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New Detoxification Processes For Asbestos Fibers in the Environment

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Abstract

Airborne asbestos fibers are associated with many serious detrimental effects on human health while the hazard posed by waterborne fibres is still object of debate. Adopting a precautionary principle, however, asbestos content in water should be kept as low as possible and polluting waters with asbestos should be avoided. We have recently reported a method for the decontamination of asbestos-polluted waters or landfill leachates from chrysotile which combines power ultrasound (US) with oxalic acid (Ox), an acidic chelating molecule (Turci et al., 2008). In the previous paper, the occurrence of antigorite, a polymorph of serpentine – the mineral group encompassing chrysotile asbestos – acted as confounding factor for complete removal of chrysotile from water. We report here new insights on the effect of US+Ox on a rather pure chrysotile asbestos from Val Malenco, Italian Central Alps. In the absence of mineral contaminants, the study evidenced a more rapid removal of pure chrysotile from water with respect to the previous specimen. After only 12 hrs of combined US+Ox acid treatment, imaging (SEM) of mineral debris indicates the complete loss of fibrous habit, crystallography and vibrational features of chrysotile are undetectable (XRD and micro-Raman) and elemental analysis indicates a very low Mg/Si ratio, i.e. the loss of the

brucitic layer in chrysotile (XRF). The occurrence of few nanometric rod-shaped debris, observed in the previous study and tentatively recognized as serpentine antigorite, are now demonstrated to be made of amorphous silica, providing further assurance about the safety of treated product. The effectiveness of the proposed method in detoxifying waterborne chrysotile asbestos fibres is here confirmed.

Introduction

Airborne asbestos fibers – i.e. six different fibrous silicates, namely: chrysotile, amosite, crocidolite, tremolite, anthophyllite and actinolite – are definitely associated with serious detrimental effects on human health, i.e. asbestosis, a debilitating and often fatal lung disease, and such malignancies as lung cancer and pleural mesothelioma, which may develop several decades after exposure (Mossman et al., 1990; Mossman, 1993). Although the carcinogenicity of asbestos minerals had been mainly reported as an occupational issue (International Agency for Research on Cancer, 1977), a growing concern has been recently expressed on the dispersion of natural occurring asbestos (NOA) in the environment (EPA, 2003; EPA, 2005; Kazan-Allen, 2005; Pan et al., 2005; Dearwent et al., 2006; Van Gosen, 2007), following reports of multiple exposure scenarios, e.g. in Libby, MT - US (Peipins et al., 2003; McDonald et al., 2004; Lee et al., 2008). Naturally occurring mineral fibers may be found both in large commercial deposits, close to active or abandoned mines, and in small non-economic occurrences throughout the mountains (Lee et al., 2008). Asbestos fibers are widely distributed also in the aqueous environment and may eventually contaminate drinking water through different scenarios: i) by weathering from natural deposits such as serpentine, ii) by release from asbestos-cement pipes, and iii) from processes associated with mining and production of certain types of iron ore (Cantor, 1997). The waterborne asbestos concentration is very variable and ranges from 10^4 fibers/l to over 10^{11} fibers/l. According to World Health Organization (WHO) there is no consistent evidence that ingested asbestos is hazardous to health (WHO, 2006). However, in some recent studies, likely not included in WHO survey, a correlation between

