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CHARACTERIZATION OF NATURAL MATERIALS CONTAINING N.O.A. (NATURALLY OCCURRING ASBESTOS): THE PROBLEM OF QUANTIFICATION

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ABSTRACT

The quantification of NOA (Naturally Occurring Asbestos) in a rock or soil matrix is complex and subject to numerous errors. Current legislation in Italy (DM 6/9/94) defines the threshold of 1000 mg/kg of asbestos fibers on the total of the material, beyond which the material is considered as hazardous waste.
The objective of this study is to compare two fundamental techniques for analysis: the first one is based on analysis with a Phase Contrast Optical Microscope (PCOM) while the second one requires the use of a Scanning Electron Microscope (SEM).
In order to provide a sufficiently reliable uncertainty of the PCOM methodology, 10 repetitions of the analysis by two different operators on two selected samples were carried out.
Another important part of this study is the comparison between SEM and PCOM analysis. Over 100 tests have been performed to date on natural samples from both cores and from excavation materials with the use of the two techniques. A good correlation between the results obtained has been found.
One of the major aims of this work is to encourage a technical discussion which includes all the countries affected by the problem of asbestos in order to adopt common technical specifications and guidelines.

KEYWORDS : NATURALLY OCCURRING ASBESTOS, QUANTIFICATION, PCOM , SEM, MICROSCOPY

1. INTRODUCTION

Fibrous minerals are common in nature but asbestos minerals are rare. All the asbestos minerals share the same common characteristic the habit of crystallization as polyfilamentous fiber bundles [1]. Asbestos exposure is linked to adverse human health effects including asbestosis and mesothelioma [2,3,4]. Asbestos, in commercial terms, as is decleared in Italian law D.M. 06/09/1994, generally includes minerals with asbestiform characteristics, separable into thin fibers or in bundles that are normally grouped into two different set varieties: serpentine of asbestos (chrysotile) and amphibole of asbestos (tremolite, antophillyte, actinolite, amosite e crocidolite). The amphibole minerals can occur in habits which are not polyfilamentous and therefore are not classified as asbestos
The presence of Naturally Occurring Asbestos (NOA) is one of the biggest dangers to deal with during excavations and tunnelling. The classification of a material as a potential hazardous waste plays a key role especially during construction projects because of the volume of material involved and the consequent problems related to the treatment of the material itself. Moreover it is also extremely important to perform the analysis on exploratory cores in order to plan the advance of the work and adopt proper safety measures.
The investigated area is located in the south of the Piedmont region characterized by geological formations that can contain asbestos such as ultramafic and ophiolitic rock but also sedimentary rock like marls. Asbestos fibres are released during digging, crushing and transport operations and by weathering processes. It is for this reason that it is essential to characterize the area during excavation work from a geological point of view in the geognostic surveys of pre-excavation, in order to reduce and/or mitigate the risks for workers and the environment [5].
The Italian law in force (dm 6/9/94) [6] define a threshold beyond which the material is considered as hazardous waste. This value is a concentration of asbestos fibers of 1000 mg/kg (0,1%). (DM 6/9/94).
Italian law in force contemplate the use of the following instruments: X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Phase Contrast Optical Microscopy (PCOM). DRX and FTIR are suitable for analysis on manmade materials but are not suitable for analysis of natural materials because of their low detection limit (1%).

The objective of this study is to compare two fundamental analysis techniques: the first one is based on Phase Contrast Optical Microscopy (PCOM) while the second one requires the use of a Scanning Electron Microscope (SEM). In the first case, each asbestos mineral has its characteristic interference colour that make it possible to identify the mineral; in the second case, the fibrous mineral are characterized by scanning electron microscopies combined with energy dispersive spectrometry. The first part of this work is a validation of the PCOM methodology for quantitative determination of asbestos while the second is a comparison on more than 120 samples between PCOM and SEM analysis.

2. METHODS

2.1 PCOM METHODOLOGY

The Phase Contrast Optical analysis is based on an optical principle: using a Polarized Light Microscope (PLM) equipped with a device for phase contrast it is possible to recognize asbestos fibers thanks to the variation of refraction index which depends on the wavelength of the incident light. If fibers are placed in specific high-dispersion liquids they show typical chromatic effects that allow their identification. The evaluation of the correct refraction index is based on three parameters: luminosity, colour and birefringence [7]. In the following figures (Fig. 1 and Fig. 2) some examples of how asbestos is recognizable in its appropriate oil in different grain size classes using PCOM observation.

