into consideration, in the ZAF procedure for massive samples, the difference in the average atomic number.

The second effect ("reduced X-Ray absorption") is linked to the particle geometry, which influences both the length of the absorption path within the particle and the X-ray take off angle. The A correction factor, which takes into account differences in the X-ray absorption of the sample and standard, could be radically different, with respect to the factor normally used in the ZAF algorithm.

Many methods, both empirical and based on ideally-shaped particles, have been proposed to solve these problems with EDS microanalysis (Laskin and Cowin, 2001; Ro *et al.*, 2003). However, these methods only obtain semi-quantitative results or they assume, for the analyzed particles, a specific geometry. Thus, for a general application they are only partially useful.

Analysis of fibrous amphiboles

In the Biancavilla area, located at southwest slopes of the Mt. Etna volcano (Sicily, Italy), a new amphibole mineral species was recently found, *fluoro-edenite*, belonging to the amphibole calcic group of the edenite \leftrightarrow fluoro-edenite series (Gianfagna and Oberti, 2001). The mineral presents two different morphological varieties - prismatic and fibrous (Fig. 1), the latter with larger compositional variability, as already reported in the papers of Gianfagna *et al.* (2003), Bruni *et al.* (2006) and Gianfagna *et al.* (2007). For a quantitative evaluation of such chemical differences, fibrous fluoro-edenite samples were studied by means of Scanning Electron Microscopy (SEM) and X-rays Micro-analysis (EDS).

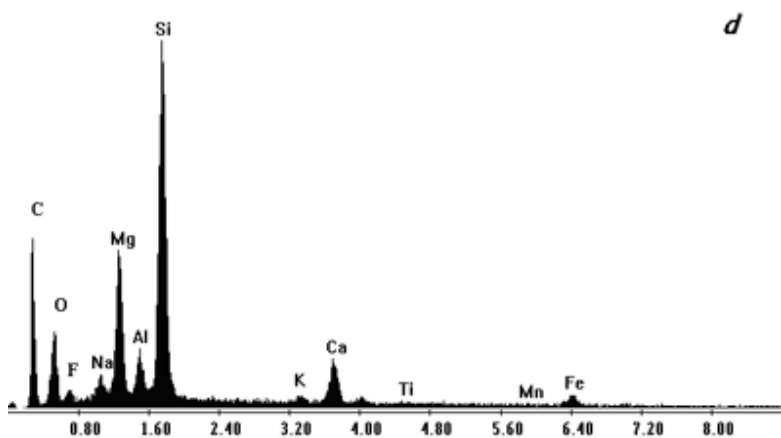
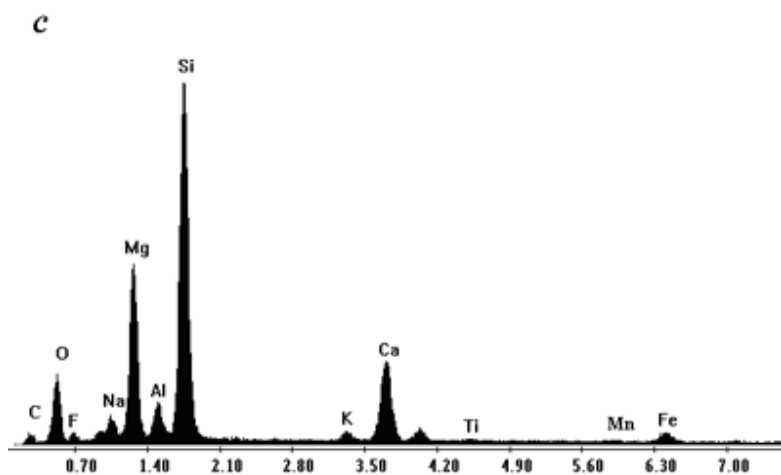
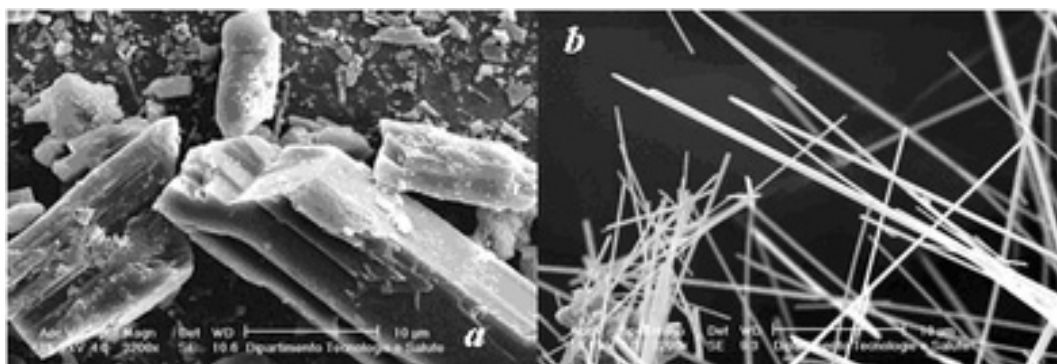