gastrointestinal cancer and asbestos ingestion was established (Webber, 1993; Kjaerheim et al., 2005). Furthermore, the occurrence of asbestos in irrigation water may generate aerosolized fibers to a concentration which is, despite some reliable attempts to standardize airborne fibers per asbestos mass (e.g., EPA, 1980), quite unpredictable. The Italian Regional Agency for Environmental Protection (ARPA – Piemonte) has accepted the conversion factor proposed by Webber and coworkers (1988) of ca. 2 airborne fibers per liter of indoor air produced by 100,000 fiber/l in water. Following a precautionary principle, US Environmental Protection Agency (EPA) indicates a protective standard of $7 \cdot 10^6$ fiber/L based only of fibers with length $> 10 \mu\text{m}$. If one considers that 80-95% of waterborne asbestos fibers is shorter than $1 \mu\text{m}$ (Cantor, 1997) and that U.S. EPA TEM analysis method 100.1 consider fibers longer than $0.5 \mu\text{m}$, the EPA standard clearly underestimate waterborne asbestos occurrence. In a nutshell, since asbestos fibers, both airborne and waterborne, remain in the environment for decades or longer (<http://www.epa.gov/ogwdw/dwh/c-ioc/asbestos.html>) an environmentally responsible approach should be enforced in order not to disperse asbestos in surface and ground waters. The methods currently adopted to abate waterborne asbestos amount are conventional water treatments such as coagulation/filtration and direct and diatomite filtration. However, the filtering process of asbestos fibers requires low porosity filters, which are prone to be clogged by organic matter and other suspended inorganic particles. Furthermore, if asbestos filtration applies with a certain success to small and controlled systems such as drink-water supply or landfill leachate (e.g., EU project FALL, LIFE03 ENV/IT/00323), but are hardly adapted to bigger scenarios such as the natural release of fibers from asbestos spoil dumps and excavated faces in formerly quarried or mined sites or the dumping of mining tailings into river or lakes (e.g., San Vittore, Balangero, Italy (Buzio et al., 2000) and MABE, Kozani, Greece (Anastasiadou and Gidarakos, 2007)). To cope with such issue, an innovative method based upon synergistic action of power ultrasound and acidic metal chelator was recently proposed (Turci et al., 2007b) and tested upon a commercial sample of chrysotile fiber form Balangero mine (Turci et al., 2008).

In the present paper, current knowledge on the effectiveness of the ultrasound (US) and oxalic acid (Ox) association in detoxifying chrysotile fiber is expanded and a second, rather pure, type of chrysotile is tested for dissolution. This asbestos sample, from Val Malenco, Italian Central Alps, has been thoroughly characterized by means of several physico-chemical techniques and resulting data were used as reference to evaluate the efficacy of US-Ox-driven remediation of waterborne chrysotile. For comparison purpose, fibers were subjected to US treatment alone (in pure water suspension) or leached by oxalic acid solution. Following treatments, morphology (SEM) and crystallographic pattern (XRD) of residuals were assessed in order to check fibrous habit and the occurrence of serpentine or newly-formed phases. Spectroscopic techniques (micro-Raman and XRF spectroscopy) were performed for confirming crystallographic findings and quantify chrysotile dissolution by evaluating relative amount of structural elements.

Materials and methods

Asbestos

Two different type of chrysotile were selected:

1. A relatively pure commercial sample (classified 6D) from the San Vittore disused asbestos mine (Balangero, Italy), kindly provided by R.S.A., the society managing the mine.
2. A pure chrysotile specimen, with limited iron contamination, from Val Malenco (Italian Central Internal Alps).

See previous works for further details (Turci et al., 2008; Favero-Longo et al., 2009)

Reagents

All reagents were purchased by Sigma–Aldrich. For all experiments ultrapure Milli-Q (Millipore, USA) water was used.

Sono-chemical treatment of chrysotile

Chrysotile asbestos fibres (500 mg) were suspended in 50 ml of ultrapure water (Milli-Q) or in 50 ml of 0.5 M oxalic acid solution (fibres/solution ratio = 10 mg/ml) and placed into the US reactor. The cavitating tube frequency was set to 19.2 kHz and stabilized with an automated adjustment device (frequency hook). The fibres were sonicated for 12 or 21 h at an input power of ca. 150 W. The reactor was cooled in order to keep suspension temperature below 50 °C and it was sealed in order to minimize evaporation of the liquid. In order to discriminate between the effect of acidic/chelating leaching and sonication, chrysotile (10 mg/ml) was also sonicated in pure water at the same experimental conditions or was leached with oxalic acid at 50 °C under mild mixing in a thermostatic vessel.

Following the sono-chemical treatments, the fibres were separated from the liquid by filtration through CA membranes (pore size 0.45 µm), rinsed and dried. The solid residues were subjected to surface area measurements (BET) and structural analysis of the bulk (SEM imaging, XRD and micro-Raman spectroscopy) and elemental analysis (XRF) were performed.

SEM-EDS

Scanning electron microscopy (SEM) observations were performed on gold-coated fibres with a Stereoscan 410 Leica equipped with Oxford Link EDAX. The accelerating voltage was 15 kV and the secondary electron detector was used.

XRD

X-ray diffraction analyses were performed on the solid residues with a Phillips PW 1830, with θ -2 θ geometry and Cu K α radiation. The patterns obtained were compared with those contained in the J.C.P.D.S. (Joint Committee of Powder Diffraction Standard) archives.

