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

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The fibrous habit of these minerals links the asbestos exposure to adverse human health effects.

The presence of Naturally Occurring Asbestos is one of the biggest dangers to deal with during excavations and tunnelling.

Asbestos, which is an accessory or occasional mineral of secondary origin, can be found into ophiolites rocks (serpentinite-metabasite..) also called green stones.
The threshold beyond which the material is considered as hazardous waste is a concentration of asbestos fibres of 1000 mg/kg. (152/2006)

<table>
<thead>
<tr>
<th>TECHNOLOGY</th>
<th>USE</th>
<th>DETECTION LIMITS</th>
<th>RESOLUTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>DRX</td>
<td>• Asbestos weight determination</td>
<td>Concentration &gt;1%</td>
<td></td>
</tr>
<tr>
<td>IR</td>
<td>• Asbestos weight determination</td>
<td>Concentration &gt;1%</td>
<td></td>
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</tbody>
</table>
| SEM        | • Asbestos weight determination  
• Determination of airborne fiber number concentration | Concentration > 0,1% (quantitative) < 0,01%  
Concentration > 0,1% (semi-quantitative) | 5 nm |
| PCOM       | • Asbestos QUALITATIVE determination  
• Determination of airborne fiber number concentration (not used) | Concentration < 1%  
Fiber diameter > 0,75 μm | 0,2 μm |
Mild grinding of a rock sample

Classification of the sample (100 gr) into 5 grain size classes (< 2 mm) by wet sieving

Preparation of the set of slides (1 set = 4 slides) using oils with note refractive index
The observation and measuring (length, diameter) of the asbestos fibers on a fixed number of fields is based on optical properties as pleoclorism and interference color.

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

PCOM is used in the US (ASTM D7521 – 16)
The methodology is based on point-count.
“This test method has an analytical sensitivity of 0.25 % by weight with optional procedures to allow for an analytical sensitivity of 0.1 % by weight.”

Chrysotile
> 0.075 mm; 10x.
The measurements are expressed by two values: mean ($\mu$) and standard deviation ($\sigma$).

Operator 1: 433.1 ± 109.19
Operator 2: 413.1 ± 71

<table>
<thead>
<tr>
<th>Sample</th>
<th>$Ca_{tot}$ (mg/kg)</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>546</td>
<td>393</td>
</tr>
<tr>
<td>Sample 2</td>
<td>575</td>
<td>406</td>
</tr>
<tr>
<td>Sample 3</td>
<td>449</td>
<td>514</td>
</tr>
<tr>
<td>Sample 4</td>
<td>423</td>
<td>515</td>
</tr>
<tr>
<td>Sample 5</td>
<td>398</td>
<td>370</td>
</tr>
<tr>
<td>Sample 6</td>
<td>567</td>
<td>485</td>
</tr>
<tr>
<td>Sample 7</td>
<td>228</td>
<td>370</td>
</tr>
<tr>
<td>Sample 8</td>
<td>326</td>
<td>380</td>
</tr>
<tr>
<td>Sample 9</td>
<td>405</td>
<td>294</td>
</tr>
<tr>
<td>Sample 10</td>
<td>414</td>
<td>404</td>
</tr>
<tr>
<td>Mean ($\mu$)</td>
<td>433</td>
<td>413</td>
</tr>
<tr>
<td>Standard deviation ($\sigma$)</td>
<td>109</td>
<td>71</td>
</tr>
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</table>
Dispersion of an amount of a powder sample with a granulometry between 10 and 100 µm in a known volume of a solution

Filtration of a part of the suspension on a polycarbonate filter

Observation, at 1000-2000X, of a number of microscopic fields adapted to the limit of detection, in order to analyse 1-4 mm²

Scanned image by SEM (mag = 4000 X)

Scanned image by SEM (mag = 16000 X)
Identification of the asbestos fibers by X-ray dispersion energy spectroscopy (EDS), and measurement of their dimensions.

Determination of the asbestos content obtained by the relationship between the weights of the fibrous component compared to the granular one.
SEM vs PCOM

- Amount of analysed sample portion: 0.008 mg SEM; 150 mg PCOM
- Grinding
- Distinguishing between chrysotile and antigorite
SEM vs PCOM

- 43% Not Detected
- 33% <120 mg/Kg
- 24% Others

- 41% Not Detected by SEM
- 38% Incongruous
- 21% Not Detected by PCOM
- Others
DISCUSSION

ASBESTOS NOT DETECTED BY PCOM:

ASBESTOS NOT DETECTED BY SEM:

SEM HIGHER THAN PCOM:

PCOM HIGHER THAN SEM:

Thin fibers / Antigorite
Representativeness of the sample
Out of scale objects / Antigorite
Representativeness
PCOM ADVANTAGES

Representativeness
Reproducibility
Cost
Time
Sustainability

CONCLUSIONS
Thank you for your attention