# Microwave thermal inertisation of asbestos containing waste and its recycling in traditional ceramics

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## Abstract

Asbestos was widely used as a building material prior to the 1970's. It is well known that asbestos is a health hazard and its progressive elimination is a priority for pollution prevention. Asbestos can be transformed to non-hazardous silicate phases by microwave thermal treatment. The aim of this investigation is to describe the microwave inertization process of asbestos containing waste (ACW) and its recycling in porcelain stoneware tiles, porous single-fired wall tiles and ceramic bricks following industrial manufacture procedure. Inertised asbestos powder was added in the percentages of 1, 3, and 5 wt.% to commercially available compositions and then fired following industrial thermal cycles. Water absorption and linear shrinkage of the obtained industrial products do not present significant variations with additions up to 5 wt.% of microwave inertised ACW.
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*Keywords:* Microwave; Asbestos; Inertisation; Recycling; Traditional ceramics

## 1. Introduction

### 1.1. Asbestos generalities, identification and properties

The word "asbestos" refers to several types of fibrous minerals. There are many different types of asbestos fibre, divided into two main categories: amphibole and serpentine asbestos, which contain different percentages of the six minerals reported in Table 1. Amphiboles are double-chain silicates, of general formula $A_{0-1}B_2C_5T_8O_{22}(OH, F, Cl, O)_2$, where A = Na, K; B = Na, Li, Ca, Mn $Fe^{2+}$, Mg; C = Mg, $Fe^{2+}$, Mn, Al, $Fe^{3+}$ Ti; T = Si, Al (if it is present it is just in trace); which may assume a fibrous habit being structurally elongated in one preferred crystal direction. Serpentines are sheet silicates (with a tetrahedral and an octahedral sheet) which may roll to counterbalance the misfit between the tetrahedral and octahedral layer assuming a characteristic fibrous habit with formula $Mg_3(OH)_4Si_2O_5$ (essentially chrysotile). An asbestos fibre is defined by the World Health Organization (WHO) as a particle that has a length >5 μm, with a diameter <3 μm and an aspect ratio greater than 3:1 [1].

Due to asbestos favourable properties (incombustibility, low thermal conductivity and high electrical resistance, resistance to alkali and acid attacks and to micro-organism) it has been utilised in the past for a large number of applications. Moreover, asbestos exhibits a remarkable mechanical resistance and sound absorption capabilities, properties that made asbestos an useful building material. Its low cost and low/medium density suggested its use as an insulator for the transportation industry. Up until the 1970's, some 3000 products were made with asbestos fibres of all types [2]. Today, some 60 countries still use asbestos, but only the chrysotile variety and primarily in cement building materials such as roofing materials, cladding and pipe (99% of the world's current asbestos production is chrysotile) [2]. Italy was among the first countries to appreciate the many potential uses of asbestos. The main quarries in Italy are found in "Val Malenco" (Lombardia), "Balangero in Val di Lanzo", "Val di Lana" and "Val di Susa" (Piemonte) and "Val D'Aosta".

Strong concerns about the health hazards associated with asbestos had been described many times over the years [3–7]. The fine asbestos fibres are easily inhaled, and can cause a number of respiratory complaints, including a potentially serious lung fibrosis called asbestosis. Exposure to asbestos has also been determined to cause a form of cancer, mesothelioma, that occurs in the chest and abdominal cavities. Recent investiga-

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Table 1
Chemical and physical properties of common asbestos minerals