Micro-Raman

Micro-Raman spectra were acquired using an integrated confocal Raman system which includes a micro-spectrometer Horiba Jobin-Yvon HR800, an Olympus BX41 microscope and a CCD air-cooled detector operating at -70 °C. A polarised solid state Nd laser operating at 532.11 nm and 80 mW was used as the excitation source. Correct calibration of the instruments was verified by measuring the Stokes and anti-Stokes bands and checking the position of the Si band at 520.7 cm⁻¹. To optimize the signal to noise ratio, spectra were acquired using an integration time of about 100 seconds for each spectral region.

micro-XRF

All samples were analyzed using an EDAX Eagle III energy-dispersive μ XRF spectrometer equipped with a Rh X-ray tube and a polycapillary exciting a circular area of nominally 30 μ m diameter. Data collection occurred at each point for 45 s detector live time, with X-ray tube settings adjusted for 30% dead time. About $1 \cdot 10^6$ Cps were counted per scan. At least 25 points were collected for each sample.

Surface area

The well known BET method (Brunauer et al., 1938) was adopted to measure the specific surface area (SSA) of the natural and modified samples. A fully automated surface area analyzer (ASAP 2020, Micromeritics – USA) was used.

Results and discussion

Morphological analysis

Fibrous habit is widely recognized as a key-factor in asbestos toxicity. Airborne fibers are usually not considered toxic for humans if their length is lower than 5 μ m. US-EPA standard for waterborne

asbestos fibers is calculated considering only fibers longer than 10 μm . The micro-morphological features of untreated and sono-chemically treated chrysotile sample are shown in Figure 1.

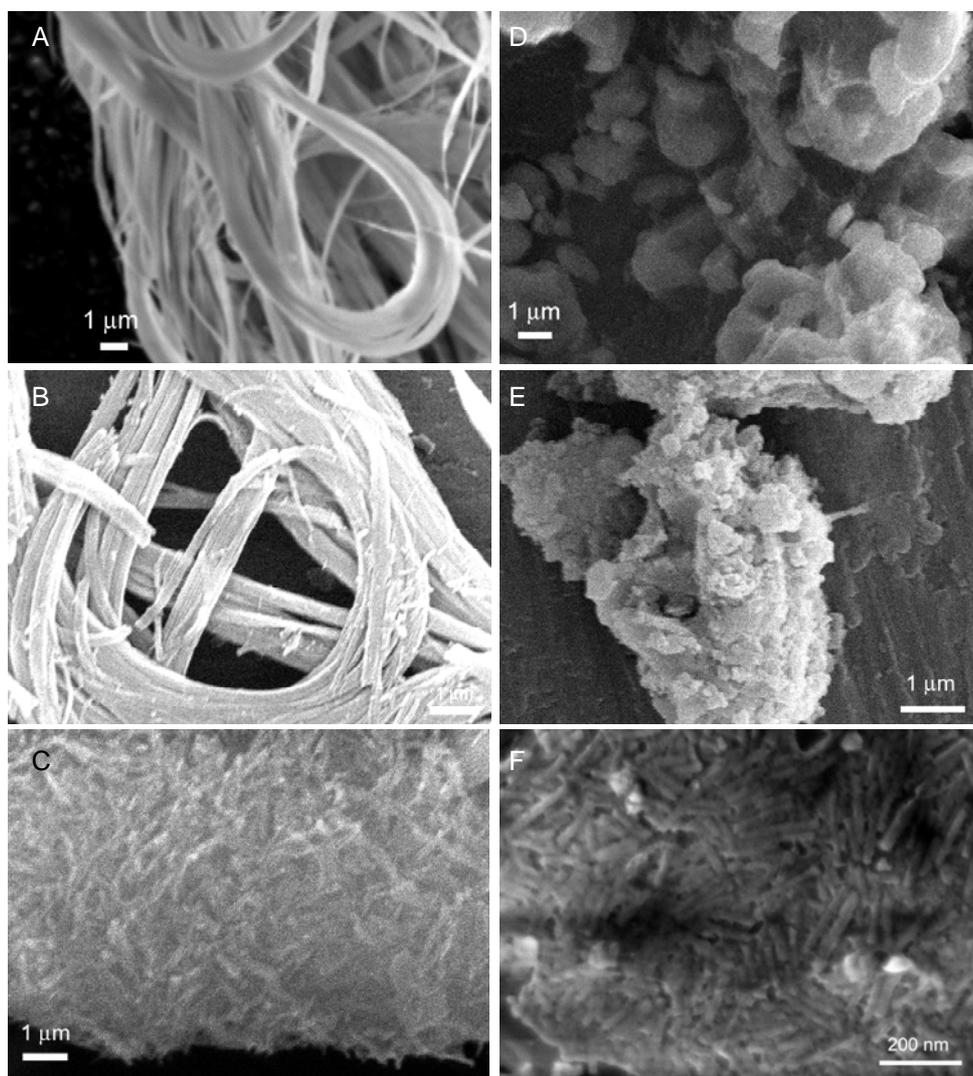
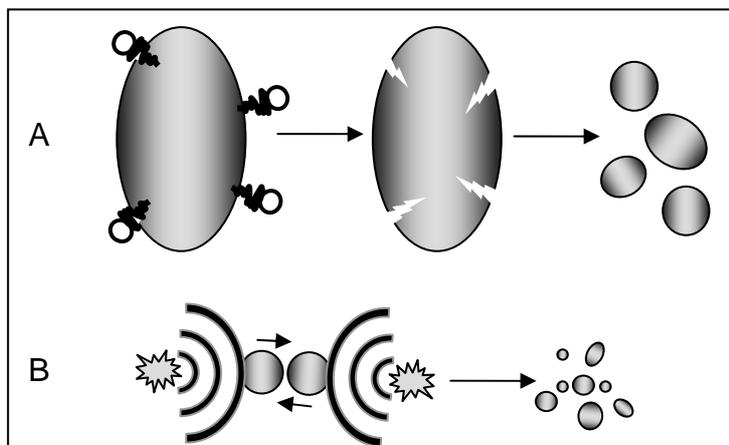


