



## Assessment of the potential hazard represented by natural raw materials containing mineral fibres—The case of the feldspar from Orani, Sardinia (Italy)



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### ABSTRACT

This work describes the nature of the potentially hazardous fibrous amphibole found in the Orani's feldspar mine (Sardinia, Italy). To identify its nature, a protocol of analysis including morphometric, chemical and crystallographic characterizations was applied. Thanks to this approach, it was possible to classify the observed fibres as tremolite after comparing chemical data, SEM/TEM observations, FTIR/ Raman spectra and X-ray diffraction data with those reported for a standard sample. The unit cell parameters of the investigated tremolite phase are  $a = 9.82(1) \text{ \AA}$ ,  $b = 18.08(3) \text{ \AA}$ ,  $c = 5.27(1) \text{ \AA}$ , and the angle  $\beta$  corresponds to  $104.4(1)^\circ$ . The mean concentration of asbestos tremolite in the Orani's feldspar is 0.28 wt%. Most of the fibres (0.26 wt%) are respirable 'regulated' fibres, representing a potential hazard. Because the total amount of tremolite in the sample is 0.6 wt%, a large fraction of it has a crystal habit other than fibrous-asbestiform or acicular. The obtained results allowed us to suggest possible solutions for a safe exploitation and mineral processing of the Orani's mine. The procedure proposed herein may be a general tool suitable to identify the mineralogical nature of fibrous minerals in raw materials and assess if they may represent a potential health/environmental hazard.

### 1. Introduction

The family of asbestos minerals includes chrysotile (serpentine asbestos) and the amphibole minerals actinolite asbestos, amosite (fibrous asbestiform variety of grunerite), anthophyllite asbestos, crocidolite (fibrous asbestiform variety of riebeckite), and tremolite asbestos [1–3]. A universal ban exists for amphibole asbestos whereas chrysotile asbestos, despite its inclusion by the International Agency for Research on Cancer (IARC) in Group 1 "substance carcinogenic to humans" [4–6], can still be used "in a safe mode". Length (L) and diameter (D) of asbestos fibres play a key role in the toxicity and carcinogenic potential [7,8]. Fibres with  $L < 5 \mu\text{m}$  are cleared from the lungs with similar kinetics and mechanisms as isomorphous particles [9] and hence should not be considered hazardous. For longer fibres, the 'Stanton hypothesis' [10] predicts that the optimum morphology for induction of intrapleural tumours in rats is  $D \leq 0.25 \mu\text{m}$  and  $L > 8 \mu\text{m}$  as fibres with

$L > 8 \mu\text{m}$  cannot be eliminated by phagocytic cells like macrophages [11], leading to a process known as frustrated phagocytosis. Pott [12] and Spurny et al. [13] found that asbestos fibres with most striking effects on mesothelioma development in rodents had  $L \geq 10 \mu\text{m}$  and  $D \leq 0.25 \mu\text{m}$  or  $L \geq 5 \mu\text{m}$  and  $D \leq 0.25 \mu\text{m}$ , respectively. Lippmann claimed that mesothelioma is caused by biopersistent fibres  $< 0.1 \mu\text{m}$  and  $> 5 \mu\text{m}$  whereas excess lung cancers and pulmonary fibrosis occur with fibres  $> 20 \mu\text{m}$  [14]. Fibres with  $5 < L < 20 \mu\text{m}$  represent the transition range between those fibres that are cleared and those fibres (with  $L > 20 \mu\text{m}$ ) that macrophages cannot fully phagocytised [15]. From a regulatory standpoint, the World Health Organization indicated the following counting criteria to classify (regulated) respirable fibres:  $L \geq 5 \mu\text{m}$ , width  $\leq 3 \mu\text{m}$ ,  $L/\text{width}$  (aspect) ratio  $\geq 3:1$  [16].

Restrictions to the mining activity concern not only the mines of asbestos raw minerals but also the mines of natural raw materials in areas where asbestos is present. In the latter case, operations should

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eventually be carried out under severe monitoring and differential processing. Unfortunately, regulation of the exploitation activity in mining areas associated with asbestos is missing worldwide with the exception of New Caledonia where the exploitation of serpentinite rocks contaminated by chrysotile, fibrous tremolite and fibrous antigorite [17] is strictly regulated [18]. Another example is represented by the case of the Libby mine (Montana, USA) where vermiculite contaminated with amphibole asbestos has been mined and processed for approximately 70 years, until 1990 [19–22].

In general, for the cases described above, if a preliminary inspection using optical and/or electron microscopy evidences particles with apparent fibrous shape (indicatively, particles with an aspect ratio > 3:1) in the raw material, a thorough investigation should embrace:

- (1) mineralogical characterization of the particles with apparent fibrous shape in order to verify if their mineralogy is compatible with that of the minerals forming the asbestos family (serpentine, actinolite, anthophyllite, grunerite, riebeckite, tremolite);
- (2) if the mineralogy of the investigated particles is compatible with that of a mineral from the asbestos family, it should be determined whether it is actually ‘asbestos’ or not. This task is apparently very simple but turns out to be a major source of confusion because to date there is no unequivocal and conclusive definition of ‘asbestos’ [23–27], with the ‘mineralogical’ and ‘regulatory’ definitions that sometimes contradict one another [23]. The section *Supplementary Material 1* reports a critical review of the existing definitions of ‘asbestos’. To circumvent the open issue on the definition of ‘asbestos’, the strategy proposed here was to compare the investigated particles with apparent fibrous shape to standard samples of asbestos (e.g. tremolite asbestos in this specific case).
- (3) if the particles with apparent fibrous shape are classified as ‘asbestos’ by comparison with known standards, their concentration in the raw material must be determined;
- (4) on the basis of the morphometric parameters, the potential hazard of the asbestos phase according to the existing regulation needs to be assessed;
- (5) on the basis of all the information collected in the previous steps, eventual legal restrictions for the use of this raw material needs to be assessed.

This analytical protocol requires the use of various experimental techniques.

In this study, we report the case of a natural feldspar raw material from Orani (Nuoro, Sardinia, Italy) that was mined since 1970. The presence of asbestos like particles in the mine was discovered in 2015 (see Section 2.1). The aims of this work are: (i) to apply the protocol of analysis described above to this case so to accomplish an unquestionable mineralogical and regulatory classification of the fibrous mineral phase; (ii) to objectively point out advantages and disadvantages of the applied protocol in view of a general application to other raw materials (such as erionite rich zeolitites); (iii) to assess if the feldspar from Orani represents a potential hazard; (iv) to investigate the mineralogical association of the raw material so to recommend solutions to the specific problem.

## 2. Experimental

### 2.1. The Orani’s case

From preliminary examinations, the feldspar from the Orani’s mine in Sardinia turned out to be an example of a raw material which contains amphibole asbestos as a natural contaminant. The mining activities at the Orani’s mine started in the 1970’s but massive exploitation began in 1990. In the processing plant of the mine, feldspar rich rocks underwent primary and secondary crushing and were subsequently mixed with quartz rich sands [28]. This material was then sent to a

productive site in Central Italy for fine milling with a mean final production of 60,000 t/y. The final product was marketed and distributed in many production sites of Central Northern Italy for the manufacture of traditional ceramics [29], glazes and sanitary ware. Prompted by the owners of the production plant, a monitoring activity revealed a concentration of regulated tremolite fibres of about 100(15) ff/l in the proximity of the grinding, mixing and bagging machinery [28]. Based on diffraction, optical and electron microscopy studies, these mineral fibres were classified as tremolite asbestos [28], although no detailed mineralogical crystallographic investigation was conducted prior to the current study. Following this discovery, because the Italian Law N. 257, 1992 [30] bans the mining, import export, marketing and production of asbestos (including tremolite asbestos) or asbestos containing materials, the Orani’s mine was impounded by a state’s attorney in the late 2015, with the suspension of its mining activity and prohibited access until the end of the legal action aimed at assessing, among other factors, the nature of the fibrous mineral phase.