| Characteristic, chemical analysis (avg) | Serpentine, chrysotile $Mg_3Si_2O_5(OH)_4$ | Amphibole | | | | |
|---|---|---|---|---|---|---|
| | | Crocidolite $Na_2Fe^{2+}3Fe^{3+}2(Si_8O_{22})(OH)_2$ | Amosite $(MgFe^{2+})7Si_8O_{22}(OH)_2$ | Anthophyllite $Mg_7(Si_8O_{22})(OH)_2$ | Tremolite $Ca_2Mg_5Si_8O_{22}(OH)_2$ | Actinolite $Ca_2(MgFe^{2+})5Si_8O_{22}(OH)_2$ |
| $SiO_2$ | 38–42 | 49–56 | 49–52 | 53–60 | 55–60 | 51–56 |
| $Al_2O_3$ | 0–2 | 0–1 | 0–1 | 0–3 | 0–3 | 0–3 |
| $Fe_2O_3$ | 0–5 | 13–18 | 0–5 | 0–5 | 0–5 | 0–5 |
| $FeO$ | 0–3 | 3–21 | 35–40 | 3–20 | 0–5 | 5–15 |
| $MgO$ | 38–42 | 0–13 | 5–7 | 17–31 | 20–25 | 12–20 |
| $CaO$ | 0–2 | 0–2 | 0–2 | 0–3 | 10–15 | 10–13 |
| $Na_2O$ | 0–1 | 4–8 | 0–1 | 0–1 | 0–2 | 0–2 |
| Others | 11.5–13 | 1.7–2.8 | 1.8–2.4 | 1.5–3 | 1.5–2.5 | 1.8–2.3 |
| Decomposition temperature (°C) | 450–700 | 400–600 | 600–800 | 600–850 | 950–1040 | 620–960 |
| Fusion temperature of residual material (°C) | 1500 | 1200 | 1400 | 1450 | 1315 | 1400 |

Table 2
Common industrial, scientific and medical (ISM) bands for industrial and commercial applications

| Frequency (MHz) | Admitted tolerance (+/−) | Area |
|---|---|---|
| 433.92 | 0.2% | Austria, Holland, Portugal, Switzerland, Germany |
| 896 | 10 MHz | Great Britain |
| 915 | 13 MHz | North and South America |
| 2375 | 50 MHz | Albany, Bulgaria, Czech Republic, Hungary, Rumania, |
| 2450 | 50 MHz | Worldwide level except where it is used 2375 MHz |
| 3390 | 0.6% | Holland |
| 5800 | 75 MHz | Worldwide |
| 6780 | 0.6% | Holland |
| 24150 | 125 MHz | Worldwide |
| 40680 | | Great Britain |

tions focus into the mechanisms involved in mineral particle-induced toxicity and asbestos detoxification and inertisation indicating the importance of their surfaces in the pathological consequences [8].

It is estimated that the amount of ACW in Italy can reach 30 million tons, i.e. 18 million $m^3$, of which ∼8 million $m^3$ are asbestos [9]. Moreover, asbestos contained in the products is not degradable: it can contaminate superficial and deep waters and the surrounding atmosphere if dumps are damaged. An associated problem is the high cost of the disposal of ACW, which is of the order of 1.50 euro/kg [10]. The situation is similar in other European countries. It is thus evident that the safe disposal of this kind of waste represents a difficult issue, both from environmental and economic points of view.

Therefore, it is necessary to avoid, or minimize to the extent possible, the amount of waste created by adopting the most efficient production techniques, including recycling. When asbestos is fired at temperatures >1000 °C, either serpentine or amphiboles species, which are hydrate silicate, transform into Mg–Fe silicates. In this study, thermal inertisation by microwave irradiation is presented as an innovative and cost-effective way to treat ACW. It is also investigated the potential recycling of the inert magnesium silicates obtained as secondary raw materials for the fabrication of traditional ceramic materials.

### 1.2. Microwave and materials interaction

Microwaves are electromagnetic waves with a wavelength longer than infrared light, but shorter than radio waves located in the high frequency electromagnetic field between 0.3 and 300 GHz. The Industrial, Scientific and Medical (ISM) microwave band were originally reserved internationally for the unlicensed use of high frequency electromagnetic fields for industrial, scientific and medical purposes. The ISM frequency range can vary according to location (see Table 2), but most of the microwave heating applications use the 900 and 2.45 GHz. In Italy microwave ovens usually operate at 2.45 GHz.