FIG. 1. SEM image of natural and sono-chemically modified chrysotile. Chrysotile fibres untreated (A), leached with oxalic acid for 21 hrs at 50 °C (B), and sonicated for 21 hrs in deionized water (C). Synergistic effect of oxalic acid and power ultrasound for 12 (D) and 21 hrs (E) give origin to micrometric debris. Some rod shaped nano-crystals, likely made of SiO_2 , are visible at higher magnification upon flat surfaces of chrysotile treated with oxalic acid and US for 21 h (F).

Accordingly with previous findings (Favero-Longo et al., 2005; Turci et al., 2007a), the long curled natural fibers of chrysotile (Fig. 1A) leached with oxalic acid for 21 hrs preserve their fibrous habit and are morphologically unaltered, though few small debris can be observed (Fig. 1B). Following

power sonication for 21 hrs in pure Milli-Q water (Fig. 1C), the long fibers of chrysotile are strongly reduced in length, but fibrous habit is largely preserved and fibers length falls within the micrometric range. Consistently with what reported in pioneering works (Spurny et al., 1979; Spurny et al., 1980), the effect of US on asbestos fibers in pure water is mechanical in nature. Ultrasound in heterogeneous systems may act on suspended particles, i.e. asbestos fibers, through two different cavitation mechanisms, which depend on US resonance frequency and particle size: microjet impact and shock-wave damage (Suslick, 1990). A sketch of this two mechanism is reported (Scheme 1). If particle size is several times larger than resonant bubble size, the asymmetry of the near-surface environment induce a deformation of the resonant cavity during its collapse. This self-reinforcing deformation generates a high speed stream of liquid that impact on the surface with a velocity greater than 100 m/s. The impingement of microjets on surfaces is the well-known cause of corrosion and erosion phenomena of metals and machinery and ultrasonic cleaning of surfaces (Preece and Hansson, *Adv. Mech. Phys. Surf.*, 1981). If particle size is small enough not to disturb the liquid, the behaviour of the system will be closer to what observed for homogeneous systems, and shock waves will be produced following acoustic cavitation. Such waves can create high-speed velocity interparticle collisions which may induce melting of metals particle or further fragmentation of ceramic particles (Doktycz and Suslick, 1990). When oxalic acid 0.5 M is added to the chrysotile suspension the fibrous habit of asbestos is rapidly disrupted during treatment of 12 and 21 hrs (Fig. 1D and 1E, respectively) and large aggregates occurs. This finding is consistent with data previously obtained during similar treatment on chrysotile sample form Balangero. The loss of fibrous habit on Val Malenco chrysotile after 12 hrs of US-Ox treatment is even more pronounced than what reported from Balangero one, the former being possibly more rapidly disrupted by US-Ox treatment. One representative high-magnification image of some particles undergone US-Ox treatment for 21 hrs is reported in Fig. 1F. Some apparently flat surfaces are covered with highly packed rod-shaped nanometric particles. Such particles were