Fig. 1 — a) Prismatic fluoro-edenite; b) fibrous fluoro-edenite; c) EDS spectrum of a prismatic fluoro-edenite crystal; d) EDS spectrum of fibrous fluoro-edenite.

To obviate the problem of the small sizes of the fibers (average diameters between 0.30 and 0.70 μm) in SEM-EDS analysis, an empirical procedure was developed. This procedure is based on the assumption that both the “absent mass” and the “reduced absorption” effects will have, with good approximation, the same significance in the analysis of both the fibers and the fluoro-edenite crystal fragments with similar sizes (0.2 to 1 μm). To evaluate these effects, X-ray spectra of fragments of a prismatic fluoro-edenite crystal were collected and compared with the spectrum obtained by the same original crystal, previously analyzed in Electron Microprobe Analyzer (EPMA).

EXPERIMENTAL METHODS

Sample preparation and observation conditions

Appropriately sized fragments of a fluoro-edenite crystal were obtained by grinding the large prismatic sample in an agate mortar with acetone. These particles were dispersed in doubly-distilled water, mounted on a sample holder (aluminium stub with double adhesive carbon tape) and carbon coated. The size of the fragments were determined in the SEM; the error on the particle size was evaluated in the range of ± 20 nm.

During the EDS analysis of both the small fragments and the large prismatic crystal, the instrumental operating conditions were fixed: 15 keV beam energy, 10 mm WD, tilt angle 0° , spot analysis mode.

RESULTS

Analysis of the prismatic fluoro-edenite fragments

To determine the significance of the “absent mass” and “reduced absorption” effects, the “apparent” concentrations of the different chemical elements in the fragments were calculated on the acquired spectra by the ordinary ZAF procedure, adopting the bulk crystal as standard. Fig. 2 shows the results for thirty-six fragments of the prismatic fluoro-edenite sample, with dimensions between 0.2 and 1 μm . The concentrations of the most abundant five elements (Na, Mg, Si, Ca and Fe),

as obtained by the ZAF procedure, are plotted versus the particle sizes without consideration of the “absent mass” and the “reduced absorption” effects. Moreover, the same figure shows for comparison the actual elemental values (known from the measures on the massive crystal) for each element.

Based on these plots, a relationship between the element apparent concentrations and the particle sizes can be observed. In Fig. 2, the regression curves calculated on the results obtained for the five analyzed elements are also shown. We did not select the type of the regression models to use (e.g., a linear regression), because the experimental results are caused by a combination of several different processes (absent mass, reduced absorption, fragment geometry), which do not show a linear dependence to the fragment size. We thus selected the empirical regression model which yielded the best fit with our data (polynomial, linear, power). Such models were considered the better estimation of the correlation between the apparent concentrations of the elements and the sizes of the fragments.

In Table 1 the correlation coefficients R of the regression equations are reported; moreover the widths of the residual distributions around the relative regression curves (estimated by the standard deviation σ) are given.

