

## Research Article

# Quantification of Tremolite in Friable Material Coming from Calabrian Ophiolitic Deposits by Infrared Spectroscopy

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The aim of this study is to identify the infrared absorption band suitable for quantifying tremolite in three powdered samples (fine, medium, and large size classes) coming from a quarry of ophiolitic friable rocks in the western part of the Calabria region of Italy. Three IR bands were considered: OH stretching band between 3700 and 3650  $\text{cm}^{-1}$ , the stretching bands of the Si-O-Si linkage between 1200 and 900  $\text{cm}^{-1}$ , and the absorbance band at 756  $\text{cm}^{-1}$  attributable to tremolite. The amount of tremolite in the test samples was quantified by using the curve parameters of the three analytical bands. The quantitative analysis of tremolite using the band due to OH stretchings (3700–3650  $\text{cm}^{-1}$ ) and the bands attributed to the Si-O-Si stretchings (1200–900  $\text{cm}^{-1}$ ) showed high values for all test samples. Their use overestimated the tremolite amount because both bands were affected at the interfering mineral silicates such as talc, kaolinite, chlorite, and serpentinites. The abundant presence of antigorite in studied samples mainly in medium size class sample had a key role in our findings. The band at 756  $\text{cm}^{-1}$  was not affected at the interfering minerals and can be used for quantitative analysis of tremolite in sample coming from ophiolitic deposits.

## 1. Introduction

The ophiolites, commonly known as greenstones due to their typical colouring, may contain asbestos; they represent a grouping of rocks of magmatic origin which characterise, in the form of outcrops, numerous areas in almost all continents and have long been the subject of interest of researchers for their geological and petrographic aspects. In Italy, the presence of numerous deposits is confirmed in the Alps and mainly along the Apennines. These outcrops extend from the Ligurian-Piedmont through the Tuscan-Emilian Apennines as far as Val Tiberina and continue, in disjointed groupings, to the Calabrian-Lucan Apennines [1].

Calabrian ophiolites are an important mineralogical and economical resource. The fibrous variety of asbestos found in these sites is tremolite [2–4].

Due to the presence of natural asbestos in ophiolite outcrops, characterization of the asbestos minerals extracted during excavation phases of the construction projects, as well as the development of analytical procedures for an effective characterization of the asbestos hazard, is needed.

In this study, we propose an investigation of three infrared absorption bands for the quantitative determination of tremolite in the material used as inert filler coming from a quarry of ophiolitic friable rocks in the west of the Calabria region of Italy. The first infrared band is in the region of OH group stretching vibration; the other two IR bands are associated with stretching Si-O-Si and O-Si-O vibration. The data allowed for identifying the most indicative IR absorption band for quantifying tremolite in samples coming from ophiolitic deposits.

## 2. Materials and Methods

Test samples containing tremolite came from a quarry of ophiolitic friable rocks in the neighbouring of Platania, town in the province of Catanzaro in the western part of the Calabria region (Italy).

The material produced was used as inert filler.

The production of the friable material was subdivided into two phases; the first took place directly in the quarry with selection of raw material and the second occurred at the crushing and shifting plant where the material, transported from the greenstone quarry, was unloaded inside the hopper and crushed. Next it was moved by a belt conveyor to vibrating sieves where it was subdivided into three homogeneous size classes: fine, medium, and large. The final processing phase consisted of loading the material for transport onto motor vehicles. It is worth noting that the crushing and sizing plant was equipped with a clarification system of recycled water and mud.

Samples from each of the three size classes were analysed by scanning electron microscopy (SEM, LEO 440) complete with energy dispersive X-ray spectroscopy (EDS, Oxford Instrument INCA) and FTIR (Spectrum one, Perkin Elmer) to identify the tremolite. In order to know the amount of tremolite in three size classes of materials by FTIR, a calibration curve was necessary to set up by plotting the intensity of the absorption peaks as a function of the known concentration of pure tremolite.

In nature, tremolite is close in composition to its ideal end-member formula, although it can incorporate appreciable amounts of extra Mg as a result of Mg substitution for Ca and Na, K, Fe, Al, and F are always present in small amounts [5]. The variability in composition is a direct consequence of the fact that the structure can accommodate many different ions in the space between the silica ribbons, which form the fibers, and that the variable nature of the host rocks can contribute different ions to this structure [6].

For this reason, it was decided to use the powdery phase of friable rock coming from San Mango, a town near the studied quarry, as standard sample.