already observed following identical treatment upon the chrysotile from Balangero and were hypothesised to be nano-fibers of antigorite (a serpentine polymorph) (Turci et al., 2008).



SCHEME 1. Sketch of the two possible mechanisms of acoustic cavitation in liquid-solid systems.

A) If particle size is several times larger than resonant bubble size, the particle creates an asymmetry of the environment near the surface which induces, during bubble collapse, a fast-moving stream of liquid through the cavity at the surface with velocity greater than 100 m/s. Such impacts are often used to clean surfaces and are responsible of the characteristic pitting in the surface. B) If particles are smaller than ca. 200 μm (with US operating at ca. 20 kHz), the shock waves created by homogeneous cavitation can accelerate particles and induce high-energy interparticle collisions.

Crystallographic modification induced by sono-chemical treatment

The X-ray diffraction (XRD) patterns of untreated Val Malenco chrysotile fibres is reported (Fig. 2, pattern *a*) and compared with fibres treated with US only for 21 hrs (*b*), US-Ox for 12 and 21 hrs (*c* and *d*, respectively). Untreated fibres gave the well-characterized X-ray diffractogram of chrysotile that was recognised by comparison with standards from the Joint Committee of Powder Diffraction (JCPD) archive. Chrysotile main reflections (002 and 004, marked with § on the figure) were unaffected by sonication in pure water, which only induces a reduction in size. When oxalic acid is added and the suspension sonicated for 12 and 21 hrs (patterns *c* and *d*, respectively), the

crystallographic features of chrysotile are suppressed and only reflexes due to magnesium oxalate are recorded (marked with *, in figure). Some oxalate reflexes around $2\theta = 44$ are superimposed with a doublet due to the occurrence of some steel debris from the ultrasonic reactor.

Diffractograms of chrysotile from Balangero untreated and US-Ox treated for 21 hrs are reported for comparison (*e* and *f*, respectively). As previously reported, the crystallographic features of serpentine (§) are still visible in the US-Ox treated sample, indicating the occurrence of some antigorite which was resistant to the sono-chemical treatment.