### 2.2. Description of investigated area and sample collection

The geology of the investigated mining site is described in *Supplementary Material 2*.

In this study we have investigated samples collected around the mining area to determine the mineralogical phase compositions. A product labelled MM, composed of plagioclase, quartz, mica and minor phases possibly including the asbestos like phase, was also characterised. In the past, this feldspar was ground, mixed with other quartz rich grades, packed in bags and sold as raw material to various manufacturing plants in Italy (see above) for the production of traditional ceramics [28]. The sampling points are geo localized in Fig. 1. The list of samples is reported in Table 1. Preliminary screening using X ray diffraction confirmed the presence of an amphibole phase in samples 1, 4 and MM. Both the powder sample and single fibre bundles removed from the MM sample using optical microscopy were studied. The full mineralogical chemical characterization was conducted using the experimental techniques indicated by the current Italian regulations for massive materials (optical microscopy, scanning electron microscopy (SEM), X ray powder diffraction (XRPD) [31]), and infra red spectroscopy (IR). Given that these validated methods do not allow to classify unequivocally the mineralogical nature of the fibres present in the sample (e.g. to discriminate between tremolite and winchite or richterite), complementary techniques such as Rietveld quantitative phase analysis, micro Raman spectroscopy, high resolution transmission electron microscopy (HR TEM) and electron micro probe analysis (EMPA) were used to this aim. The optical, structural, microstructural and morphometric parameters of the investigated mineral fibres were compared to those of the tremolite asbestos from Saltworks Mine, Death Valley, San Bernardino County, California, U.S.A. provided by the UK Health and safety Executive (HSE) [32,33].

### 2.3. Methods

A detailed description of the methods is reported as *Supplementary Material 2*. Quali quantitative observations of the samples were performed both with Polarized Light Optical Microscopy (PLOM) and Phase Contrast Optical Microscopy with chromatic dispersion (PCOM).

The XRPD study was conducted using a conventional Bragg Brentano diffractometer with  $\Theta$  2 $\Theta$  geometry and CuK $\alpha$  radiation. Quantitative phase analysis was performed with the Rietveld method [34] using the GSAS package [35] and the graphical interface EXPGUI [36].

Fourier transform infrared spectroscopic (FTIR) analyses were performed on powder samples using a spectrometer operating in reflection geometry while micro Raman spectra were collected on single fibres/fibre bundles.

A quantitative determination of tremolite asbestos in the MM

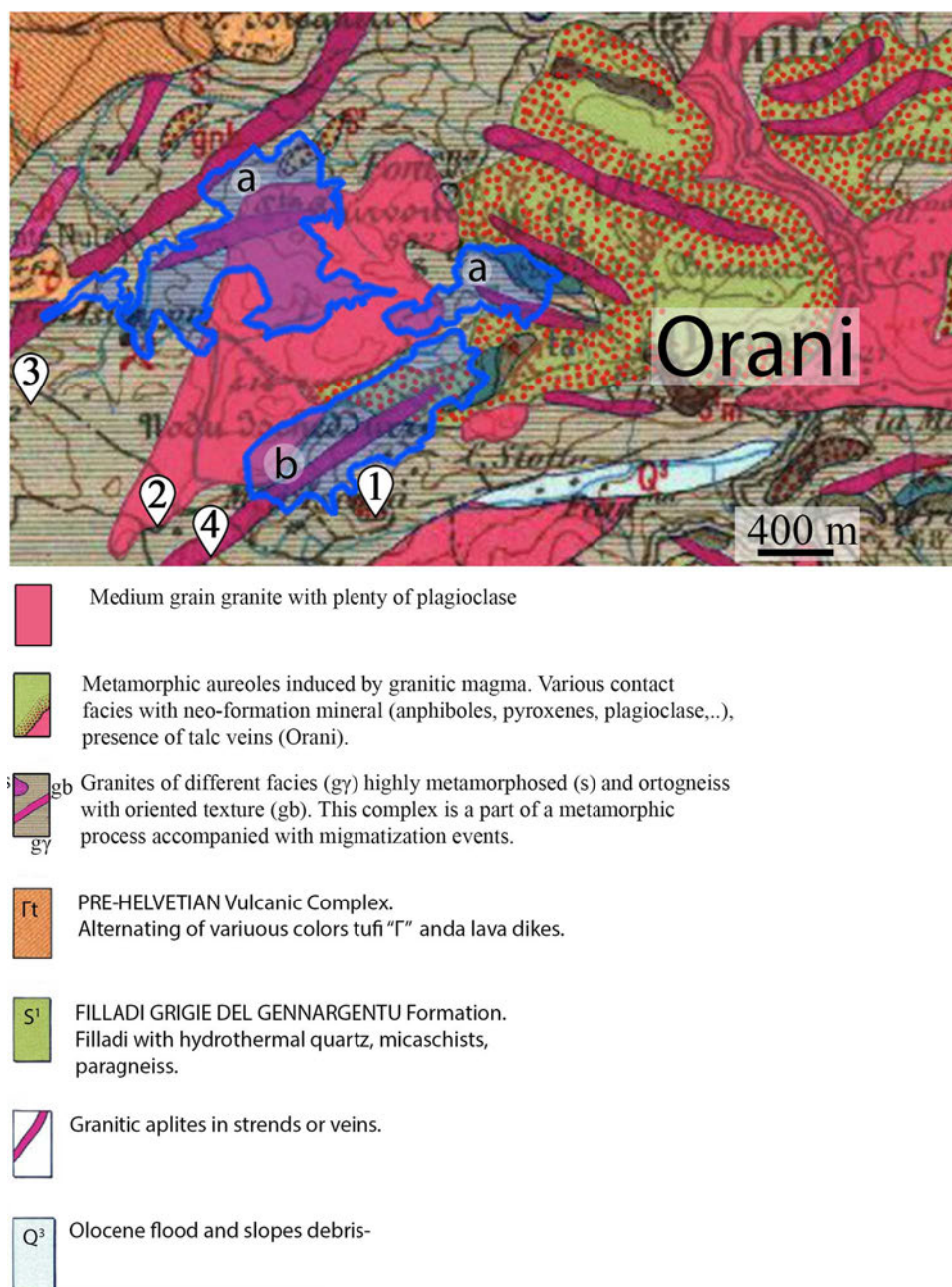


Fig. 1. Portion of the geological map (1:50000) of Orani with indication of the two extractive areas: a) Areas of the feldspar quarry; b) Area of the "Sa Matta" talc quarry. The sampling points are also indicated (MM sample not reported, being a commercial product resulting from a mixture of different extraction points).

sample was performed using the SEM technique. The MM product contains quartz with a mean particle diameter  $> 5 \mu\text{m}$  (i.e. not respirable) wherefore not considered when assessing the potential hazard of the material.

For the SEM quantitative analyses, the procedure suggested by the Italian Minister Decree 06.09.94 [31] (code: preparation 1) was applied. An alternative procedure (code: preparation 2) was also used to test the influence of the sample preparation on the final estimates. In this alternative procedure, an excess of sample powder (314 mg) was suspended in 500 ml of deionized water and shredded in ultrasonic bath so to obtain a final weight of the material deposited on the filter of 1.72 mg.