Interactions between microwaves and materials can be represented by three processes: space charges due to electronic

conduction, ionic polarization associated with far-infrared vibrations, and polarization of electronic dipoles. The dielectric losses are due to ionic conduction, particularly at low frequencies, and rotation of permanent dipoles at higher frequencies. The ionic conduction losses are due to ohmic losses that occur when ions move through the material and collide with other species. Ionic conduction decreases with increasing frequency because the time allowed for transport in the direction of the field decreases with increasing frequency. Both processes contribute to the losses, and it is difficult to experimentally differentiate between the two loss mechanism. Therefore losses are typically reported as effective losses and expressed in terms of the imaginary part of the dielectric constant ($\varepsilon''_{eff}$), when the loss mechanism are unknown or cannot be clearly separated. Since dielectric properties are deeply related to microwave/material interaction, the microwave processibility of a wide range of non-magnetic materials is controlled by the quantitative relationships of absorbed power per unit volume ($P_A$) and depth of penetration ($D_P$) (defined as the distance in the direction of penetration at which the incident power is reduced to half its original value) [11]. Eqs. (1) and (2) give the simplified expression of power per unit volume ($P_A$) and depth of penetration ($D_P$) respectively:

$$P_A = 2\pi f \varepsilon_0 \varepsilon''_{eff} E_{rms}^2 \ (\text{w/m}^3) \tag{1}$$

$$D_P = \lambda_0 \sqrt{\varepsilon'/2\pi\varepsilon''} \ (\text{m}) \tag{2}$$

where $f$, frequency of the field; $\varepsilon_0$, permittivity of free space; $\lambda_0$, free space wavelength of the microwave radiation and $E_{rms}$, root mean square internal electric field per unit volume, and $\varepsilon'$ and $\varepsilon''$ are the relative values of the dielectric constant and loss factor, respectively.

In general materials fall in three categories with respect to their interaction with microwaves: (a) microwave reflectors, typified by bulk metals and alloys, such as brass, which are therefore employed in making microwave guides; (b) microwave transmitters which are transparent to microwaves, typified by fused quartz, several glasses, and ceramics (not containing any transition element), PTFE etc.; they are used for making cookware and containers for carrying out chemical reactions in microwaves and (c) microwave absorbers which take up the energy from microwave field and get heated up very rapidly.

The possibility of microwave processing ceramics was already known in the late 1950's, and was investigated on a limited basis in the 1960's by Tinga and Voss [12]. However, it has been also object of many research studies in recent years [13]. Microwave irradiation offers a number of unique benefits when it is used as thermal treatment in ceramic materials. In contrast with other heat sources, such as conventional ovens where heat is applied externally to the surface of the material, microwave irradiation penetrates and simultaneously heats the bulk of the material because material itself converts energy to heat. In addition, heating is instantaneous (on/off) with power.

Even if some ceramics materials are transparent to microwaves at room temperature, however, the vast majority of ceramic materials absorb microwaves from a lesser to a greater degree. In a number of materials, such as alumina ceramics, during microwave heating the increase of temperature is found to be gentle at first but very rapid later when a threshold is reached. This phenomenon, characterized by a sudden rise of temperature, is referred in literature as thermal runway and it has been focus of numerous studies [14–18]. The process of thermal runway is caused by resonance within the irradiated medium due to the temperature dependence of $\varepsilon''$ [18]. A potential consequence of the rapid increase in $\varepsilon''$ with temperature is that, unless the sample is heated uniformly, localized hot spots can develop. On the other hand this phenomenon permits the microwave processing and rapid heating of many materials that are poor absorbers at ambient temperatures [19].

### 1.3. Microwave inertisation of ACW

Various publications from 1960 up to now have reported industrial applications on ceramic materials containing asbestos tailings, wastes or asbestos like primary materials, fired by conventional [20–22] or microwave thermal treatment [23,24]. On the other hand a great amount of papers reports thermal degradation of asbestos and other inertisation techniques [25–30]. By considering that legislation does not allow handling asbestos materials, the authors considered to use previously inertised by microwave ACW as a secondary raw material for the production of industrial ceramic products, as porcelain stoneware tiles or ceramic bricks.

Microwave processing of asbestos waste has been applied since 1997 in Italy and numerous studies have been carried out by joint research efforts of the University of Modena and Reggio Emilia (DIMA) and ENEA-UTS Tecnologie Fisiche Avanzate [31–33]. This inertisation process modifies the dangerous fibrous structure of asbestos and transforms it into inert magnesium oxide containing compounds in the form of solid blocks. The composition of these blocks is based on forsterite ($Mg_2SiO_4$) as major crystalline phase and no hazardous minerals are present after the thermal treatment [33].