The observed dispersions around the average trend (the regression curves) are basically due to two effects:

1. The geometry (shape and orientation) of the standard crystal shards: it causes a random take-off angle which affects the range of the X-ray photon in the sample, adding a random effect on the intensity of the peaks;
2. The compositional variability of the fluoro-edenite shards: the standard prismatic crystal is not compositionally homogeneous so the shards, because their very small size, will show a varying composition; this effect may be particularly evident for the element that presents the greatest substitution in the fluoro-edenite structure.

A third effect contributing to the observed spreading around the average trend is the measurement error on the fragment size; however, it is possible to assume a random distribution of this error with a virtually zero mean value and a width equal to \pm the resolution (instrumental error)

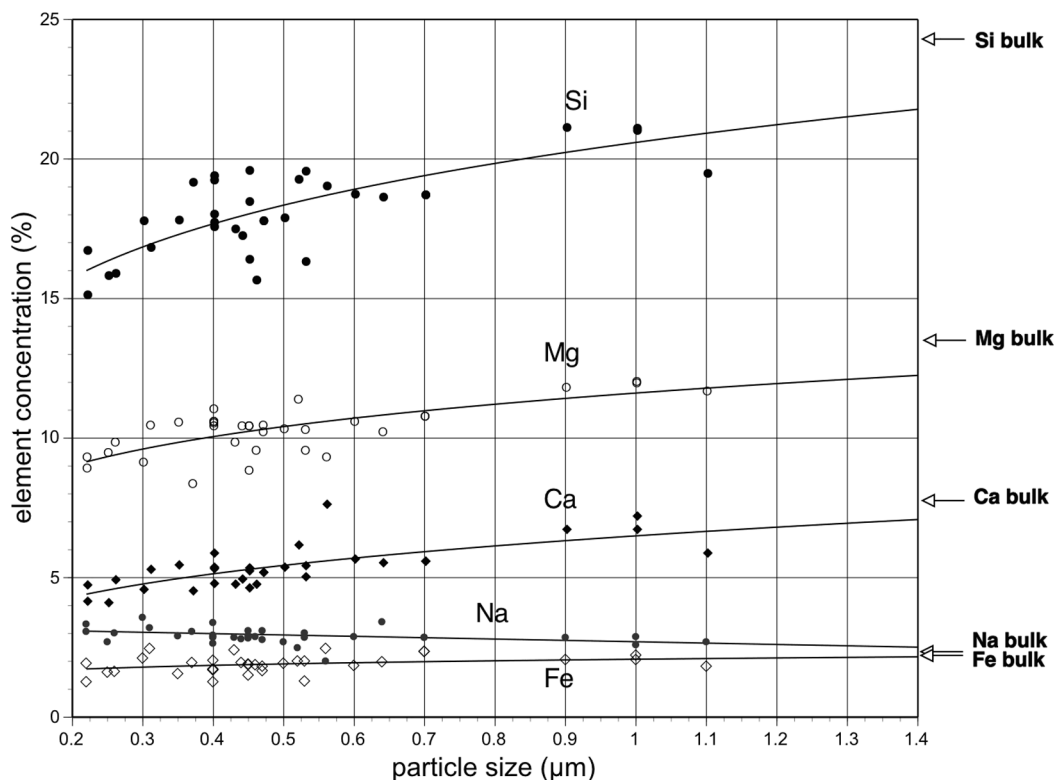


Fig. 2 – Concentrations of the five more abundant elements (Na, Mg, Si, Ca, and Fe) as a function of the particle sizes, using the ZAF procedure.

obtained in the instrumental operative conditions. Thus, the result of this effect on the data average trend (the regression curves) may be disregarded. The result on the widths of the residual distributions

TABLE 1
Correlation coefficients of the regression curves, and dispersions of the residual distributions (estimated by the standard deviation around the curves)

	Na	Mg	Si	Ca	Fe
Correlation coefficients of the regression curves	0.52	0.76	0.81	0.79	0.37
Standard deviation of residuals around the regression curves (σ)	0.135	0.628	1.028	0.539	0.294
Data dispersion (σ) from 14 analyses on a fluoro-edenite bulk sample	0.040	0.135	0.165	0.107	0.085
Contribution of the fluoro-edenite compositional variability to the residual dispersions	30%	22%	16%	20%	29%
Contribution of measurement error on particle sizes to the residual dispersions	6%	19%	19%	18%	5%

may be evaluated (Table 1) taking into account the regression curve slope in the size range of interest (0.2 – 1.1 μm).