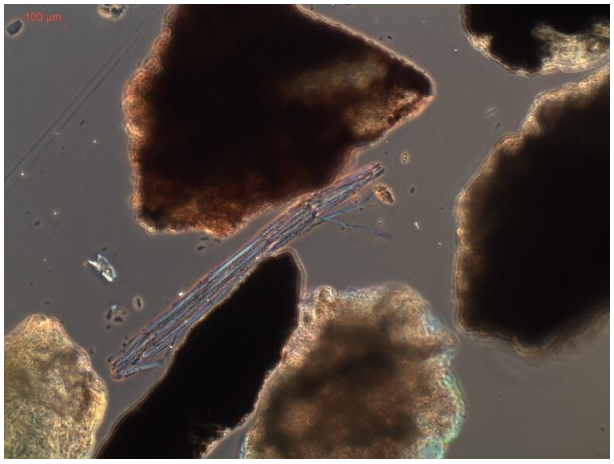

Figure 1: 0,6-0,3 mm ; chrysotile in 1.550 oil

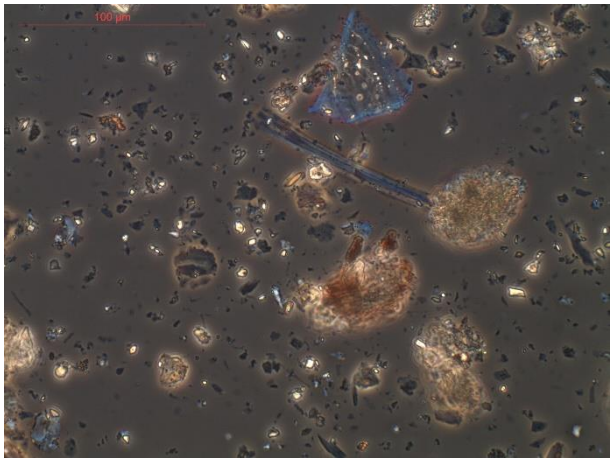

Figure 2: <0.075 mm ; chrysotile in 1.550 oil

The methodology based on PCOM involves the coning and quartering of the starting sample. The purpose of the treatment is also to facilitate the optical microscope examination, by division into granular fraction. The procedure begins with the mild milling of the starting sample, with a mass of around 100-120 gr. The powder material is classified by wet sieving in different grain size classes in order to select the powder according to different particle size: Large (0.6 or 0.3 mm), medium (0.3 or 0.15), small (0.15 or 0.075) and the last (less than 0.075). The powder retained from the sieve is filtered using a quantitative analysis filter and then is dried in an oven at 150 C°.

For each class one slide with oil of a known refractive index is prepared in order to recognize the asbestos fibers. After the preparation of a set of slides (one slide for each grain size class) each slide is analyzed with the PCOM. The total area of the slide of the large class is observed under 10x objective. For the others classes the investigation concerns 25 field view, instead the total area of the slide.

After recognition and measuring (length, diameter) of the asbestos fibers, the weight of observed asbestos is calculated by multiplying the volume with its density (2,6 gr/dm^3 for asbestos of serpentine and 3,0 gr/dm^3 for asbestos of amphibole). The determination of the asbestos content is obtained by the relationship between the weights of the fibrous component compared to the one granular. The total concentration of asbestos (Ca_{tot}) is obtained by the sum of asbestos weights in each class (Mc_i) multiplied by the weight of the class itself (Ci) and then divided by the total weight of the sample (M_{tot})

$$Ca_{tot} = \frac{M_{c1}c_1 + M_{c2}c_2 + \cdots + M_{c4}c_4}{M_{tot}}$$

2.2 SEM METHODOLOGY

The Scanning Electron Microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The SEM used for asbestos analysis must be equipped for energy-dispersive X-ray spectroscopy (EDX) in order to ensure the correct characterization of the asbestos by estimating the abundance of elements in selected point in the sample. The resolution of the instrument is very high (0.5 μn) but there is not a deep correlation between instrumental resolution and methodology resolution.
The treatment of the sample starts with an intensive milling of the quartered sample. A weighed part of the sample is put in a beaker with 200 mL of water and it is shaken using an ultrasonic bath. Part of the solution is recovered by filtering on a vacuum pump equipped with a nitrate of cellulose membrane. The next step consist in mounting the filter on a SEM stub and coating with Au by cathodic sputtering. The analysis is usually carried out using a 1000x magnification on a selected number of microscopic fields. The asbestos fibers are identificated by their morphology, their chemical composition by X-ray dispersion energy spectroscopy, and then measured (length and diameter)[8].
The concentration of asbestos is calculated (as the PCOM methodology) by the relationship between the weights of the fibrous component compared to the one granular.

3. RESULTS

3.1 UNCERTAINTY OF THE PCOM METHODOLOGY

In order to determine the uncertainty and validity of the PCOM analysis, 10 tests on the same sample were performed and two different operators analyzed the areas required for the analysis. In this way, it was estimated the uncertainty of the methodology and the operator-related error. The asbestos content Ca_{tot} [mg/Kg] of 10 samples obtained by the operator 1 and operator 2 following the methodology described above, is shown in the following table (Table 1)

	Operator 1	**Operator 2**
	Ca_{tot} (mg/kg)	Ca_{tot} (mg/kg)
Sample 1	546	393
Sample 2	575	406
Sample 3	449	514
Sample 4	423	515
Sample 5	398	370
Sample 6	567	485
Sample 7	228	370
Sample 8	326	380
Sample 9	405	294
Sample 10	414	404
Mean (μ)	433	413
Standard deviation (σ)	109	71