It was showed [2] that the samples of San Mango area had two distinct phases: the first one was white and the second one green with compact nature. SEM and FTIR analysis showed that the white powdered phase was tremolite.

To use the white phase of the mineral as standard sample, a careful separation of white phase from that green one was necessary by the stereomicroscope (Leica M205C).

Tapping slightly the mineral sample with spatula, the white phase fell down heavily and it was collected on a porcelain crucible. The coarse impurities were detected and removed by stereomicroscope. The collected powder was heat-treated in a muffle furnace (Gefran 400) at 450°C for 12 hours. The sample was analyzed by FTIR and by SEM-EDS. The analysis confirmed that the powder was tremolite.

Ten powdered samples of tremolite were weighted by a Sartorius scale (MC1, capacity 210 g, precision 0.01 mg, Sartorius AG) equipped with a device to neutralize electrostatic

charges. The weighted quantities were in the range of 50 µg–1 mg. A quantity equal to 3 mg of each sample was mixed with about 250 mg of pure KBr previously dried at 120°C in an oven (Falc Instruments) for 12 hours. Ten transparent pellets of diameter 13 mm were obtained by applying a force-weight of 10 tons for about two minutes.

IR absorbance was recorded from 4000 to 400 cm<sup>-1</sup>. Before analyzing the samples, spectrum acquisition of a blank consisting of a pure KBr pellet was performed. The analyses were carried out with the following operating conditions: 4000–400 cm<sup>-1</sup> acquisition interval, 4 cm<sup>-1</sup> resolution, and 32 scans. The use of a resolution of 4 cm<sup>-1</sup> was suitable for a good separation of the bands in the spectra. The optical compartment of the instrument was flushed with nitrogen before starting sample analysis. Before each scan, the sample compartment has been saturated with the same gas for about ten minutes to clean the air.

The chosen bands for quantitative analysis were between 3700 and 3650 cm<sup>-1</sup>, 1200 and 900 cm<sup>-1</sup>, and the band around 756 cm<sup>-1</sup>.

Calibration curves were built with ten analytical points for each analytical band, by plotting the net area of the band integrated from the baseline to its minimum of absorption.

Test samples coming from three size classes, fine, medium, and large, with unknown content of tremolite, were ground using an agate mortar with some drops of absolute ethanol for analysis for reducing mechanical stress and for obtaining a particle size around 1–2 µm.

For an accurate quantitative analysis by FTIR, the particle size should not exceed 2.5 µm. In general, under this value, the scattering losses can be ignored [7]. The particle size was checked by SEM observations.

Five pellets were realized for each size class sample. The two worst pellets, which showed inhomogeneities, lesions, opacities, or imperfections, were rejected. Six IR spectrum acquisitions were performed for each of the remaining three pellets, three for each pellet side in order to mediate possible inhomogeneities invisible with naked eye. An average area and deviation standard of each studied IR band was obtained. The three average areas were used to calculate an area-weighted average of the IR band. The analytical procedure employs weighting factors  $w_i$  which serve to take into account the accuracy of measurement  $X_{mi}$ . Statistical theory shows that the variance of the weighted average is minimized when the individual weights are taken as the inverse of the variance of the individual measurement [8]. Low weights are given to values with high variance.

It is assumed that the best estimate of band area is area-weighted average  $X_{\text{best}}$  with a standard error  $\sigma_{\text{best}}$ :

$$X_{\text{best}} = \frac{w_1 X_{m1} + w_2 X_{m2} + w_3 X_{m3}}{w_1 + w_2 + w_3}, \quad (1)$$

where the weighted factor  $w_i$  is

$$w_i = \frac{1}{\sigma_i^2}, \quad (2)$$

$$\sigma_{\text{best}} = \sqrt{\frac{1}{w_1 + w_2 + w_3}}.$$

### 3. Results and Discussion

An essential feature of amphibole structure is a double chain of corner-linked silicon-oxygen ( $\text{SiO}_4$ )<sup>4-</sup> tetrahedrons that extend indefinitely parallel to the *c* crystallographic axis, the direction of elongation. The tetrahedrons alternately share two and three oxygen atoms to produce a silicon-to-oxygen ratio of 4:11. The structure contains, besides the tetrahedral sites that constitute the chains, additional cation sites labeled A, M4, M3, M2, and M1. In amphibole, the M1, M2, and M3 sites are octahedral and occupied by  $\text{Mg}^{2+}$  and  $\text{Fe}^{2+}$ , while the M4 sites are typically occupied by  $\text{Ca}^{2+}$  and A-sites may be occupied by  $\text{Na}^+$  or be vacant.

