Decomposition and utilization of asbestos-cement waste

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Abstract
Asbestos-cement is an important material, both on landfills of hazardous waste and in the form of various products that are part of the currently used constructions. Since asbestos has been classified as a hazardous chemical, it is desirable to propose a practical and safe procedure for the disposal of asbestos-cement products, or even better to re-use them. In this work, the temperature conditions of degradation of samples of various types of asbestos and products containing asbestos were examined. Samples were burnt at different temperatures and monitored by X-ray diffraction, DTA, light and electron microscopy. By evaluation of changes at different burning temperatures, the previous findings on the decomposition of asbestos minerals have been confirmed. Chemical analysis revealed that asbestos-cement products have a favorable composition that corresponds to low-saturated Portland cement clinker. It has been found that the burning of asbestos cement materials alone or with the addition of limestone at the temperature above 1,100 °C leads to the decomposition of hazardous asbestos and, after the addition, with or without the addition of a solidification regulator, quality hydraulic binder is formed with sufficient technological parameters that can be utilized in the building industry.

Keywords
asbestos, asbestos-cement, dangerous waste, construction material, decomposition, hydraulic binder

1. Introduction

The term asbestos involves fibre minerals from a group of silicates which occur in nature mainly in two mineral groups - serpentine and amphibole. In the past, asbestos was widely used for its very specific and excellent technological properties in various industries, massively also in construction industry. It can be said that in the Czech Republic asbestos can be found more or less in every building built before 1990. Its use was banned by the classification as dangerous chemicals (carcinogenic and mutagenic group). The liquidation process of asbestos still means the sending to landfills; in addition, these landfills are a potential danger for future generations. It seems to be beneficial to propose a practical and safe way of remediation of asbestos products, or even better a conversion of asbestos into a form usable for other purposes.

Although many articles have been written on this topic, decomposition and degradation of asbestos minerals and materials containing asbestos is still very actual.

One of the chapters in the book "Handbook of recycled concrete and demolition waste" represents a review on recycling of asbestos-contaminated materials. The main discussed question is whether these materials should be deposited in landfills or recycled. Recycling of thermally processed asbestos-cement materials for use in concrete and geopolymers production is depicted in details. It is stated that at temperatures 650 – 750 °C, chrysotile is dehydroxylated and forms forsterite followed by enstatite. Crocidolite decomposes to pyroxene, enstatite, hematite and cristobalite at 1,050 – 1,100 °C. Tremolite decomposes at 1,050 °C to form diopside, enstatite and cristobalite. Amosite is transformed to spinel, hematite, pyroxene and amorphous SiO_2 in the temperature range from 800 to 1,100 °C [1].

Gualtieri et al. depicts a complete reaction pathway of thermal processing of asbestos-cement coverings up to 1,200 °C. Light and electron microscopy and RTG diffraction were used to prove that recycling product formed by burning in a tunnel kiln is suitable for use in small amount as an additive into a stoneware paver blocks meal for burning [2].

Polish authors also tested thermal degradation of asbestos. They measured chrysotile, crocidolite, amosite and tremolite samples using DTA, RTG diffraction, FT-IR and SEM. They found that different temperatures are necessary to destruct different asbestos minerals - 700 – 800 °C for chrysotile and more than 900 °C for amphiboles. The resulting material has dehydroxylated anhydrous structure but fibrous morphology remains unchanged. Although the morphology is preserved, the analyses show that the typical asbestos structure is destroyed. The resulting material is easily ground to powder suitable to use in ceramic industry [3].

In Italy, Viani et al. examined thermal degradation of varied asbestos wastes. They chose temperature 1,200 °C and annealing time 15 and 30 minutes. To analyze resulting products, XRF, XRD, SEM with EDS, FT-IR and Raman spectroscopy were used. Their results can serve as a base for recycling of these materials and for inertization process optimization in industrial scale [4].

Many authors considered the temperature of chrysotile transformation to olivine (810 – 820 °C) to be the point when chrysotile changes to biologically inactive compound. However, it was found that also mechanical or thermal changes at temperatures even hundreds of degrees lower can be effective [5].