Table 1

The measurements are expressed by two values: mean (μ) and standard deviation (σ).

$$Operator\ 1 = 433{,}1 \pm 109{,}19$$

$$Operator\ 2 = 413{,}1 \pm 71$$

The statistical analysis has proved that the averages of the data obtained by the two operators are very similar to each other and also the standard deviation presents low values (Operator 1 with $\mu_1 \pm \sigma_1 = 433 \pm 109{,}7$ and Operator 2 with $\mu_2 \pm \sigma_2 = 413 \pm 70{,}9$). Operator 1 shows a measurement uncertainty of 25%, while operator 2, only 17%. This is probably due to the fact that operator 1 has less experience in recognizing asbestos fibers than the operator 2 and to the variability of the distribution of the material on the slide.

3.2 COMPARISON BETWEEN PCOM AND SEM

After the previous validation test of the technique, tests were conducted using both the PCOM methodology and the SEM methodology to evaluate theirs comparability. More than 120 analyzes of natural samples were performed, the results are presented in the following chart (Fig. 3).

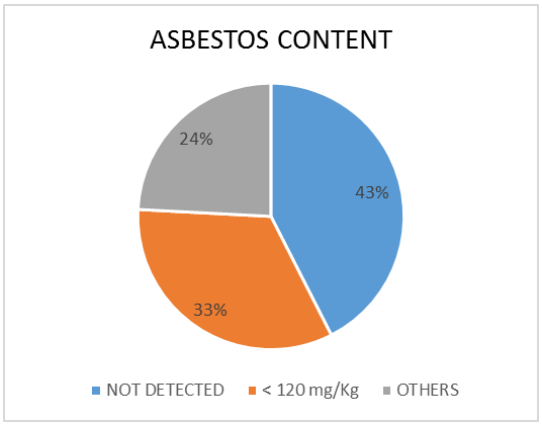

Figure 3: Asbestos content

In 43% of the analyzed samples it has not been detected the presence of asbestos with both analysis techniques. In 33% of cases, however, both techniques yielded a value minor than 120 mg / Kg , which is a concentration commonly adopted as analytical detection limit (dm 94). The remaining 24% of the tests carried out are divided into three different cases as shown in the following chart (Fig. 4).

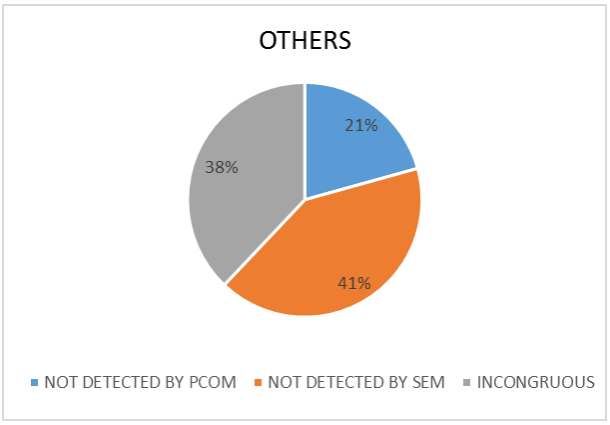

Figure 4: different cases of inconsistent data

In 41% of the cases, electronic microscopy did not detect asbestos, whereas 21% of cases did the opposite. The incongruous values are the remaining 38% corresponding, however, to less than 10% of the total of the tests

The main reasons for inconsistent data can be:

- Asbestos not detected by PCOM: the higher resolution of SEM allows to detect even the finer fibers, but it is more probable to be an incorrect interpretation of the type of mineral observed. Antigorite and chrysotile provide very similar spectra and electronic microscopy recognition is almost exclusively based on morphology so antigorite it can easily be mistaken for chrysotile.

- Asbestos not detected by SEM : in case of low quantity of asbestos the representativeness of the sample may be unsatisfactory
- SEM higher than PCOM: in this case the problem could be the uncorrect recognition of chrysotile (from antigorite) but there is another important problem: the presence of out-of-scale objects. When analyzing a small amount of material the presence of a small bundle of fibers may appear to count heavily in the final result although it is not representative of the sample.
- PCOM higher than SEM : better representativeness of the sample

4. DISCUSSION AND CONCLUSIONS

The results obtained in the comparison tests demonstrate that the Phase Contrast Optical Microscopy technique is effective for quantitative analysis of asbestos content even in the presence of low concentrations. Moreover PCOM has many advantages over SEM as well:

- **REPRESENTATIVENESS:** the PCOM analysis is more representative than SEM. It is more unlikely to make macro-errors (especially in the case of a standardization of procedures)
- **REPRODUCIBILITY**: both techniques provide data on the reproducibility of the same order of magnitude
- **COST:** the PCOM requires less costly equipment and less maintenance.
- **TIME :** times for the analysis (including the preparation of the sample) are similar
- **SUSTAINABILITY :** the PCOM analysis is less stressful for the operator: preparation of the sample is longer but microscopic analysis time is shorter.

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