An evaluation of the significance of the second effect was attempted in considering the compositional variability of the fragments comparable to the observed variability in the chemical analyses carried out through EPMA technique, on different zones of a prismatic fluoro-edenite crystal. In Table 1 the data dispersions (standard deviation σ) of fourteen EPMA analyses are reported.

Both the contribution (as percentage values) of the fluoro-edenite compositional variability and the contribution of the measurement error on particle sizes to the residual dispersions are shown.

Correction factors for the “apparent” concentrations

The element correction factors were calculated comparing the regression curves of the “apparent” elemental concentrations of the fluoro-edenite fragments with the “true” elemental concentrations of the massive standard sample. In the graphs of Fig. 3 the factors for Si, Mg, Fe and Na, are plotted as a function of the dimensions of the fragments. These were used to correct both the “absent mass” and the “reduced absorption” effects for the fibers. Also shown in Fig. 3 are the correction factor errors, which are assumed equal to the standard error (σ/\sqrt{n}) of the residual dispersion for each regression curve.

These results show that the concentrations of all the elements, except for Na, are underestimated in the fragments analysis, and even more when the fragment size decreases, therefore, the “absent mass” effect is predominant, with the exception of the Na $K\alpha$ line (1.041 KeV) for which the “reduced absorption” effect prevails. For the Fe $K\alpha$ line, with relatively high energy (6.40 KeV), both the “absent mass” and the “reduced absorption” effects appear to cancel each other for particles whose widths are greater than about 0.6 μm . The validity of the calibration curves, which describe the correction factor magnitude, is limited to the dimensional range between 0.2 - 1 μm (i.e., the size ranges for the particles we analyzed).

To obtain the true composition of a fibrous sample, the correction factors, valued by the

calibration curves, will be applied to the “apparent” composition obtained through the EDS spectra (**true compositions = apparent compositions x correction factors**).

The “true” composition of the fiber will be determined through a total relative error [$\Delta C\%$] corresponding to the sum between the correction factor relative error [$\Delta F\%$] and the relative error on the apparent fiber composition [$\Delta\text{apparent}C\%$]:

$$[\Delta C\%] = [\Delta\text{apparent}C\%] + [\Delta F\%]$$

Compositions of the fluoro-edenite fibers

Four fibrous fluoro-edenite samples were analyzed using the above described method. The analyzed fibers were collected in sites named 1, 2, 3, and 4, from the Biancavilla volcanic area, in which the prismatic fluoro-edenite crystals were first discovered. All fibers from the four sites have diameters in the range 0.2 to 1.0 μm .

For each fiber sample (each site), twenty-five fibers were analysed and the corresponding average compositions determined.

The true compositions of each fiber sample, expressed as % of the element, are reported in Table 2, together with the experimental total errors [assumed equal to the standard error (σ/\sqrt{n}) of the data dispersion]. For comparison, the composition of prismatic fluoro-edenite used to make the fragments preparation is also reported. The absence of OH groups for both the prismatic and fiber samples was evaluated by Infra-red Spectroscopy; consequently, the fluorine and chlorine content was fixed to the value of the EPMA analysis. A general chemical characteristic for all analyzed fluoro-edenite fibers is the greater Fe content with respect to the prismatic sample, whereas Al and Ca are generally lower. The compositional correlations between fibrous and prismatic fluoro-edenite samples are reported in Fig. 5, where the “similarities” of the compositions, reported in Table 2 and in Fig. 4 respectively, were taken into account. Fig. 5 shows that the fibers of sample 2 are “distant” from all the other samples, whereas sample 4 and sample 1 are very similar in composition; finally, sample 3 has a composition very close to that of prismatic fluoro-edenite.