Several studies of amphiboles have addressed the role of cation occupancy of the M1, M3, and A-sites on the position of OH infrared absorption bands, very useful in the discrimination between the members of the same series (e.g., tremolite-actinolite).

The chemical composition of amphiboles is not necessarily fixed due to the possibility to form solid solution series with other minerals being their end-members.

Calcic amphiboles crystallize in the monoclinic crystal system [9]. Tremolite is a calcic amphibole with ideal formula  $\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$ . It is an end-member of the tremolite-ferroactinolite series. Term tremolite is used when the ratio  $\text{Mg}/(\text{Mg} + \text{Fe}^{2+}) \geq 0.90$ , actinolite when the ratio  $\text{Mg}/(\text{Mg} + \text{Fe}^{2+})$  is between 0.50 and 0.90, and ferroactinolite when the ratio  $\text{Mg}/(\text{Mg} + \text{Fe}^{2+}) < 0.50$  [10, 11].

Both vibrational spectra of tremolite and actinolite proved difficult to discriminate in the 1300–400  $\text{cm}^{-1}$  spectral region while in the region of hydroxyl group stretching vibrations (3700–3600  $\text{cm}^{-1}$ ) these spectra are different and prove more easily distinguishable.

The first IR band used for quantifying tremolite was in the range of 3700–3650  $\text{cm}^{-1}$ .

According to Della Ventura et al. [12–14] and Hawthorne et al. [15], in the 3700–3600  $\text{cm}^{-1}$  spectral region, the hydroxyl group-metal ion bonds are present. This means that the substitution of different cations can be seen in this spectral region where the disappearing, shifting, and splitting of the bands are used in order to gain information about cation ordering, as well as supplementary details necessary in the discrimination between the minerals from the same series.

Depending on the presence and/or the absence of  $\text{Fe}^{2+}$  in M1 and M3 sites, the number of bands of the OH stretching region could range from one in tremolite up to four in ferroactinolite samples [16, 17]. The data from the literature [18] assign the weak band around 3660  $\text{cm}^{-1}$  to the presence of Mg in M1 sites and of  $\text{Fe}^{2+}$  in the M3 site and the stronger band at 3675  $\text{cm}^{-1}$  to the (3  $\text{Mg}^{2+}$ )-OH vibration (where the  $\text{Mg}^{2+}$  ion is present in all nearest-neighbour octahedral sites, i.e., M1, M2, and M3).

The shifting of the OH stretching bands towards a lower frequency, starting the replacement of  $\text{Mg}^{2+}$  by  $\text{Fe}^{2+}$  in configuration, is correlated with an increase of the electronegativity of the substituting element [19, 20].

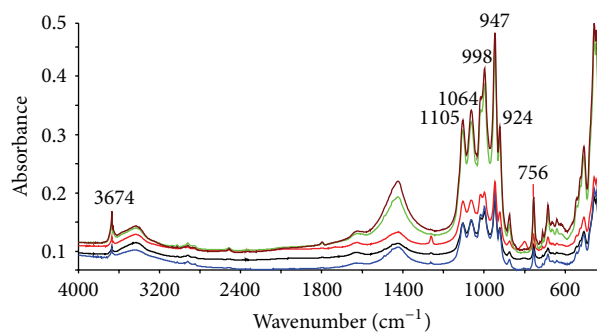


FIGURE 1: FTIR spectra at increasing quantities (50  $\mu\text{g}$ –1 mg) of the tremolite used as standard sample to set up the calibration curve.

According to Kasir et al. [21], for amphiboles, well-established correlation exists between OH stretching frequency and chemical formula of the different M-cations bonded to hydroxyl. For example, for each  $\text{Fe}^{2+}$  substituting for Mg at M1 and M3 sites, there is a decrease in OH wave number of 15  $\text{cm}^{-1}$ . Similarly for each Al substituting for Mg, there is a reduction of 20 to 30  $\text{cm}^{-1}$ . The value of OH stretching wave number is also affected by the contents of Na.

The second spectral band between 1200 and 900  $\text{cm}^{-1}$  chosen for quantifying tremolite is assigned to the asymmetric stretching of the Si-O-Si and O-Si-O vibrations [10, 22–28].