Considering the actual cost of magnesium containing raw materials, it is interesting to consider an efficient use of inertised asbestos in the formulation of traditional ceramic materials, such tiles, bricks and refractories [34]. This is an attractive approach because inertised asbestos could be offered in the market at competitive price.

Therefore, the purpose of this research is to investigate the process of thermally induced transformation of asbestos containing materials into magnesium oxide containing compounds and to demonstrate the viability of recycling inertised asbestos in porous single-fired wall tiles, porcelain stoneware floor tiles and ceramic bricks. The changes in the microstructure and technological properties introduced by adding different percentages of inertised ACW in traditional ceramic materials are also investigated.

## 2. Experimental

### 2.1. ACW characterization

The investigated samples were almost pure serpentine asbestos, containing a small amount of an organic binder,

used for building insulation and provided by ENEA (Ente per le Nuove tecnologie, l'Energia e l'Ambiente). The thermal behaviour of the samples was determined by means of differential thermal analysis (DTA, Netzsch, STA 409) on as-received samples. X-ray diffraction (XRD) analyses were carried out on powdered samples (Cu Kα, Ni-filtered radiation, Model PW3710 Philips) to determine crystalline phase modification due to thermal treatments. The powders (<30 μm) were prepared by sampling different regions of each specimen following internal laboratory safety standard procedure according to Italian Decrees no. 626/94 and D.Lgs no. 25/02 which cover policies applicable to all employees and students engaged in the laboratory use of hazardous chemicals.

The microstructures were examined through scanning electron microscopy (SEM, Philips, PSEM 505). Dielectric properties of ACW were determined to asses the capability of this hazardous material to absorb microwaves. These properties were measured on representative samples by using a network analyzer (HP-85107B), and a dielectric probe (HP-85070B). Measurements were performed in the range 2–10 GHz, by slightly pressing the probe onto the sample surface, kept at room temperature.

### 2.2. Microwave heat-treatments at 2.45 GHz

The microwave treatment consisted of 13 min irradiation at 2.45 GHz at room temperature in a multimode cavity (VPMS, Radatherm). ACW samples were added with a very small amount of a microwave-sensitive activator and temperature detection was performed using a Pt–30% Rh/Pt–6% Rh type B thermocouple positioned in contact with the sample and monitored by means of a two-channel fibre optic system (ENEA, adapted) in the 300–1200 °C range.

Another thermal treatment was performed on the as-received material in order to achieve a vitrified structure in the ACW samples. The operating conditions were the same mentioned above but the sample exposure to microwave radiation was 25 min.

### 2.3. ACW recycling

Inertised asbestos powders were added in the percentages of 1, 3 and 5 wt.% to the ceramic bodies. In the case of stoneware and porous single firing bodies, the raw materials were wet-milled for 40 min in porcelain jars, disk-shaped specimens (diameter, 40 mm; thickness, 5 mm) were prepared by uniaxial pressing at 29 and 25 MPa, respectively without addition of any binder. The bricks samples were prepared directly in the plant following the industrial procedure. The porcelain stoneware and porous single-fired samples were fired in electric furnace (melt-two, Remet) following industrial thermal cycles. The maximum firing temperatures were 1220 and 1130 °C, respectively. Bricks were fired in the industrial kiln at 950 °C. In order to verify the sintering degree of the ceramic materials after addition, water absorption (WA%) and linear shrinkage (LS%) were measured on representative samples. WA% was measured following ISO 10545-3 in the case of porous single-fired wall and porcelain stoneware tiles and UNI 8942/3 for the bricks. The perma-

nent linear shrinkage was determined by measuring the relative diameter variation of the test discs before and after the thermal treatment using Vernier callipers.

## 3. Results and discussion

Fig. 1 shows the DTA curve of an as-received sample of ACW. The exothermic peaks at $T_1 = 288$ °C and $T_2 = 326$ °C are due to the decomposition of the organic reinforcement fibres inserted during the material manufacture. The endothermic peaks at $T_3 = 648$ °C and $T_4 = 680$ °C can be attributable to serpentine OH$^-$ release. Since the last exothermic peak at $T_5 = 803$ °C indicates the change from fibrous morphology to crystalline structure [31–33], the ACW samples must be heated at least at this temperature to attain thermal inertisation. Dielectric property measurements performed at room temperature of the as-received samples provided an average value of the complex permittivity of 2.068-j0.053. Since the imaginary part of the dielectric constant represents the effective losses, this value suggests to consider as-received ACW a material with satisfactory microwave energy absorbing properties. Higher microwave frequency (6–15 GHz) would lead to better absorption, but at the cost of penetration depth.