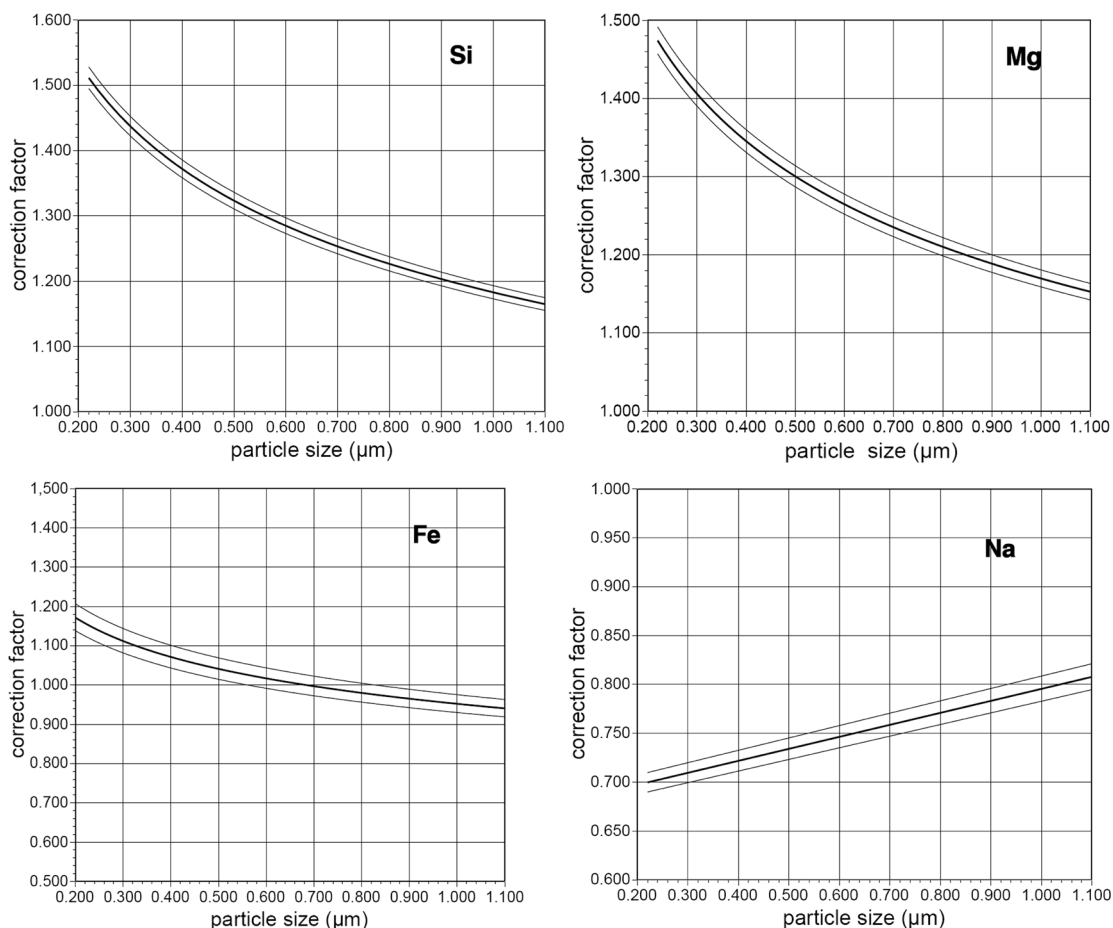


Fig. 3 – Correction factors curves for Si, Mg, Fe and Na, as a function of fiber diameters. There are also reported the correction factor errors assumed equal to the standard error (σ/\sqrt{n}) of the residual dispersion for each regression curve.

Fibrous Richterite

The same procedure used in the analysis of the fibrous fluoro-edenite from Biancavilla was adopted to analyze samples of richterite fibers from Libby, Montana, USA (Meeker *et al.*, 2003; Bandli *et al.*, 2003; Gunter *et al.*, 2003). The richterite amphibole can be found in prismatic as in the fibrous morphology, like the fluoro-edenite amphibole. The richterite fibers analyzed in this work occurred with a diameter range between 0.2 to 1 μm . Unlikely, in the Libby samples (fibrous and not fibrous material) there are not any prismatic crystals so large to use as a reference standard.