The filters prepared with the two procedures (code preparation 1 and 2) were cross analysed with two different experimental set ups (code: set up 1 and 2), a FEI Nova NanoSEM 450 FEG SEM equipped with a X ray energy dispersive spectrometer (X EDS) Bruker QUANTAX

200 system, and a Zeiss EVO 40 XVP in combination with an Oxford Inca Energy 250 EDAX system. The length  $L$  and width (assumed as the diameter  $D$ ) of all the observed fibres were determined and their nature was supported by EDS spot analyses. The concentration of tremolite fibres was calculated from both the total number of fibres according to the indications given in [31] and the number of 'regulated' fibres according to the WHO criteria [25].

The concentration  $C$  (in ppm) of the tremolite fibres was calculated using the equation below:

$$C = \frac{A \times (w_a)}{n \times a \times W} \times 10^6$$

with  $A$  = filter surface ( $\text{mm}^2$ );  $w_a$  = total weight of counted amphibole fibres (mg) using a density for tremolite of  $3.05 \text{ g/cm}^3$ ;  $n$  = number of analysed fields;  $a$  = area of the fields ( $\text{mm}^2$ );  $W$  = weight of the sample on the filter (mg).

**Table 1**  
Mineralogical composition (wt%) of samples from Rietveld quantitative phase analysis.

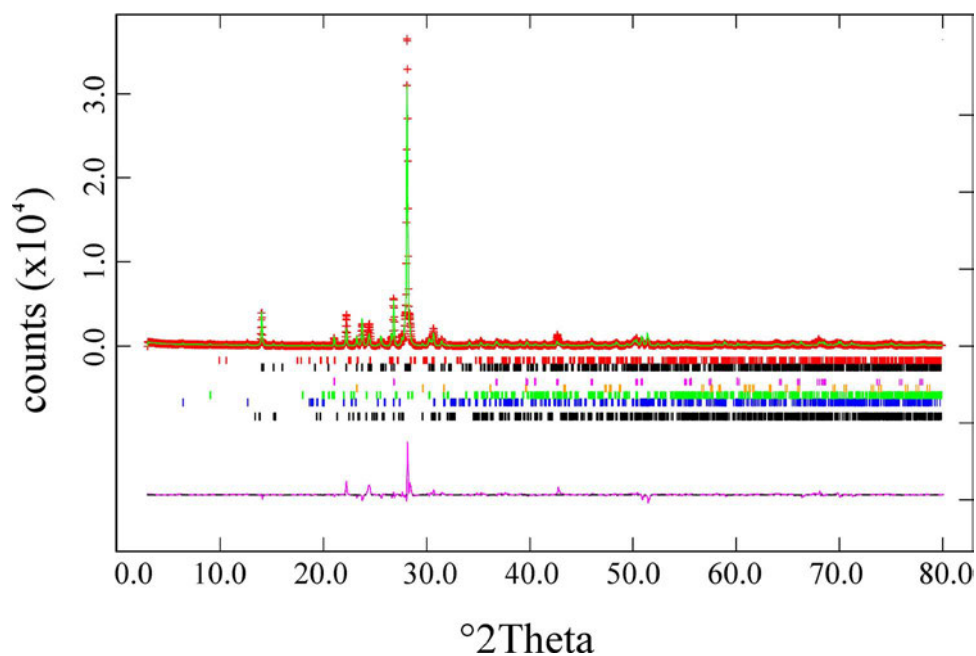
Sample	1	2	3	3-bis	4	MM
Agreement factors:						
R <sub>wp</sub> (%)	28.1	14.5	18.1	16.2	20.0	25.4
R <sub>p</sub> (%)	22.8	10.7	13.3	12.2	15.2	18.9
Phases (wt%):						
Calcite	–	–	–	–	1.6 (1)	0.2 (1)
Kaolinite	–	–	–	–	–	–
Chlorite	14.6 (7)	–	0.3 (2)	1.1 (2)	0.7 (2)	1.7 (3)
K-Feldspar	–	23.5 (5)	27.4 (4)	28.3 (5)	1.3 (1)	0.9 (3)
Mica / illite	–	3.1 (2)	4.4 (3)	2.5 (2)	2.6 (2)	2.6 (3)
Plagioclase	–	27.7 (2)	24.2 (3)	28.4 (3)	80.6 (8)	83.1 (1)
Quartz	–	45.7 (1)	39.7 (2)	38.1 (2)	12.1 (2)	10.9 (2)
Smectite	–	–	4.0 (5)	1.6 (2)	–	–
Talc	84.1 (2)	–	–	–	0.6 (2)	–
Tremolite	1.3 (4)	–	–	–	0.5 (1)	0.6 (3)

TEM investigations were accomplished using a JEM ARM 200 CF equipped with EDS and Electron Energy Loss Spectroscopy (EELS). Electron Micro probe analyses (EMPA) using a JEOL 8200 Super Probe instrument allowed us to determine the chemical composition of the MM sample.

### 3. Results

The results of the quantitative phase analyses performed on all the samples are reported in Table 1. As an example, the Rietveld graphical output of the refinement of the MM sample is shown in Fig. 2. The refined unit cell parameters of the tremolite phase in the MM sample are  $a = 9.82(1) \text{ \AA}$ ,  $b = 18.08(3) \text{ \AA}$ ,  $c = 5.27(1) \text{ \AA}$ , and the angle  $\beta$  corresponds to  $104.4(1)^\circ$ , matching the values from the literature:  $a = 9.84 \text{ \AA}$ ,  $b = 18.02 \text{ \AA}$ ,  $c = 5.27 \text{ \AA}$ ,  $\beta = 104.9^\circ$  Tremolite ICDD reference 00 013 0437 [33].

Fig. 3 reports selected images collected through Polarized Light (a,b) and Phase Contrast (c,d) optical microscopy. The calculated optical parameters are reported in Table 2. The table also reports the comparison of the optical properties of the investigated tremolite with the standard HSE tremolite asbestos from California, U.S.A. [32,33], showing a match between the two samples.



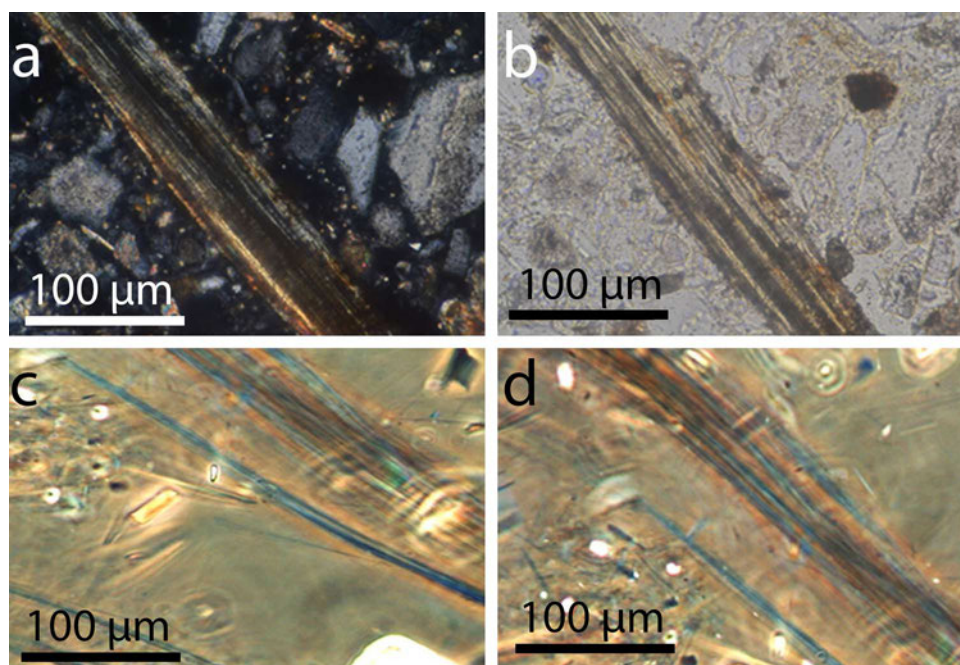
**Fig. 2.** Example of Rietveld refinement of the MM sample. Observed (crosses), calculated (continuous line), and difference (bottom line) curves are reported. Vertical bars mark Bragg reflection positions corresponding to the following phases (from top to bottom): tremolite; plagioclase; quartz; calcite; mica/illite; chlorite; K-feldspar.