For the lower region (775–600  $\text{cm}^{-1}$ ), the vibrations observed in this spectral domain are assigned to the symmetric stretching of the Si-O-Si linkage [23, 24]. Moreover, in this spectral range, additional bending modes of the OH group [23] and of the Si-O-Si may appear [29].

Figure 1 shows some among the tremolite standard spectra used to set up the calibration curve.

Besides the typical tremolite absorption band at 3675–3673  $\text{cm}^{-1}$  assigned to the vibration of the O-H dipole bonded to three Mg cations [12–14, 30–35], a well-developed band is observed at 3660  $\text{cm}^{-1}$ . This latter was attributed to the  $\text{Fe}^{2+}$  environment [22]. Moreover, no  $\text{Fe}^{3+}$  is present because absorption bands at  $\Delta = -50 \text{ cm}^{-1}$  from tremolite reference band are absent [36].

The presence of Fe is confirmed by EDS spectrum shown in Figure 2 with SEM image of sample used as standard.

In the spectral range between 1200 and 900  $\text{cm}^{-1}$ , the main absorption band system is found, consisting of partly overlapping bands. This absorption band system has several shoulders and peaks near 1100, 1060, 1000, and 950  $\text{cm}^{-1}$  and is attributed to Si-O and Si-O-Si stretching. Another, but less intense, band system is observed in the region between 775 and 600  $\text{cm}^{-1}$ . The absorbance peak at 756  $\text{cm}^{-1}$  that is one of the wave numbers attributable to tremolite [37–41] was the third band chosen for quantifying tremolite.

In Table 1, assignment of the bands observed in IR spectra (wavenumbers,  $\text{cm}^{-1}$ ) of tremolite used as standard sample is showed.

Using the three bands mentioned above, linear calibration graphs were established by the method of band area.

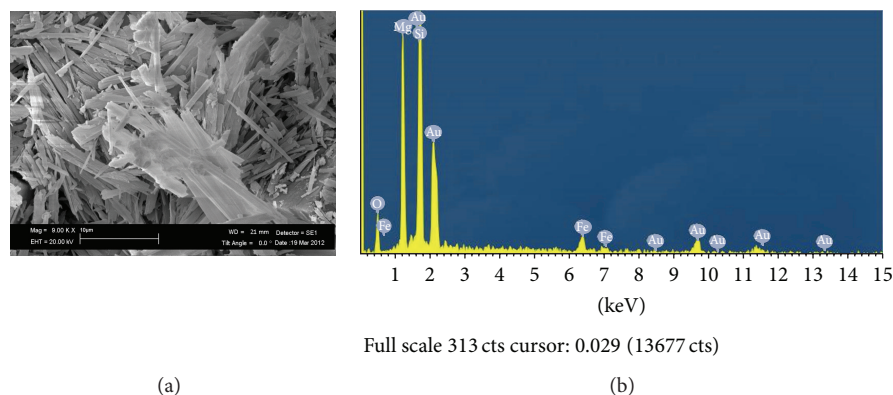


FIGURE 2: SEM image of the sample used as standard (a) with its EDS spectrum (b).

TABLE 1: Infrared spectra (wavenumbers,  $\text{cm}^{-1}$ )\* of tremolite used as standard sample.

Absorption frequency ( $\text{cm}^{-1}$ ) of tremolite (San Mango, Italy)	Assignment [12, 29]
3674 w	OH stretching vibration
3660 sh	
1105 vs	Si-O stretching Si-O-Si and O-Si-O stretching
1064 vs	
1016 sh	
998 vvs	
947 vvs	
924 vs	
756 s	Si-O and Si-O-Si stretching
545 sh	Metal-O stretching and Si-O bending vibrations
526 sh	
508 s	
458 vvs	
447 vvs	

Note: \* relative intensities are denoted by the following:  
s = strong, m = medium, w = weak, v = very, and sh = shoulder.

The basis for quantitative analysis of absorbance spectrometry is Lambert-Beer law. For a single compound in a homogeneous medium, the absorbance at any frequency is expressed as

$$A = abc, \quad (3)$$

where  $A$  is the measured sample absorbance at the given frequency,  $a$  is the molecular absorptivity,  $b$  is the absorption path length (pellet thickness), and  $c$  is the concentration of the substance.