Therefore, a prismatic richterite crystal from the Ontario, Canada, was obtained from the collection of the Mineralogical Museum of the Earth Sciences Department, “Sapienza Università di Roma”, Italy. The crystal, whose composition was previously and carefully determined by means of EPMA technique, was used as reference standard. On the basis of its composition, the massive crystal was defined as a fluor-richterite amphibole.

The crystal was **appropriately ground to obtain** fragments with suitable sizes. The acquired EDS spectra of the fragments were utilized to calculate the “apparent” concentrations of the different elements, using the ZAF procedure. The calibration

TABLE 2

Averaged compositions (% of elements) for the four fluoro-edenite fibers, obtained on about 25 analyses for each sample, compared to the prismatic standard. The experimental error is shown in brackets

Sites	1	2	3	4	Prismatic Fluoro-edenite
# of analyses	25	26	26	26	14
Na % ± [er]	2.33 ± [0.335]	1.69 ± [0.285]	2.37 ± [0.336]	2.1 ± [0.411]	1.08
Mg % ± [er]	12.49 ± [0.717]	12.00 ± [0.653]	13.41 ± [0.687]	12.89 ± [0.662]	13.53
Al % ± [er]	1.16 ± [0.247]	1.59 ± [0.225]	1.40 ± [0.262]	1.22 ± [0.249]	0.96
Si % ± [er]	25.07 ± [0.988]	24.45 ± [0.942]	24.89 ± [0.726]	25.2 ± [0.719]	24.26
Ca % ± [er]	6.01 ± [0.621]	7.31 ± [0.592]	6.50 ± [0.658]	5.93 ± [0.609]	7.72
K % ± [er]	0.66 ± [0.291]	0.58 ± [0.026]	0.61 ± [0.201]	0.68 ± [0.141]	0.35
Ti % ± [er]	0.08 ± [0.040]	0.073 ± [0.021]	0.05 ± [0.024]	0.07 ± [0.030]	0.012
Mn % ± [er]	0.73 ± [0.286]	0.57 ± [0.140]	0.60 ± [0.191]	0.63 ± [0.292]	0.32
Fe % ± [er]	4.40 ± [0.594]	4.85 ± [0.546]	2.75 ± [0.621]	3.95 ± [0.203]	1.99