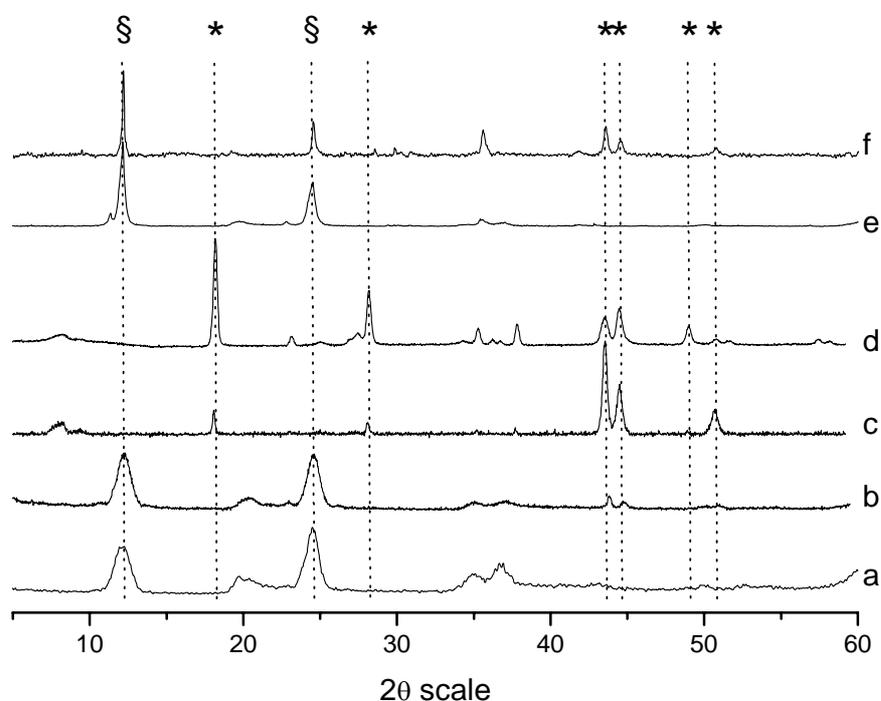


FIGURE 2. X-ray patterns of natural (a) and treated chrysotile fibers from Val Malenco. Asbestos was treated with US only for 21 h (b) or with the association of US and 0,5 M oxalic acid for 12 and 21 h (c, d). Recorded patterns were compare with natural fibers of chrysotile from Balangero (e) and treated with US + 0,5 M oxalic acid for 21 h (f). § indicates reflections due to chrysotile; * indicates reflections due to Mg oxalate.

Spectroscopic characterization of solid residues

Among spectroscopies, micro-Raman is by far the easiest technique able to characterize asbestos fibers and resolve serpentine polymorphs (Groppo et al., 2006). In Fig. 3 the representative spectra of chrysotile fibers untreated (a), sonicated for 21 hrs (b), leached for 21 hrs with oxalic acid (c) and US-Ox treated for 21 hrs (d) are reported. Spectrum *a* shows the two most intense bands for SiO_4 bending at 390 cm^{-1} (antisymmetric $\nu_5(\text{e})$) and 693 cm^{-1} (symmetric $\nu_2(\text{a}_1)$). As expected, both spectra *b* and *c*, recorded with fibers treated with US or Ox respectively, reports the chrysotile-related SiO_4 vibrational features. Following US-Ox combined treatment, spectrum *d*, the chrysotile peaks are not recorded and only magnesium oxalate is detected (Frost, 2004).

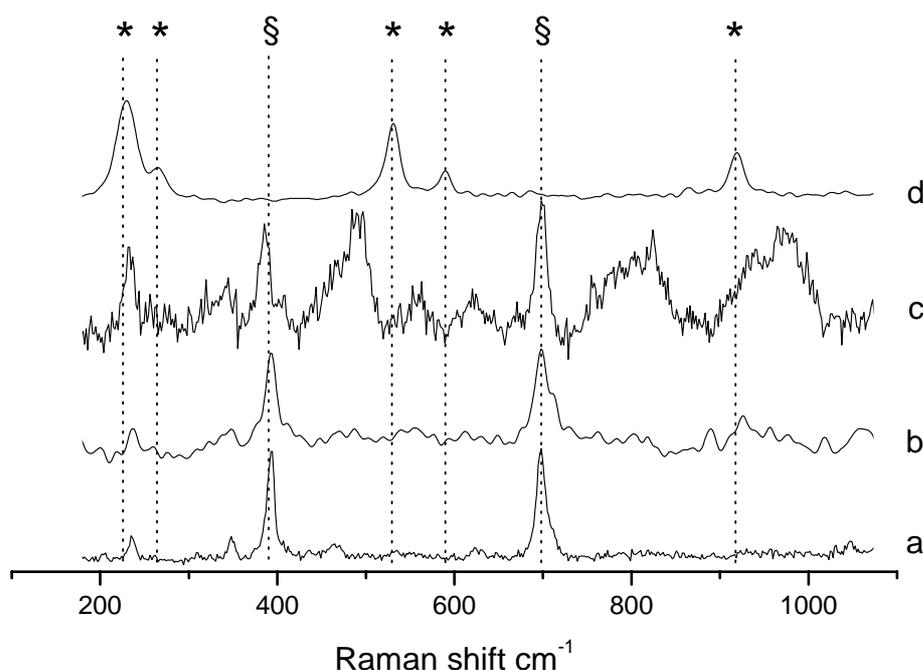


FIGURE 3. Raman spectra of untreated chrysotile fibers (a), chrysotile sonicated for 21 h (b), chrysotile leached with Ox for 21 hrs (c), and chrysotile treated with US-Ox for 21 hrs (d). A polarised solid state Nd 80 mW laser operating at 532.11 nm was used as the excitation source. Representative spectra from at least 30 independent measurements are reported. Light microphotographs of sampled area are also reported.