It is known that [42] Lambert-Beer law is restricted to monochromatic light, noninteracting absorbers, very dilute solutions, absence of a Christiansen effect [43], particle size smaller than wavelength used, absence of scattering, linearity of the detector, and the assumption that the slits are infinitely small. Despite all these restrictions, it has been empirically demonstrated [42, 44–47] that the mid-IR absorbance of properly ground ( $\approx 2 \mu\text{m}$ ), evenly dispersed minerals at low

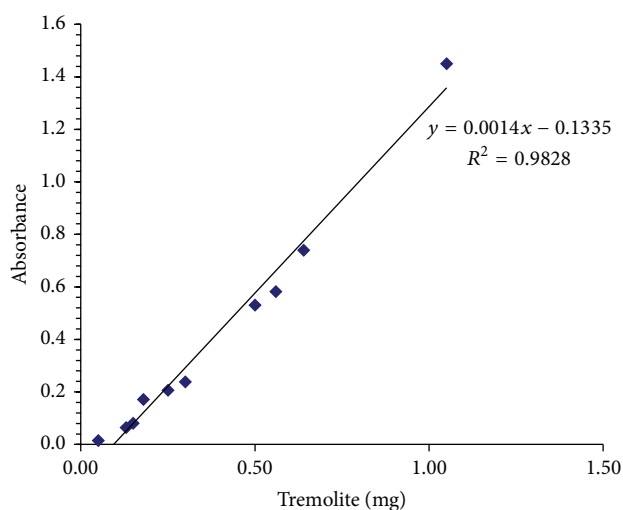


FIGURE 3: The linear calibration graphs calculated for the first analytical band of tremolite between  $3700$  and  $3650 \text{ cm}^{-1}$ , with the parameters of their linear regression equation.

concentration (on the order of  $0.2$ – $0.3\%$ ) in a KBr pellet shows a linear correlation with concentration.

The linear calibration graphs calculated for the three analytical bands of tremolite are shown in Figures 3, 4, and 5 with the parameters of their linear regression equation. The amount of tremolite in the test samples was quantified by using the curve parameters of the three analytical bands.

Figures 6, 7, and 8 show IR absorption band of three size class samples.

The unknown concentration of tremolite in the test samples was determined by using the area-weighted average  $X_{\text{best}}$  with a standard error  $\sigma_{\text{best}}$ .

Tables 2, 3, and 4 show the weight concentrations of tremolite detected for each size class sample and each analytical band with the corresponding standard error.

The quantitative analysis of tremolite using OH stretching band between  $3700$  and  $3650 \text{ cm}^{-1}$  and the Si-O-Si and O-Si-O stretching band between  $1200$  and  $900 \text{ cm}^{-1}$  gave high

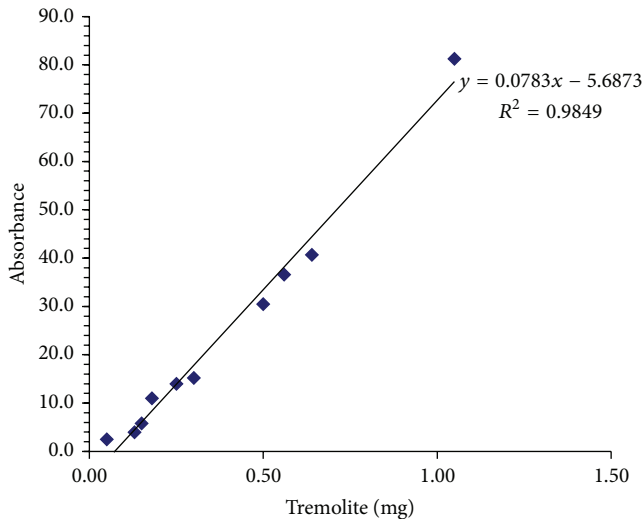


FIGURE 4: The linear calibration graphs calculated for the second analytical band of tremolite between 1200 and 900  $\text{cm}^{-1}$ , with the parameters of their linear regression equation.