The XRD patterns of untreated and 13 min microwave thermally treated ACW samples, collected in the same $2\theta$ (°) range at room temperature, are shown in Fig. 2. It can be seen that serpentine is the main crystalline phase in the as-received ACW, as expected. On the contrary, the XRD pattern of microwave thermally treated ACW sample evidentiates the formation of a new crystalline phase forsterite ($Mg_2SiO_4$). The microstructure of forsterite is characterized by platelets of an average dimension of 10 μm as can be observed in the SEM micrograph of Fig. 3b referred to a microwave thermal treatment of 13 min. This fact confirms an effective sample inertisation, since forsterite is considered a totally harmless magnesium silicate.

SEM observations evidenced the complete loss of the fibrous structure in inertised ACW samples, as can be seen in Fig. 4, where a comparison with the as-received sample is presented.

When microwave processing time is increased to 25 min, temperature rise well above 1200 °C and vitrification of the samples is achieved. During the treatment the samples loose their original
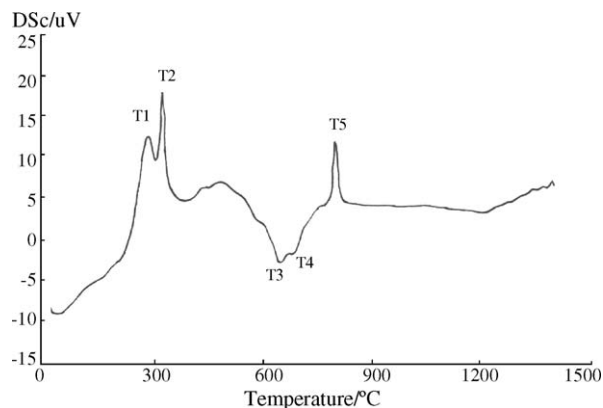
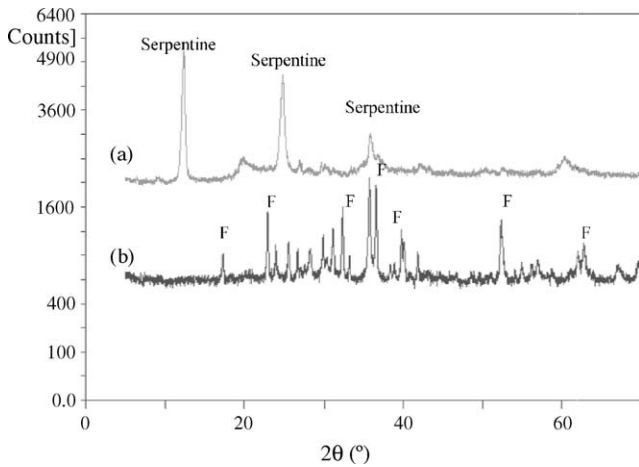

Fig. 1. DTA curve of an as-received sample of ACW.

Fig. 2. XRD patterns of (a) as-received ACW and (b) microwave thermally treated ACW samples, collected in the same $2\theta$ (°) range at room temperature. F, Forsterite.