It is note worthy that in this sample, opposite to what reported for chrysotile from Balangero, antigorite is not detected, while in both samples SEM imaging showed the occurrence of some rod-shaped nano-structure. These nano-fibers were previously thought to be made of antigorite, but their occurrence also in the antigorite-free chrysotile indicates that they are likely made of amorphous silica, providing further assurance about the safety of treated product.

Elemental analysis

The relative amount of Mg with respect to Si is a well established approach to describe depletion of brucitic layer in chrysotile and to investigate the congruence/incongruence of mineral dissolution (Favero-Longo et al., 2005; Turci et al., 2007a). X-ray florescence spectra of the two untreated chrysotile and some treated samples were collected and intensity of integrated Mg peak were normalized by Si peak. Both Val Malenco and Balangero fibers are slightly affected by sonication in pure water (light gray bars) with respect to untreated mineral (empty bars). The combination of oxalic acid and US induced an almost complete removal of Mg from the Val Malenco sample after a 12 hrs treatment (dark gray bar), supporting the hypothesis that the rod-shaped nanometric particles are made of amorphous silica. A similar result was obtained by leaching chrysotile under extreme conditions, i.e. HCl solution 3M at 373 K (Wang et al., 2006). The Mg/Si ratio measured on the sample from Balangero after the same US-Ox treatment is much higher than what reported for Val Malenco, likely due to the occurrence of antigorite which is not completely disrupted during the US-Ox treatment.

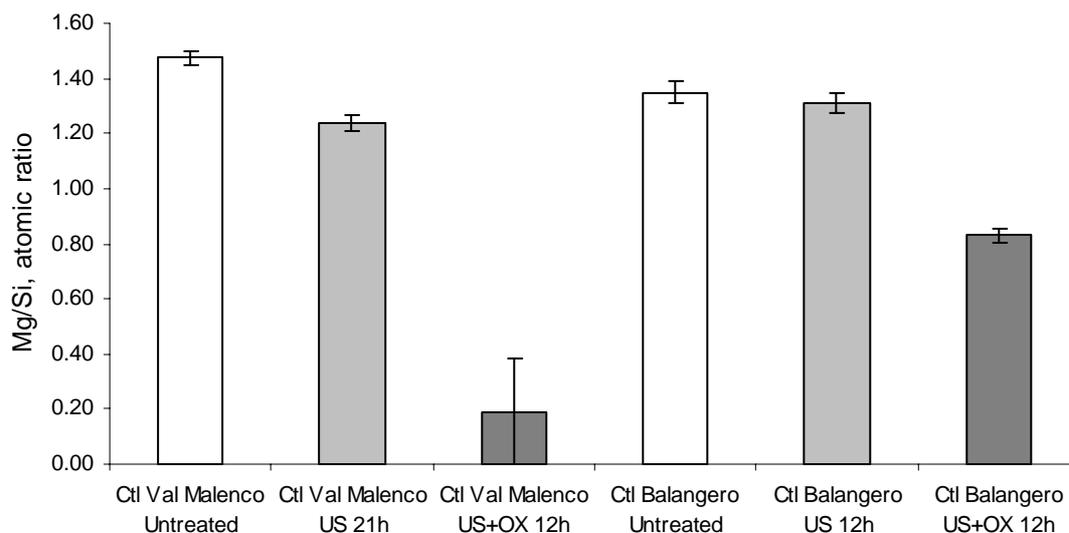


FIGURE 4. Atomic ratio (Mg/Si) calculated from XRF peaks on natural and treated chrysotile (Ctl) from Val Malenco and Balangero.

Conclusions

Despite toxicity of airborne asbestos fibres is well established, the hazard for humans of waterborne fibres is still object of debate. Nevertheless, a precautionary principle should invite us to abate or at least control waterborne asbestos fibres concentration whenever possible. A method for removing serpentine asbestos (i.e. chrysotile) in water suspension had been reported and has been here further evaluated. Elemental analysis (XRF) and crystallographic pattern (XRD) indicate the complete disruption of chrysotile fibres from Val Malenco (Italian Central Alps) after 12 hrs of treatment. The parallel loss of fibrous habit, evidenced by SEM imaging, further accounts for the loss of potential hazard of post-treatment mineral debris. Current findings support what previously reported for the chrysotile specimen from Balangero (Italian Western Alps) and clarify the amorphous nature of some rod-shaped nanoparticles found on both sample upon US-Ox treatment. The effectiveness of the proposed method for treating waterborne chrysotile fibres is confirmed.

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