Yoshikawa et al. states that during heating the chrysotile ($Mg_6Si_4O_{10}(OH)_8$) and amosite ($Mg_7Si_8O_{22}(OH)_2$) fibres over 650 °C, dehydration and formation of $Mg_6Si_4O_{14}$ and $Mg_7Si_8O_{23}$ occurs. These products could be designated "metachrysotile" and "metaamosite". The authors examined the influence of microwave radiation on the detoxication mechanism of asbestos and also used CaO and quasi-asbestos phase $Mg_3Si_4O_{12}$ mixture to reveal the positive influence of CaO on asbestos decomposition [6].

Granat et al. used microwave radiation for asbestos-containing waste materials neutralization (asbestos-cement boards, roofing, textiles and gypsum-asbestos products) and found that the mechanism was economic and effective [7].

Other authors produced zeolite precursors from asbestos by leaching it in HCl or H_2SO_4 at varied temperatures up to 80 °C and varied reaction times. These zeolite asbestos derivatives exhibit excellent absorption capacity and absorption rate and can be used in agriculture or as carbohydrate synthesis catalyst [8].

In Germany, a pilot production experiment of asbestos-cement waste destruction was conducted in a rotary kiln for Portland clinker production. It is an effective and guaranteed way of degradation and the waste is completely processed to make a new product at the same time. The main issue is the hazardous waste transportation and its preparation – grinding to raw meal grain size and its deposition in the silo. Material was dosed in several wt. % of the raw meal directly into the burner of the rotary kiln, where the temperature is the highest [9].

In this paper, a way of utilization the asbestos-cement waste materials is depicted. By thermal processing, the asbestos is degraded and transformed into belitic hydraulic binder suitable for use in building industry.

2. Materials and methods

Following samples were subjected to experimental works:

- A1 – pure industrial fibrous asbestos
- A2 – natural asbestos from amphibole quarry
- E – asbestos cement roofing
- K – asbestos flue pipe

Pure samples (A1, A2) were subjected to qualitative RTG diffraction analysis on Bruker D8 Advance device with Cu anode ($\lambda_{K\alpha}$=1.54184 Å) and variable slits at Θ-Θ reflection Bragg-Brentano para focusing geometry. Products (E, K) were analysed both before and after dissolving in 35% HCl.

Thermal analysis was conducted on the Perseus STA 449 F3 (Netzsch) device in temperature range 30 – 1,100 °C and gradient 10 °C/min. Sample E was subjected to total chemical analysis.

Sample A1 was subjected to burnings in a muffle furnace at temperatures 500, 700, 900 and 1,100 °C with 30 min. burning time. Sample E was burned at 700, 900 and 1,100 °C and also decomposition in HCl after burning was carried out. Phase changes were monitored using X-ray diffraction analysis.

Experimental burnings of larger amounts of asbestos-cement materials were conducted in superkanthal furnace and the temperature program used was most similar to the one used in a shaft kiln for lime burning. The program involves slow temperature increase for 3 hours up to 1,200 °C, temperature hold for 8 hours and slow cooling in furnace down to 200 °C. Two types of waste were used – asbestos-cement cladding boards (W1) and unsorted asbestos cement roofing (W2). The resulting products' composition and structure were observed using X-ray diffraction, light and electron microscopy. From the burned products, epoxide polished sections were made for observation in reflected light on optical microscope Olympus LV100. For clinker minerals identification, the surface was etched with fumes of acetic acid [10]. From the original and burnt sample W2, samples coated with carbon were prepared for analysis by electron microscopy methods. Images of the surface were taken in secondary electrons on a Field-Emission Scanning Electron Microscope HITACHI SU 6600 with an EDS analyzer. The accelerating voltage used was 5 kV and 15 kV.

Also, hydraulic binders were prepared by burning the mixture of ground limestone and asbestos-cement waste. As the asbestos-cement products are mostly flat boards, they could negatively influence the flexural conditions during the burning in a furnace and thereby cause inhomogeneity in burning. Therefore, layering the bulk and flat materials, e.g. limestone and asbestos cement roofing, will be probably advantageous in practice. Moreover, hydraulic binders could be intentionally modified in free CaO content by dosing limestone. Two mixtures of unsorted asbestos cement roofing and ground high-calcium limestone (fraction 20 – 50 mm) were used for binder preparation in such amount that the resulting product contained 15 or 30 wt. % of free CaO.