The characteristic FTIR absorption bands of tremolite, evidenced by a black circle in the pure standard sample at about  $3600 \text{ cm}^{-1}$  (due to OH stretching vibration), were not observed in the bulk analysis of the MM sample due to the low content of the tremolite phase (Fig. 4). The micro Raman spectrum collected by pointing the laser beam directly onto the fibres of the MM sample (see the left top inset in Fig. 5) was identical to that of a standard tremolite specimen (RRUFF database ID R040109). The comparison with the spectra of other amphibole species present in the RRUFF database showed bands that are absent in the spectrum of the MM fibres: actinolite (ID R040063) displays an intense band at  $125 \text{ cm}^{-1}$ ; grunerite (ID R070204) displays an intense band at  $180 \text{ cm}^{-1}$ ; anthophyllite (ID R070245) displays an intense band at  $625 \text{ cm}^{-1}$ ; richterite (ID R060471) displays an intense band at  $1125 \text{ cm}^{-1}$ ; riebeckite (ID R060028) displays an intense band at  $150 \text{ cm}^{-1}$ ; and winchite (ID R061124) displays an intense band at  $550 \text{ cm}^{-1}$ .

A gallery of FEG SEM images showing the various shapes of the tremolite particles (identified by EDS) is shown in Fig. 6. These images confirm that there are tremolite particles with fibrous asbestiform shape. This is a first attempt to produce an atlas of tremolite particles to be used for future SEM quantitative determinations. The gallery shows:

- single fibres, cleavage fragments, columnar particle or fibre clusters of regulated size according to the WHO criteria [25] counted as 1 fibre according to both [25] and [31] guidelines (a, d, h);
- controversial cases with single and multiple fibre bundles, clump (cluster) of regulated fibres that cannot be counted individually according to [25] but counted as 1 fibre according to [31], termination of a fibre bundle that should not be counted according to [25] but counted as 1 fibre according to [31], gigantic clump that cannot be properly counted (b, c, e, f, g);
- lamellar or columnar particles that are not counted as fibres (i, l, m).

EDS microanalyses of some fibre bundles showed the presence of Na. It was not possible to determine whether Na was associated with the fibres or with other phases like Na feldspar which is a major components of the sample. To this aim, an accurate chemical composition of the fibres was obtained from EMPA analyses, carried out on a wide selection of fibres (oxides wt%, reported in Table 3). The amount of Na



**Fig. 3.** Mineral fibres observed with PLOM in dark field (a) and bright field (b). c) and d) are PCOM bright field analysis of fibres founded in the MM sample. The typical colours obtained with the 1.610 refractive liquid are shown. The colour of the fibres changes from light blue to blue-purple with bright yellow halo at the vary of the microscope polarizer. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

**Table 2**  
Optical data of the tremolite from Orani (Sardinia) and standard HSE tremolite asbestos.

	tremolite from Orani (Sardinia)	standard HSE tremolite asbestos
Pleocroism fibre parallel	None	None
Pleocroism fibre perpendicular	None	None
Birefringence extinction	II Order 0.015–0.030 ≈ 10°	Moderate Parallel or small angle
sign of elongation	Positive	Positive
colour fibre parallel dispersion staining*	Yellow	Yellow
colour fibre perpendicular dispersion staining*	Blue	Blue
colour fibre parallel phase contrast*	Grey, yellow-orange halo	Dark grey, yellow halo
colour fibre perpendicular phase contrast*	Blue, yellow-orange halo	Blue, orange halo
refractive index $n_{\alpha}$	1.60–1.62	1.616
refractive index $n_{\gamma}$	1.62–1.64	1.632
* refractive index liquid 1.605		

is actually very low and witnesses a possible contamination of the sample with Na feldspar during the SEM EDS microanalyses.

A statistical SEM morpho metric quantitative analysis was conducted on the MM sample to calculate the concentration of all the fibres according to [31] and the regulated fibres according to [25]. The results of the statistical analysis are reported in Table 4. The mean concentration C of both ‘regulated and unregulated’ and ‘regulated’ tremolite fibres is 2818 ppm (0.28%) and 2646 ppm (0.26%), respectively, indicating that most of the fibres are respirable. The sample preparation has an influence because the procedure (Code 1) according to the Italian Ministerial Decree 06.09.94 [31], invariably yields a lower concentration of fibres with respect to that obtained with the alternative procedure (Code 2). This is likely due to the difficulties with this method to observe homogeneous and statistically representative areas of the sample at low concentrations (close to 1000 ppm). Fibre aggregates are not evenly distributed in the sample and a random

distribution of the fibres on the filters cannot be obtained. In concert, the Italian Ministerial Decree 06.09.94 [31] clearly reports that “For asbestos concentrations below 1000 ppm, the method provides semi quantitative results.”

The crystal structure of tremolite crystals was verified through both SAED and HRTEM Fast Fourier Transform. The images in Fig. 7 show a tremolite fibre viewed with the c axis perpendicular to the electron beam. The measured cell parameters are  $a = 9.8087 \text{ \AA}$ ,  $b = 18.1159 \text{ \AA}$ ,  $c = 5.2743 \text{ \AA}$ , and the angle  $\beta$  corresponds to  $104.36^\circ$ . The TEM study evidenced the high crystallinity of the fibres, together with an additional EDS microanalysis spectrum (see Fig. 7c,d).

#### 4. Discussion

The results of the Rietveld quantitative phase analyses (Fig. 2 and Table 1) can only tell us that 0.6 wt% of an amphibole phase with a structure compatible with tremolite is found in the sample. Given its low amount and thus very minor contribution to the diffraction pattern, little can be said about the chemical nature of the amphibole phase and it is virtually impossible to distinguish between tremolite and calcic or sodium calcic amphibole. Moreover, diffraction tells nothing about its crystal habit (asbestiform, lamellar, prismatic or others). FTIR on the bulk sample is also poorly informative because the amphibole contribution to the spectrum is negligible. This is likely due to sample inhomogeneities. The aid of micro Raman spectroscopy carried out on single fibre bundles has proven to be an invaluable tool for the identification of asbestos minerals in bulk samples [37] and allowed us to classify them as tremolite. Moreover, the optical properties of the fibres are compatible with those of standard HSE tremolite asbestos (Fig. 3 and Table 2).

The HR TEM investigation (Fig. 7) with the indexing of the SAED diffraction patterns and determination of the unit cell parameters conveys another evidence that the investigated fibrous phase is tremolite.