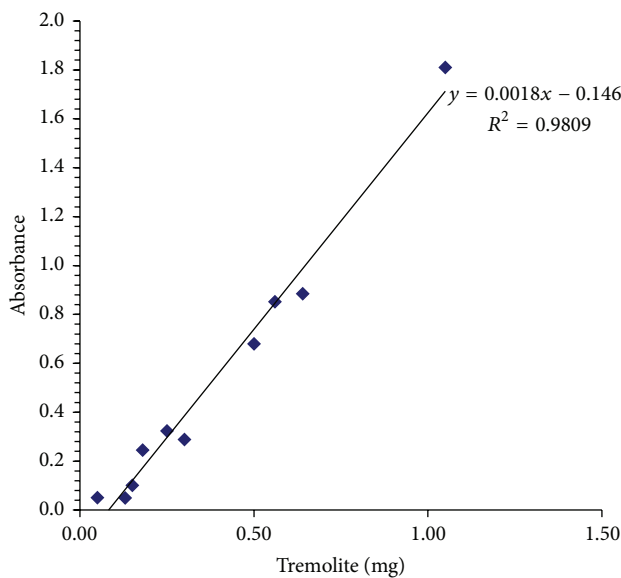


FIGURE 5: The linear calibration graphs calculated for the band around 756  $\text{cm}^{-1}$  of tremolite, with the parameters of their linear regression equation.

amount of tremolite, with unlikely values for medium size class sample.

In the medium class size sample, the absorption band between 3700 and 3650  $\text{cm}^{-1}$  gave an amount of tremolite above 100%, improbable value. This suggested that agglomeration of fibers or particles of interfering minerals with asbestos was a significant source of error.

The three size class samples were observed by optical microscopy (DM2500P, Leica) and the fine size class sample had the appearance of fine fibrils and needle-like fibers with diameter around 0.2  $\mu\text{m}$  having optical properties consistent with tremolite. Similar fibers were found also in the other

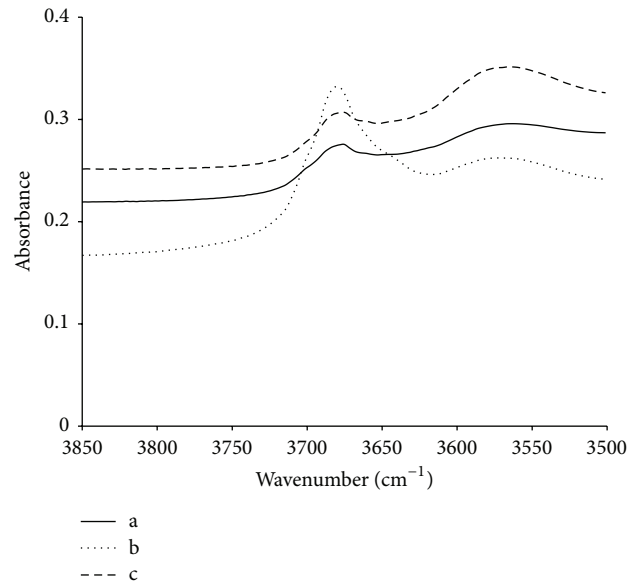


FIGURE 6: The first IR absorption band of three size class samples: a, fine, b, medium, and c, large.

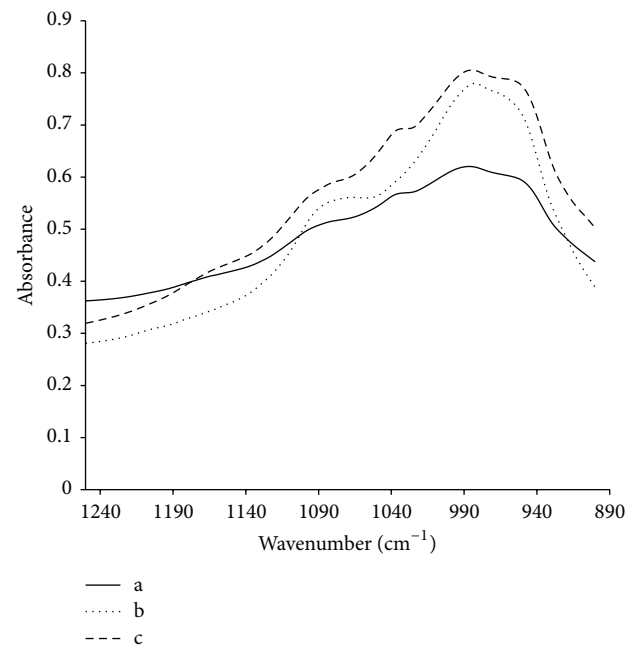


FIGURE 7: The second IR absorption band of three size class samples: a, fine, b, medium, and c, large.

two samples. SEM analysis confirmed the fibers nature as tremolite in three samples. An example of fibrous tremolite is shown in Figure 9(a) with corresponding EDS (Figure 9(b)).