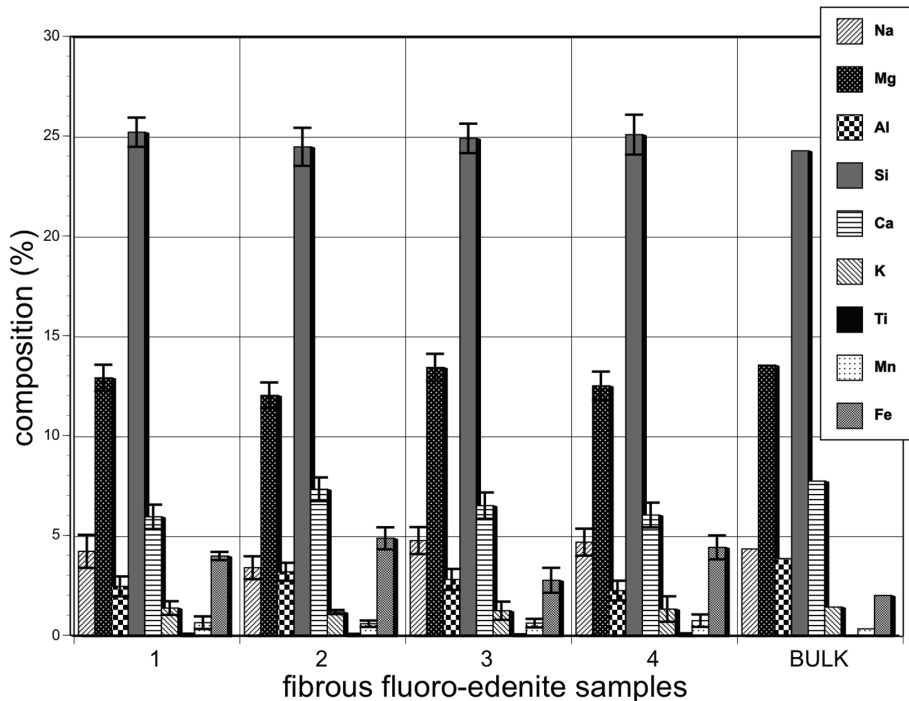


Fig. 4 – Compositions of the four fibrous fluoro-edenite samples as compared to the prismatic sample (data in Table 2). The experimental error is also plotted at the end of each bar.

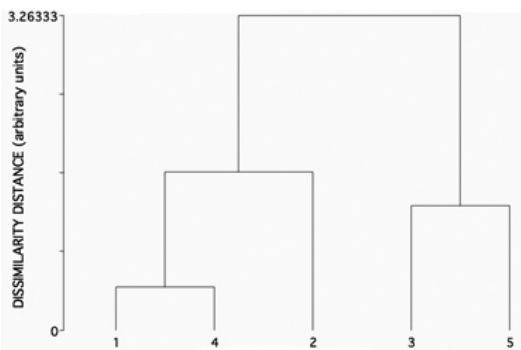


Fig. 5 – Compositional correlations between fibrous (1, 2, 3, and 4) and prismatic fluoro-edenite (5) samples.

curves were utilized to correct both the “absent mass” and the “reduced absorption” effects.

In Table 3, the average chemical composition of the richterite fibers, based on the SEM-EDS analysis (on 22 fibers), is shown along with the mean composition of the prismatic richterite (20 analyses).

For comparison, Fig. 6 shows the composition of massive richterite, used as a reference standard, and the mean composition of fibers from Libby.

TABLE 3

Average chemical composition of the richterite fibers by SEM-EDS (the experimental error assumed equal to the standard error is shown), compared with the mean composition of the prismatic richterite and the bundled richterite fibers (EMPA)

	Prismatic crystal (EMPA)	Bundled fibers (EMPA)	Single fibers (SEM-EDS)	Experimental error on fiber compositions
# analyses	20	22	22	
Si	26.53	27.62	27.4	1.548
Ti	0.21	0.05	0.14	0.06
Al	0.49	0.07	0.16	0.058
Mg	14.1	13.44	13.9	0.725
Ca	6.59	5.22	4.69	0.658
Mn	0.15	0.11	0.08	0.021
Fe	1.81	3.26	3.11	0.409
Na	1.7	1.87	1.84	0.338
K	0.56	0.66	0.77	0.264

It is interesting to note that the composition of the massive richterite shows significant chemical

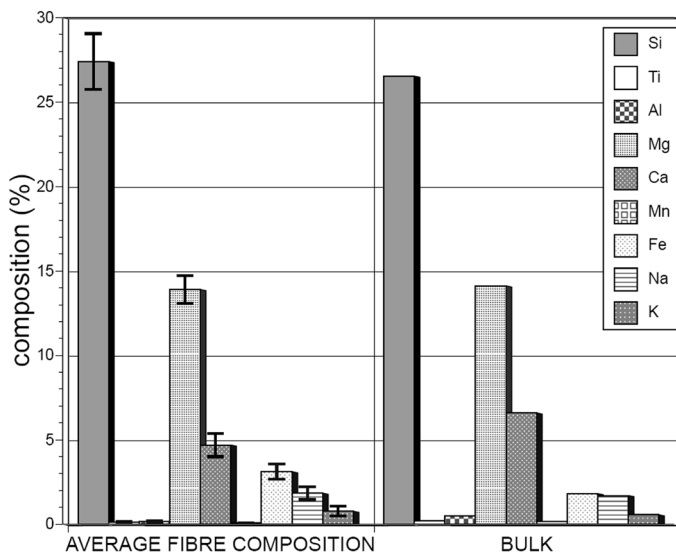


Fig. 6 – Composition of the richterite fibers from Libby, Montana, compared with that of the massive crystal. The experimental error is also plotted.

differences with respect to the analyzed fibers of the same mineralogical species, while the fluoro-edenite fibers present minor compositional variability respect to the prismatic fluoro-edenite, utilized as reference standard (see Table 2 and Fig. 4).