Regarding the chemical composition, the EDS microanalyses associated with SEM imaging is not conclusive: some fibre bundles display a chemical composition compatible with that of tremolite but other ones

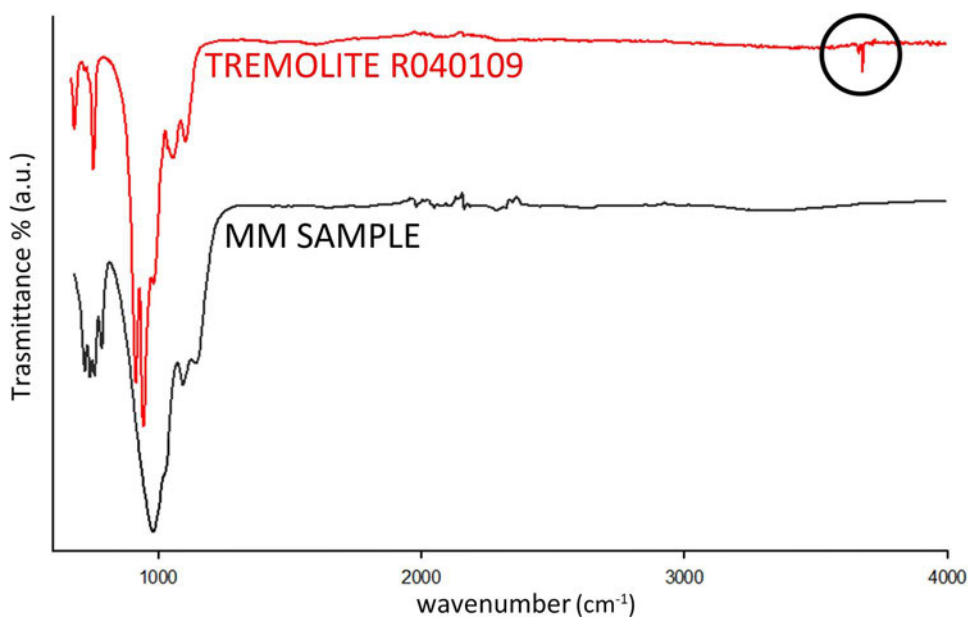


Fig. 4. FTIR spectrum collected on the MM sample (black) compared with a reference spectrum of tremolite (red); source: RRUFF database, ID R040109. The black circle evidences the position of the absorption bands of the hydroxyl molecules in the pure standard tremolite which are not visible in the investigated sample. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

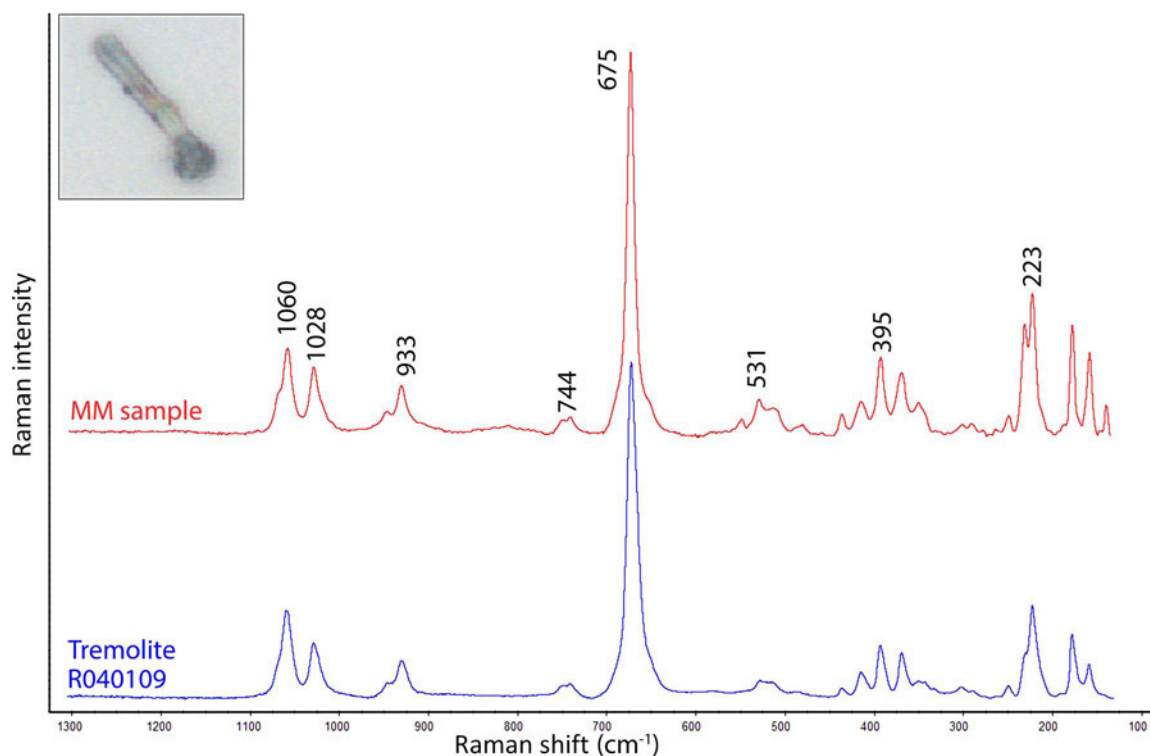


Fig. 5. Micro-Raman spectrum collected on a fibrous particle (red) and a reference spectrum of tremolite (blue); source: RRUFF database, ID R040109. The left upper inset portrays the mineral fibre analysed with the micro-Raman spot. The numbers reported in the graph are the Raman shift positions ( $\text{cm}^{-1}$ ) of the peaks. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

contain Na and hence could be classified as amphibole species other than tremolite, such as richterite  $\text{Na}(\text{CaNa})\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$  or winchite  $\text{Na}(\text{CaNa})\text{Mg}_4(\text{Al}, \text{Fe}^{3+})\text{Si}_8\text{O}_{22}(\text{OH})_2$ .