The medium size class sample differed from other two samples for the presence of several prismatic fibers and acicular fibers having optical properties different from tremolite. Optical properties of such fibers were consistent with antigorite and SEM analysis gave the only presence of magnesium

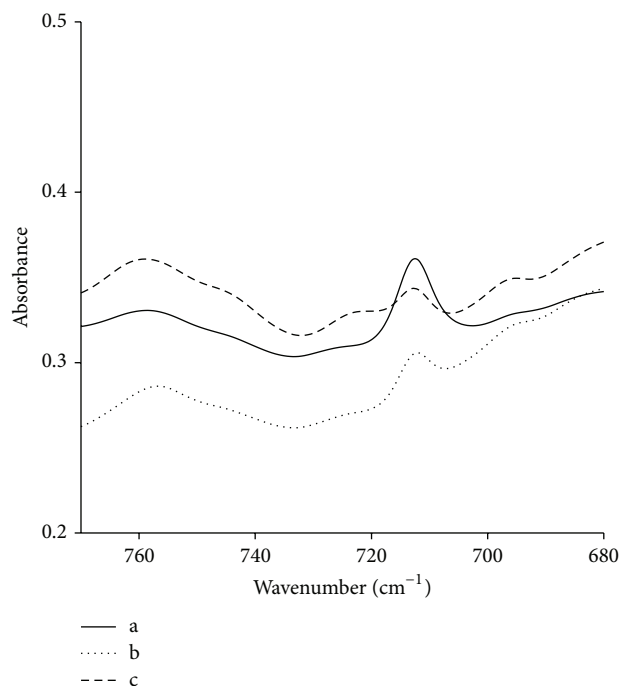


FIGURE 8: The third IR absorption band of three size class samples: a, fine, b, medium, and c, large.

TABLE 2: Weight concentrations of tremolite detected for the first analytical band and for every set of samples.

The first band 3700–3650 $\text{cm}^{-1}$			
Weight concentration ( $\mu\text{g}/\text{mg}$ )	$\sigma_{\text{best}}$	Weight%	+/-
Fine sand size			
161.5	0.1	16.15	0.01
Medium sand size			
>1000	>20	>100	—
Large sand size			
230	8	23.02	0.27

TABLE 3: Weight concentrations of tremolite detected for the second analytical band and for every set of samples.

The second band 1222–900 $\text{cm}^{-1}$			
Weight concentration ( $\mu\text{g}/\text{mg}$ )	$\sigma_{\text{best}}$	Weight%	+/-
Fine sand size			
147.3	0.6	14.73	0.06
Medium sand size			
303.3	0.2	30.33	0.01
Large sand size			
315.5	6.2	31.6	0.3

and silicon. Figure 10 shows an example of prismatic fibers in the medium class size sample.

The obtained IR spectrum of the medium size class sample confirmed the presence of antigorite.

TABLE 4: Weight concentrations of tremolite detected for the third analytical band and for every set of samples.

The third band 756 $\text{cm}^{-1}$			
Weight concentration ( $\mu\text{g}/\text{mg}$ )	$\sigma_{\text{best}}$	Weight%	+/-
Fine sand size			
66.5	0.3	6.65	0.03
Medium sand size			
85.71	0.14	8.57	0.01
Large sand size			
77.48	0.09	7.75	0.01

In the region of OH stretching vibration, the 3678  $\text{cm}^{-1}$  peak appeared and a shoulder was also evident at 3697  $\text{cm}^{-1}$  [48–50] (Figure 6).

In the region of tetrahedral sheet stretching vibration, the most evident band occurred at 980  $\text{cm}^{-1}$  and another one at higher energy, near to 1080  $\text{cm}^{-1}$  [48, 50, 51] (Figure 7). The presence of antigorite was detected also for other two size class samples but in smaller quantities.

#### 4. Conclusions

Spectroscopic methods are widely used in the quantitative analysis of asbestos. The validity of the quantitative information depends on the history of the sample.

The quantitative analysis of tremolite using OH stretching band between 3700 and 3650  $\text{cm}^{-1}$  and the Si-O-Si and O-Si-O stretching band between 1200 and 900  $\text{cm}^{-1}$  gave high amount of tremolite, with unlikely values for medium size class sample.

The sharp peak of the hydroxyl doublet at 3690  $\text{cm}^{-1}$  is used traditionally for quantitative analysis of chrysotile asbestos. In this range, other serpentine minerals of related structure (e.g., kaolinite, antigorite) show very similar absorption bands and their presence overestimates any asbestos determination based on the infrared absorption in this region.