shape becoming a viscous and black in colour glassy material, as seen in Fig. 4a. Many millimetre-sized open pores are present on the surface, probably caused by gaseous emissions ($H_2O$ from the serpentine structure). The vitrification treatment performed at this temperature is more drastic to eliminate the dangerous fibrous structure of ACW, as can be seen in Fig. 4b, where a SEM micrograph shows a polygonal shaped crystal of pure forsterite embedded in a vitreous matrix of silicate composition.
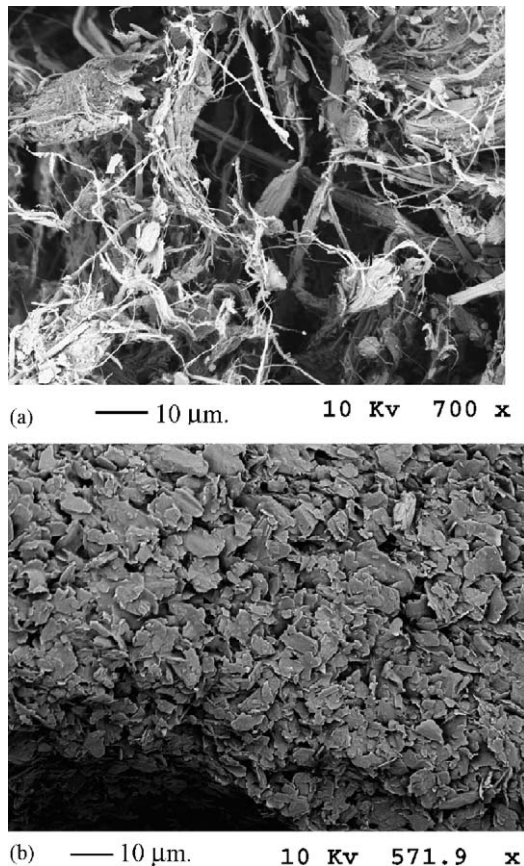


Fig. 3. SEM micrographs of the ACW sample (a) before and (b) after the microwave inertisation treatment.
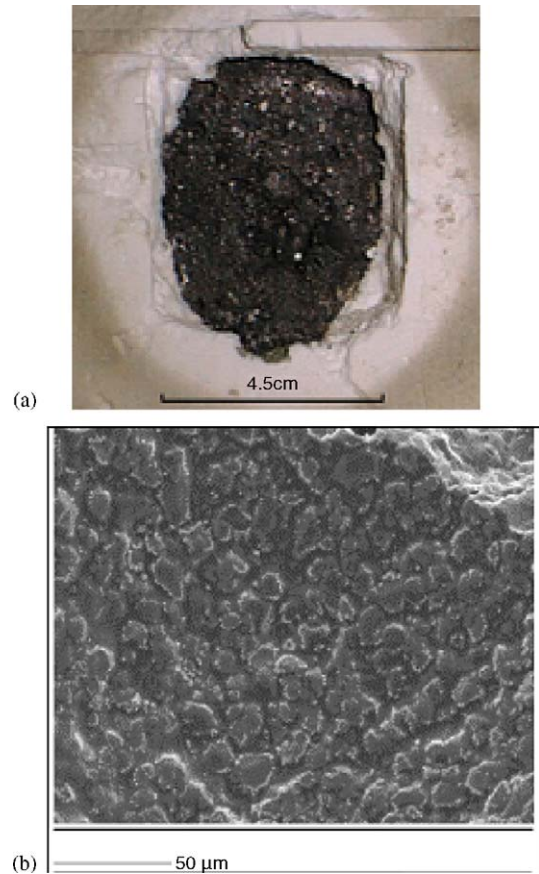


Fig. 4. ACW sample after microwave vitrification treatment; (a) Picture evidentiating the resulting amorphous structure; (b) SEM micrograph showing a microstructure where polygonal shaped crystals of pure forsterite are embedded in a vitreous matrix of silicate composition.

Either in the inertised form, or in the vitrified one, the resulting material can be used for introducing silicates and magnesium oxides in ceramic bodies compositions. Thus, different percentages (1, 3 and 5 wt.%) of inertised or vitrified ACW were introduced in bricks, porous single-fired and porcelain stoneware tiles, following the typical industrial cycles. Inert or reactive behaviour of microwave thermally inertised ACW can be expected as function of the firing temperatures. In the case of reactive behaviour, low-eutectics could form.