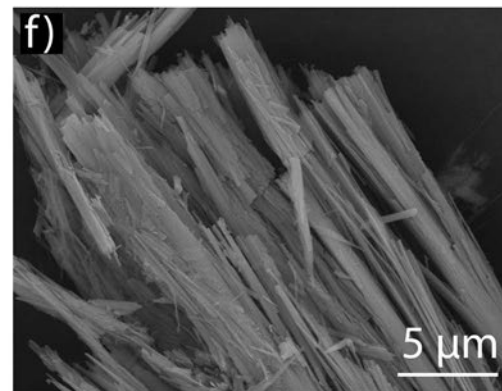
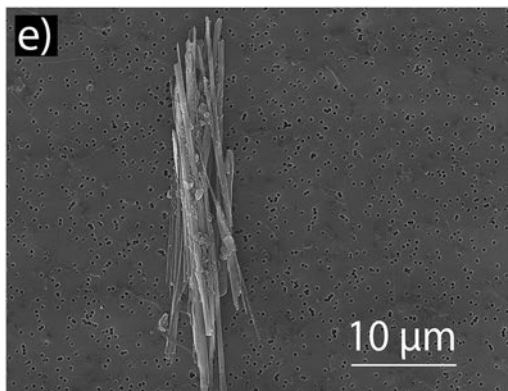
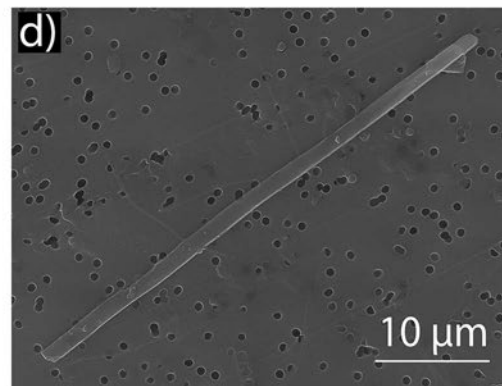
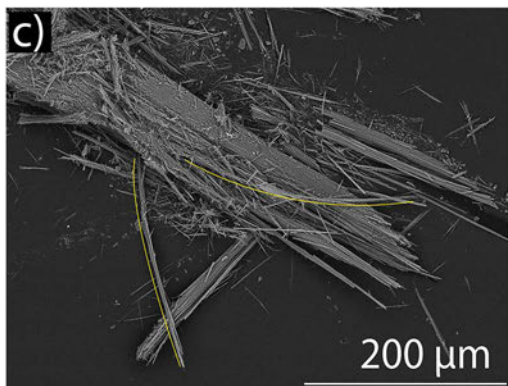
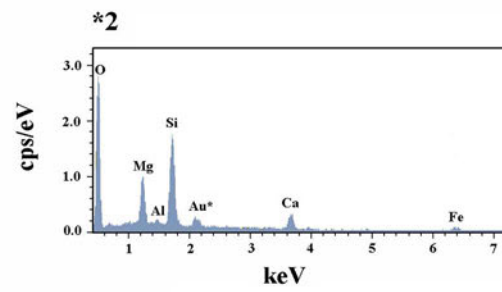
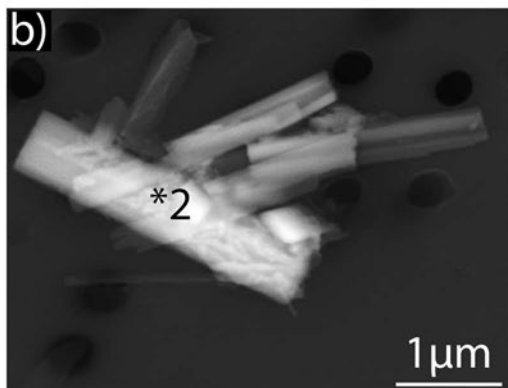
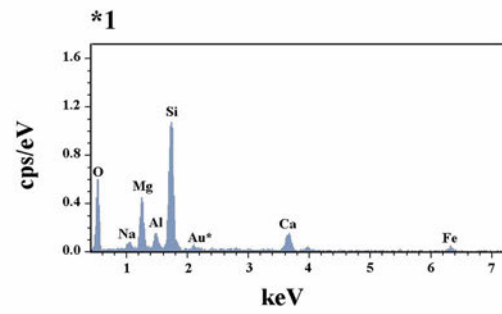
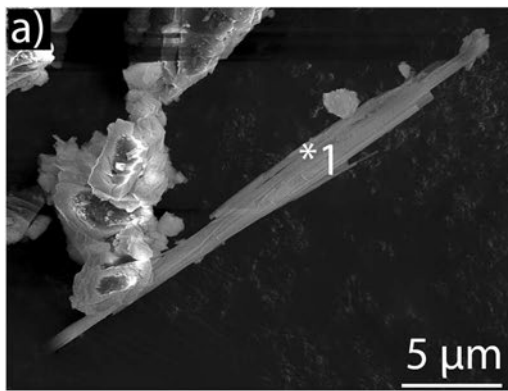
The issue was solved with the aid of EMPA which provided the calculation of the crystal chemical formula, according to the schemes proposed in [38,39]:



The formula fits the chemical standards for tremolite and tremolite asbestos (Table 2). The comparison with standard HSE tremolite asbestos was not possible because only EDS data were available.

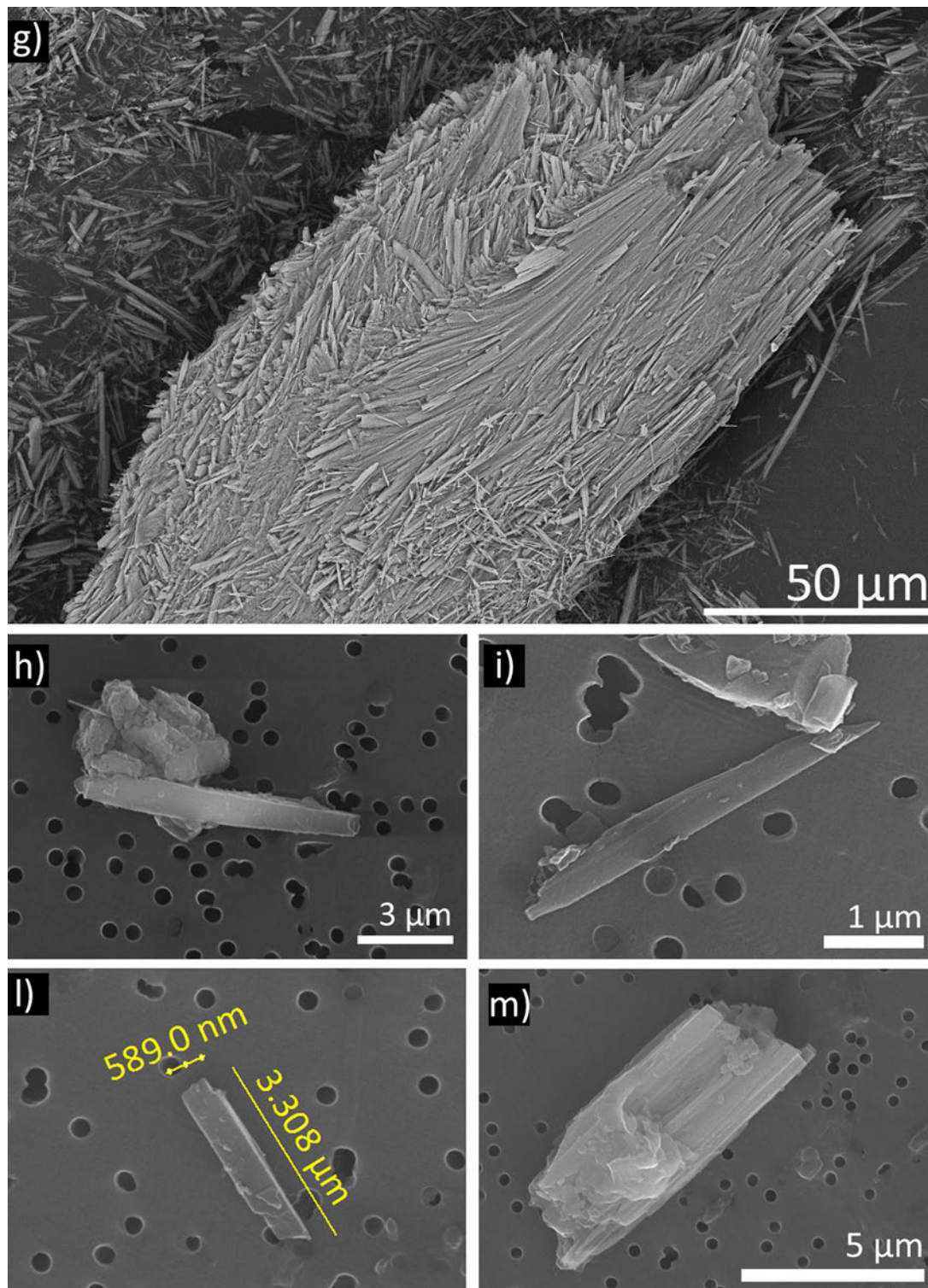
The results of the EPMA demonstrate that the contribution of Na is actually due to small Na feldspar crystals that are irradiated by the electron beam during the EDS analysis. This is one of those cases that expose the inaccuracy of the EDS quantitative microanalysis when applied to complex multiphase systems [40].