The major broad bands of silicate materials, quartz and chlorite, are present also in the 1200–900  $\text{cm}^{-1}$  range and it is impossible to select a tremolite characteristic absorption peak relatively free from interference. The unlikely values of tremolite obtained for the medium size class sample are due to the abundant presence of antigorite.

Examination of tremolite fibers detectable by optical microscopy allowed a rough visual estimation that made the amount of tremolite obtained using IR absorption peak at 756  $\text{cm}^{-1}$  (fingerprint region) more likely. The latter is not affected at the interfering minerals and can be used for quantitative analysis in sample coming from ophiolitic deposits.

It should be noted that the medium size class sample has the higher content of tremolite. The optical microscopy observations performed on all samples had visually confirmed it. Tremolite was present both as acicular and as prismatic. We do not know how many of these prismatic tremolites were produced during comminution process. It

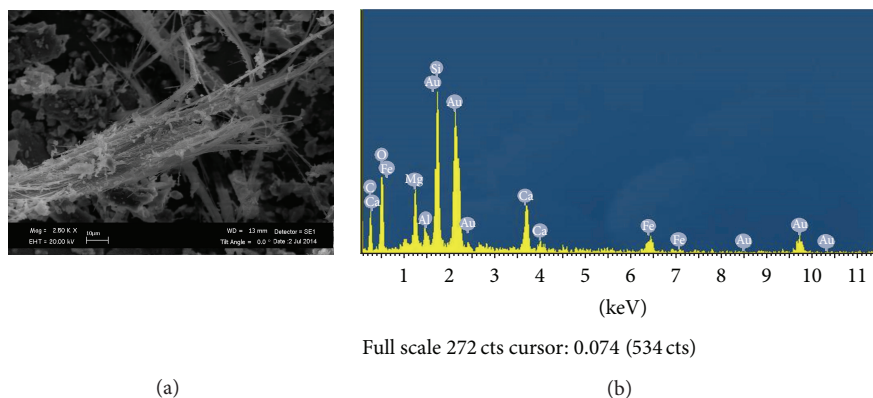


FIGURE 9: Example of fibrous tremolite (a) with its EDS spectrum (b) found in the fine class size sample.

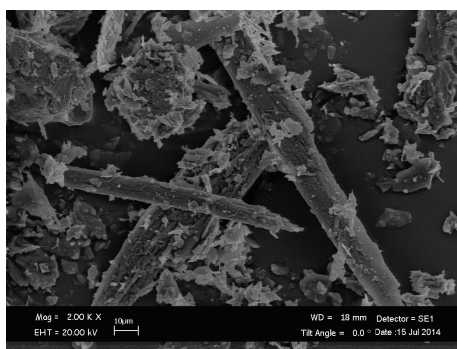


FIGURE 10: Some prismatic fibers found in the medium class size sample.

is known that comminution of amphibole minerals during crushing or grinding may produce structures known as cleavage fragments, some of which may appear as acicular or needle-like crystals because of the way in which amphibole minerals cleave. These structures are considered nonasbestiform fibers. OSHA defines cleavage fragments as mineral particles formed during ore milling, characterized by relatively parallel sides and low aspect ratios [52]. The question of the relative toxicity of asbestiform tremolite and fibrous fragments (cleavage fragments) or prismatic tremolite and their significance as possible health hazards is not yet resolved. The precautionary principle prompts the use of the same prevention measures with nonasbestiform fibres (cleavage fragments) as with asbestiform fibres of the same size.

IR technique is capable of analysing the total, fibrous plus nonfibrous, components; only electron microscopy could be expected to identify and determine the fibrous amphibole content.

### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

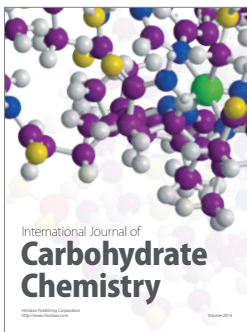
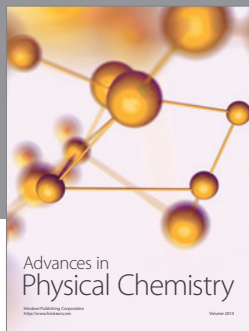
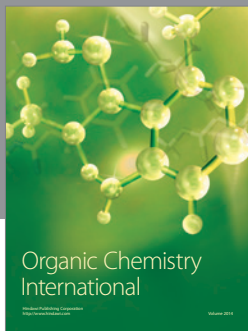
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