The external aspect of the ceramic samples without inertised ACW and with the addition 5 wt.% of inertised ACW does not show significant variations. The water absorption and linear shrinkage curves of the ceramic compositions under study are shown in Fig. 5a and b as a function of inertised asbestos amount introduced. Water absorption (WA%) is one of the most important parameter to determine the technical properties of a ceramic product. It is the quantity of water which a material can absorb under particular experimental conditions. Since this absorption takes place through the pores of the material, which are in communication with the external surface, water absorption is a measure of the open porosity and provides an immediate indication of the sintering degree of the material. Examples of some characteristics which depend upon the porosity are: mechanical resistance, abrasion resistance, frost-resistance, and chemical-
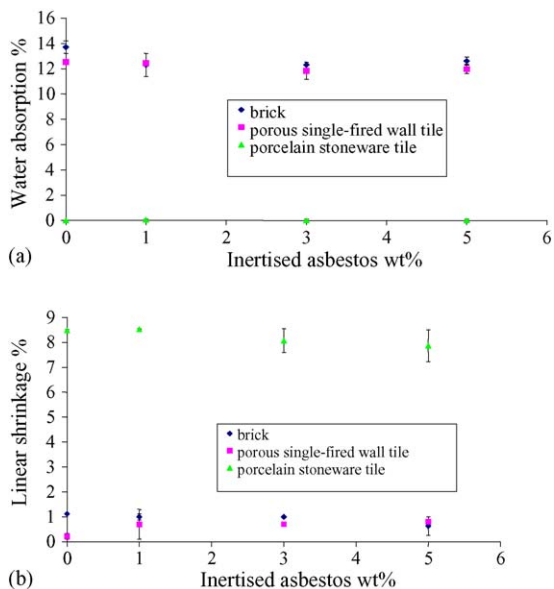
Fig. 5. (a) Water absorption and (b) linear shrinkage of the ceramic compositions under study as a function of inertised ACW amount.

resistance. From the results obtained it is possible to note that ACW addition does not modify to a great extent the WA% of the materials considered. The values of single porous tiles were similar to the standard (12 < WA% < 13). A slightly decrease of the WA values referred to the standard was observed for the bricks probably due to the higher amount of fluid phase formed in the structure due to the presence of vitrified asbestos. In the case of porcelain stoneware tiles the data reported are in agreement with ISO 13006 that asses the standard in WA% ≤ 0.05.

Linear shrinkage (LS%) is related to the compactness resulting from the degree of sintering and development of glassy phase during firing. A consequence of a high temperature firing process is that material undergoes considerable shrinkage. The addition of ACW does not introduce significant variation for the considered materials and the LS% values obtained are within the industrial tolerance.

Taking into account that the annual production in Italy of porcelain stoneware tiles reaches 360 million m² [35] and considering a weight of 18 kg/m² [36], the annual production can be estimated in approximately 6.5 million tons. In the case of ceramic bricks the annual production is about 4 million tons [37]. Hence the Italian amount of ACW, estimated in 30 million tons [9], could be eliminated in 57 years considering an addition of 5 wt.% to the products.

The microwave inertisation process costs will depend on the type of microwave system used and on the electric power cost. The microwave-material interaction determines the formation of reflected waves due to the different impedances of air and material and this affects negatively the applicator efficiency.

Considering the multi-mode applicator (1.2 kW) employed in this investigation, the results can be resumed as follows:

- Multimode applicator 25–30% reflected power: average specific energy consumption = 1.2 kWh/kg.

On an Italian basis, considering the price at 6 October 2005 (depending on the time of the day: minimum = 35 €/MWh; maximum = 100 €/MWh), the costs can be estimated about 0.04–0.12 €/kg. Bearing in mind that in Europe the asbestos wastes are recovered with 0.05–0.2 €/kg the cost of inertisation could be considered a satisfactory result because in this way a new raw materials, which can be exploited in an other productive cycle, is produced. The calculation is referred only to running and not to initial installation costs, for which an average of 1000–1500 € per microwave kW installed must be considered.

## 4. Conclusion

The possibility to inertize or to vitrify ACW samples by means of a microwave irradiation was demonstrated in this investigation. Inertised asbestos no longer contains the serpentine phase and its peculiar acicular microstructure and it was transformed into forsterite, a harmless magnesium silicate. The use of microwave energy has been proved to be competitive in comparison with the conventional treatment in terms of time reduction and energy saving. Moreover, forsterite derived from asbestos inertisation proved to be a suitable secondary raw material for the ceramic tiles and brick industries. In this work it has been successfully recycled asbestos containing materials as a component of the top product in the market of traditional ceramics: porcelain stoneware tile. The addition up to 5 wt.% of ACW does not show significant variation to the standard parameters of water absorption and linear shrinkage. This process is promising because an estimate of the costs for the recycle is 10 times lower with respect to the costs for disposal as toxic waste. Furthermore another advantage is gained: no hazardous refuses are produced with no risk of environmental pollution.

## Acknowledgement

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