All the investigated fibres evidence the same chemical composition



(caption on next page)

**Fig. 6.** High-resolution SEM-FEG images of the tremolite particles found in the MM sample. This gallery should be considered a first attempt to produce an atlas of tremolite particles to be used for future SEM quantitative determinations. Legend: (a) a clump (cluster) of fibres with the relative EDS microanalysis on spot \*1 in the right top inset (Au\* is the coating element). This cluster is of regulated size according to [25] and counts as 1 fibre according to both [25] and [31] guidelines; (b) a cleavage fragment or columnar particle with the relative EDS microanalysis on spot \*2 in the second right inset from the top (Au\* is the coating element). This particle is not counted as regulated fibre [25] but possibly counted as 1 fibre according to [31]; (c) this is one of the most controversial cases with various single and multiple bundles that are difficult to classify in terms of fibre counting. Some of the fibres display a flexible asbestos crystal habit, evidenced by the yellow line; (d) a particle with acicular crystal habit of regulated size according to [25]. It counts as 1 fibre according to both [25] and [31] guidelines; (e) a clump (cluster) of regulated fibres that cannot be counted individually according to [25]. Oppositely, individual fibres can be counted according to [31]; (f) termination of a fibre bundle that should not be counted according to [25]. On the other hand, the visible part of the fibre should be counted according to the criteria defined in [31]; (g) a gigantic clump that cannot be properly counted highlighting the limits of the techniques in terms of sample preparation; (h) a cleavage fragment or columnar particle of regulated size counted as 1 fibre for both [25] and [31] guidelines; (i) a lamellar particle not counted as a fibre; (l) a lamellar particle not counted as fibre; (m) a columnar fragment that should not be taken into account. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 6.** (continued)



**Table 3**

EMPA chemical composition of the fibres present in the MM sample. Reported chemical compositions are mean values of 10 spot analyses carried out on 10 different fibres. Reference data of Pacella et al. [47] of a fibrous tremolite from Ala di Stura (Lanzo Valley, Italy) is also shown for comparison.

Oxides	This work	Pacella et al.
SiO <sub>2</sub>	57.0 (2)	57.50 (4)
TiO <sub>2</sub>	0.02 (3)	0.02 (2)
Al <sub>2</sub> O <sub>3</sub>	1.30 (2)	0.05 (1)
Cr <sub>2</sub> O <sub>3</sub>	0.01 (2)	0.01(1)
MnO	0.08 (3)	0.26 (5)
MgO	20.3 (6)	22.64 (2)
CaO	13.11 (8)	13.14 (1)
Na <sub>2</sub> O	0.13 (3)	0.06 (2)
K <sub>2</sub> O	0.04 (1)	0.03 (1)
NiO	0.01 (2)	–
F	–	0.04 (5)
Fe <sub>tot</sub>	5.3 (9)	2.42 (3)
FeO	–	2.20
Fe <sub>2</sub> O <sub>3</sub>	–	0.24
H <sub>2</sub> O	2.02 <sup>a</sup>	2.13
Total	99.28	98.28

<sup>a</sup> estimated from stoichiometry.

**Table 4**

Results of the SEM quantitative analysis for the determination of the concentration of tremolite fibres. Legend: (a) total number of fibres; (b) regulated WHO [15] fibres. Section (b) includes the concentration of fibres with  $5 < L < 20 \mu\text{m}$  and  $L > 20 \mu\text{m}$  determined with Procedure Code 1 and Set-up Code 1 and Procedure Code 2 and Set-up Code 1.

(a)								
Fibres counted	Procedure Code 1	Set-up Code 1	Procedure Code 2	Set-up Code 1	Procedure Code 2	Set-up Code 2	Procedure Code 1	Set-up Code 2
	117		1173		102		70	
	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )
Mean	5.1	0.6	6.9	0.5	10.2	0.9	8.5	0.7
Error	0.5	0.1	0.2	0.01	0.4	0.04	0.9	0.05
Std. Dev.	3.8	0.3	4.0	0.4	4.1	0.4	7.5	0.4
Median	3.8	0.5	5.9	0.4	9.6	0.8	5.9	0.6
Max. Value	21.5	1.9	34.3	3.8	22.5	2.2	38.8	2.3
Min. Value	1.1	0.2	0.6	0.1	5.0	0.3	3.2	0.2
C (ppm)	1100.2		3771.2		4100		2300	
Mean C (ppm)				2818				
C (%)	0.11		0.38		0.41		0.23	
Mean C (%)				0.28				
(b)								
Fibres counted	Procedure Code 1	Set-up Code 1	Procedure Code 2	Set-up Code 1	Procedure Code 2	Set-up Code 2	Procedure Code 1	Set-up Code 2
	41		784		398		47	
	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )	L ( $\mu\text{m}$ )	D ( $\mu\text{m}$ )
Mean	8.5	0.7	8.4	0.5	9.2	0.8	10.3	0.7
Error	0.6	0.1	0.3	0.02	0.5	0.04	1.2	0.1
Std. Dev.	4.1	0.4	4.1	0.3	5.6	0.5	8.4	0.4
Median	7.0	0.6	7.1	0.5	8.0	0.6	7.2	0.6
Max. Value	21.5	1.9	34.3	2.5	59.8	3.0	38.8	2.3
Min. Value	5.0	0.2	2.3	0.1	2.0	0.04	5.0	0.3
C (ppm)	916.7		3044.2		4520.6		2100	
Mean C (ppm)				2646				
C (%)	0.09		0.30		0.45		0.21	
Mean C (%)				0.26				
Fibres $5 < L < 20 \mu\text{m}$	39		768					
C (ppm)	880		2983					
C (%)	0.086		0.294					
Fibres $L > 20 \mu\text{m}$	2		16					
C (ppm)	37		61					
C (%)	0.0036		0.006					

and characteristics, ruling out the possibility that other asbestos minerals are present as in the case of Libby, (Montana, USA) where a mixture of different asbestos species was discovered [9].

Having defined the mineralogical nature of the fibrous mineral phase, the second step of the protocol requires to assess whether tremolite is “tremolite asbestos” or not. Following the scheme proposed in the Introduction, by comparison with the HSE standard of tremolite asbestos, we have enough scientific evidence to classify the fibrous component of the feldspar rich rock from Orani, Sardinia (Italy) as ‘asbestos tremolite’ (Table 2).

The statistical data of the morphometric analysis yielded a mean length L of the counted fibres in the range 5.14–10.16  $\mu\text{m}$ , a mean diameter D in the range 0.51–0.85  $\mu\text{m}$  and mean aspect ratio L/D in the range 9.18–13.45 (see Table 4). As far as the regulated [25] fibres are concerned, the mean L is in the range 8.35–10.29  $\mu\text{m}$ , the mean D is in the range 0.53–0.77  $\mu\text{m}$  and mean L/D is in the range 12.00–15.75 (see Table 4). The morphometric analysis shows that most of the fibres are regulated (respirable). The optical (Fig. 2) and electron microscopy (Figs. 6 and 7) analyses evidence that the fibre bundles are flexible and hence the criteria used to define asbestos minerals on the basis of their asbestiform character (an “asbestiform” crystal habit refers to a flexible mineral fibre easily separable and arranged parallel to other fibres [41]) are also met.