To test the validity of the method also when a reference standard is used with some chemical variabilities with respect to the fibrous variety, a bundle of richterite fibers were prepared in thin section, embedded in resin, to simulate a massive sample, and were analyzed by EPMA technique. In Table 3, the EPMA analyses for the richterite bundle fibers are reported. It is significant that a very strict agreement occurs between the composition of the bundled fibers, determined by EPMA, and the composition of the single fibers, determined by the SEM-EDS procedure.

DISCUSSION AND CONCLUSION

The characterization of fibrous materials is very difficult because of their small sizes (i.e., the fiber width). Careful analyses are frequently hindered and sometimes structural determinations are impossible. Therefore, in these particular cases

it is important to develop innovative methods to characterize, identify, and correctly classify these materials.

Quantitative micro-analyses of particles and/or fibers, and their subsequent interpretability, are very difficult to perform on samples with dimensions in the order of micron or sub-micron range, particularly in presence of low elemental concentrations.

The experimental X-rays micro-analytical method here described overcomes the errors due to so-called "absent mass" and "reduced absorption" effects in the quantitative analysis of sub-micron mineral fibers.

This new method was developed and applied on fibers with dimensional range between 0.2 and 1.0 μm , and a compositional reference "standard" was available from which small fragments, with size comparable to the fiber, was possible to obtain.

The method was tested and applied for the first time on the new fluoro-edenite amphibole fibers from Biancavilla, Italy, and it was verified on fluor-richterite fibers from Libby, Montana, USA. The method can be also used for fibrous samples with slightly different elemental concentrations with respect to the massive standard crystal.

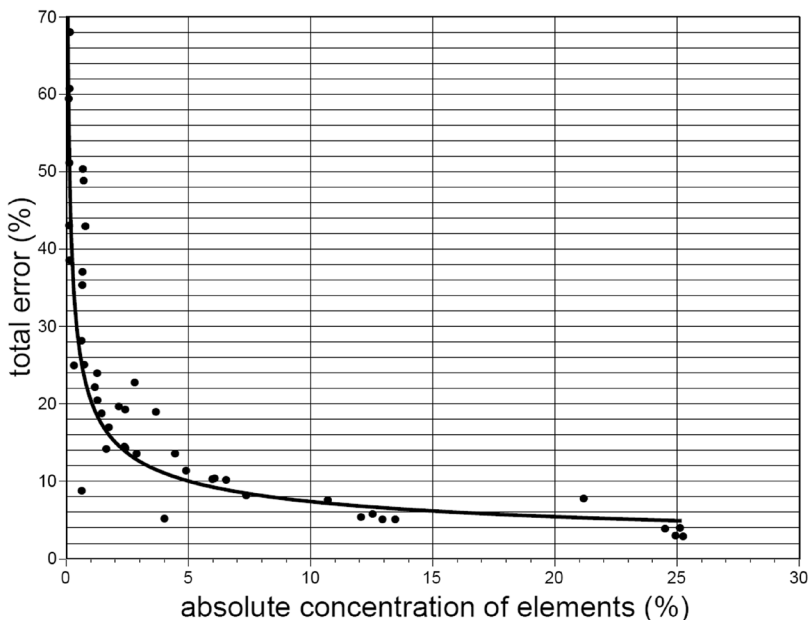


Fig. 7 – Trend of the total error (%) as a function of the absolute concentration of the measured element in a fibrous sample.

Concerning the influence of the elemental abundances in the fibrous samples, the error (%) increases with the decrease of the relative contents (Fig. 7). This was verified in both the cases considered in the present study. With this methodological procedure a measured error not greater than 5% can be expected when the element is present in concentration not less than approximately 10%. The reliability of the procedure rapidly decreases when the abundance of the elements is below 3 to 4%.

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