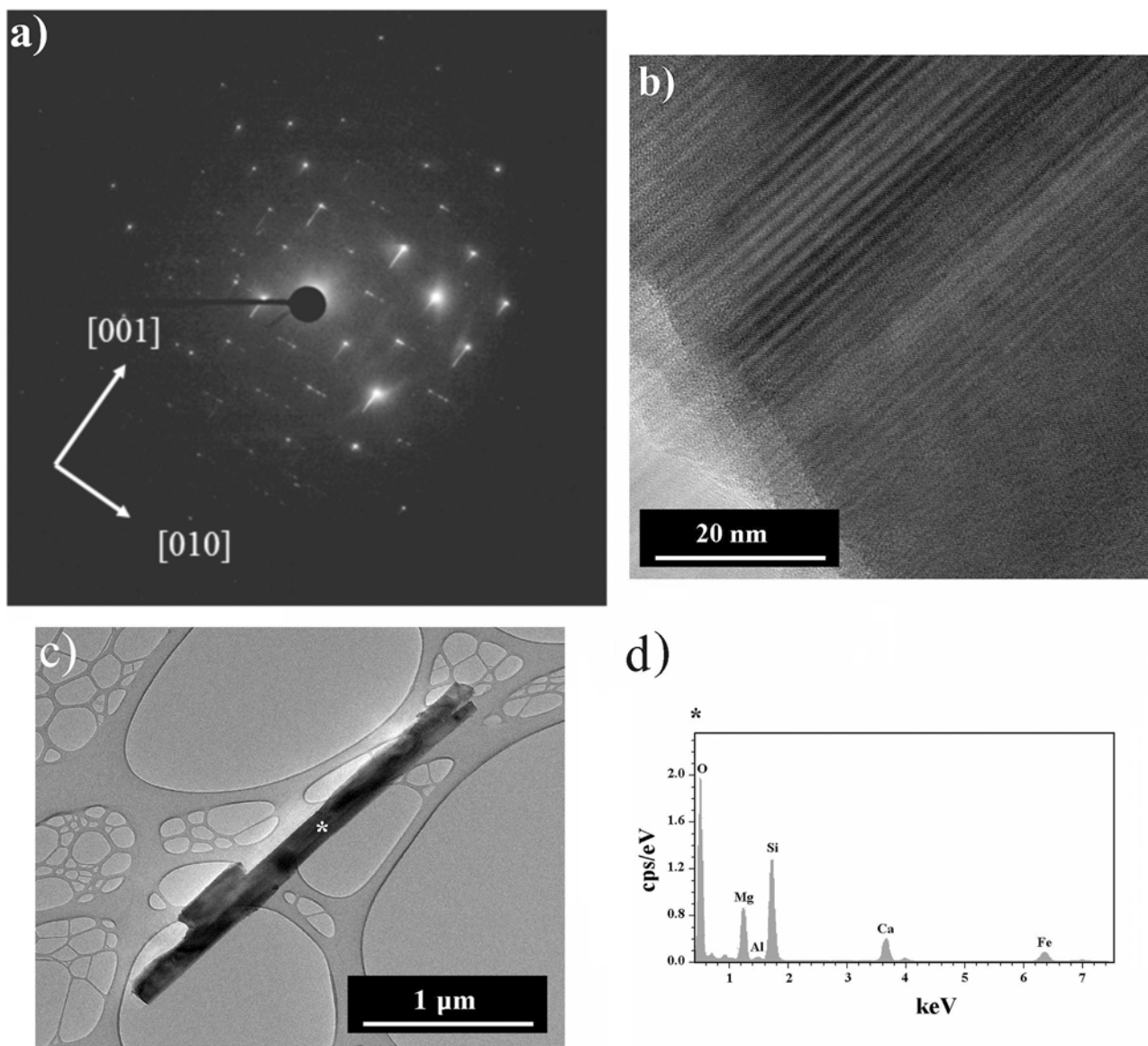


Fig. 7. Results of the TEM analysis on fibres from MM sample. Legend: (a) Indexed SAED diffraction pattern; (b) HR TEM image of a fibre; (c) Another fibre at low magnification with the relative EDS microanalysis spectrum (d).

On the basis of the mineralogical and regulatory parameters, the potential hazard represented by the tremolite asbestos found in the feldspar raw material from Orani should be considered and carefully evaluated. The potential hazard represented by asbestos tremolite is witnessed by its high biopersistence [42,43], a component of biopersistence (the longer a fibre persists in the lower respiratory tract, the greater is the likelihood that it will cause adverse effects and pathogenicity in the lungs), one of the key parameters of the fibres' toxicity paradigm [44].

Our results indicate that the concentration of asbestos tremolite in the Orani's feldspar (sample MM) is 0.28 wt% or 0.26 wt%, if only regulated [25] fibres are considered (Table 4). Considering that the quantitative phase analysis with the Rietveld method yields a phase fraction of 0.6 wt%, a large fraction of the amphibole phase displays a crystal habit other than fibrous asbestiform or acicular (see Fig. 6). New air sampling of the dust in the mine should be performed in the future to determine what fraction of the 0.26% concentration in the raw material is aerosolized.

For the existing Italian laws, the use of this raw material which contains an asbestos phase should be prohibited. The concentration

values exceed the limit of 0.1 wt% imposed to free asbestos fibres in contaminated anthropogenic soils (not naturally occurring asbestos) by the Italian Law 152/06 [45].

Although the protocol of analysis that we have applied to the case of the feldspar from Orani permitted a conclusive classification of the fibrous component, the use of a suite of validated and more advanced experimental techniques of crystal chemical and crystallographic investigation such as HR TEM and EPMA made the overall procedure costly for most of the labs involved in environmental monitoring and analysis. On the other hand, an undisputable outcome in terms of regulatory classification may have a number of benefits to address a proper management and compulsory administration of the mining activity at the site of Orani.

The outcome of this work should be used to find possible solutions for a safe exploitation and mineral processing in the mine. If, as evidenced by the mineralogical analyses (e.g., Table 1), tremolite is associated to talc, originated as a consequence of a thermo metamorphic event that affected limestones and schists, a selective exploitation excluding the talc rich areas and especially the area at the border with the adjacent talc mine should be conducted. If, on the other hand, the

presence of tremolite regards all the different rock types present in the mining lease, the selective exploitation of the ‘asbestos free’ raw material should regard the whole mining area. In both cases, a ‘safe’ regulated mining activity may be adopted following the example of selective mining for the serpentine rocks of New Caledonia under Dé libération nr. 82 [7]. A similar legislative tool has been implemented in another Italian region (Emilia Romagna) for mining activity of asbestos containing serpentine rocks [46]. The procedure must include (i) a detailed geological survey of the deposit aimed at mapping the asbestos contaminated and ‘asbestos free’ areas (updated *in itinere* when mining operations are in progress); (ii) a suitable mining operation plan to operate selective exploitation of the “asbestos free” areas; (iii) a safety plan with measures to decrease the risk of exposure to asbestos for the workers taking advantage of the best available techniques (BAT); (iv) safety and professional training of the workers to make them aware of the health hazard represented by asbestos and to safely manage mining operations in the presence of asbestos; (v) continuous monitoring of the asbestos concentration in the *tout venant* with optical (PLOM and PCOM) and electron microscopy (SEM) by the operators of the mining company and by supervisory bodies for the environmental control and monitoring; (vi) certification of the product based on the concentration of asbestos and identification of the suitable use.

## 5. Conclusions

The protocol of analysis that we have applied to the feldspar from Orani (Sardinia, Italy) allowed us to assess that ‘tremolite asbestos’ is present as impurity. The use of ‘state of the art’ techniques of crystal chemical and crystallographic analysis helped to unquestionably identify the mineralogical nature of the fibrous mineral species. All the examined fibres evidenced the same chemical composition and characteristics, ruling out the presence of other asbestos phases.

Although a large fraction of tremolite does not display a fibrous asbestiform crystal habit, most of the tremolite fibres are ‘respirable’.

To run a safe exploitation and mineral processing at the Orani’s mine, a selective exploitation of the ‘asbestos free’ raw material should be conducted following the example of New Caledonia where a specially developed legislation permits selective mining of the rocks contaminated with asbestos. New air sampling of the dust in the mine should also be planned to determine the amount of respirable fibres that are actually aerosolized.

The results herein should not be surprising to the geological community as amphiboles are ubiquitous in the natural environment. However, these results may be useful to other professionals such as lawyers and toxicologists to understand that definitions applied to asbestos in the occupational setting cannot be applied to the natural setting and a new match is required.

## Competing interests

The authors declare that they have no competing interests.

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## Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.jhazmat.2018.02.012>.

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