Binders were prepared from burned asbestos-cement materials by milling in ball mill to specific surface ca. 450 m^2/kg, determined by Blaine method. Technological tests – the time of setting, soundness and strength after 7, 28 and 90 days of hydration – were conducted according to EN 196 standard. In some cases, setting controller (tartaric acid) was used in 0.1 – 0.4 wt. %.

3. Results and discussion
3.1 Preliminary analysis

Sample A1 - The sample contains amphibole asbestos, mainly riebeckite $(Na_2(Fe^{+2}_3 Fe^{+3}_2)(Si_8O_{22})(OH)_2)$ and in smaller amount cummingtonite $(Mg_7Si_8O_{22}(OH)_2)$. The sample is polluted by negligible content of quartz (SiO_2).

Sample A2 - The sample contains mainly amphibole asbestos minerals amosite $(Mg,Fe^{2+})_7Si_8O_{22}(OH)_2$, cummingtonite $(Mg_7Si_8O_{22}(OH)_2)$, riebeckite $(Na_2(Fe^{+2}_3 Fe^{+3}_2)(Si_8O_{22})(OH)_2)$ and tremolite $(Ca_2Mg_5(Si_8O_{22})(OH)_2)$. Chrysotile $(Mg_3Si_2O_5(OH)_4)$ from serpentine group and small amount of böhmite $AlO(OH)$ are presented as well.

Sample E - The sample contains mainly portlandite $(Ca(OH)_2)$, calcite $(CaCO_3)$ and relicts of non-hydrated clinker minerals (especially β-C_2S (Ca_2SiO_4) was identified). As an asbestos mineral, only chrysotile $(Mg_3Si_2O_5(OH)_4)$ is presented in significant amount. In small amount, quartz (SiO_2) and marcasite (FeS_2) were identified. After dissolution in HCl, sample contains only chrysotile and small amount of quartz. Thermic analysis showed an endotherm at 155 °C that is related to loss of moisture and dehydration of C-S-H gels. Another insignificant endotherm has the minimum at 400 °C. Endotherm at 485 °C is connected to decomposition of portlandite. Exotherm with peak at 520 °C is most probably a sign of marcasite oxidation. Dehydration of chrysotile is related to endotherm around 650 °C. Decarbonation of calcite has its peak at 810 °C. Total loss of ignition is 18.6 wt. %. Chemical composition of the sample is shown in Table 1.

Sample K - The main components of the sample are calcite $(CaCO_3)$ and portlandite $(Ca(OH)_2)$. In smaller amount, relicts of non-hydrated clinker minerals are presented, mainly β-C_2S (Ca_2SiO_4), and also quartz (SiO_2) in very small quantity. Significant amount of chrysotile $(Mg_3Si_2O_5(OH)_4)$ is contained and also amphibole asbestos minerals – mainly tremolite $(Ca_2Mg_5(Si_8O_{22})(OH)_2)$ and in smaller quantity also cummingtonite $(Mg_7Si_8O_{22}(OH)_2)$ are presented. After dissolution in HCl sample contains only chrysotile, tremolite, cummingtonite and small amount of quartz. Thermogram of the sample starts with endotherm with minimum at 150 °C that is connected to loss of moisture and dehydration of C-S-H. Decomposition of portlandite is represented by endotherm with minimum at 495 °C. Following endothermic effect around 650 °C is probably related to decomposition of chrysotile. A significant endotherm belongs to decarbonation of calcite with minimum at 850 °C. Total loss of ignition up to 1,100 °C is 27.9 wt. %.

Table 1. Chemical analysis of sample E in wt. %

Component	Content
LOI	18.05
SiO_2	19.72
SO_3	0.69
CaO	47.36
TiO_2	0.30
P_2O_5	0.16
Na_2O	0.16
K_2O	0.39
MgO	4.21
MnO	2.60
Fe_2O_3	2.49
Al_2O_3	3.72
Cl^-	0.029

3.2 Thermal degradation of asbestos

Sample A1 - Results of XRD analysis show that the sample remains almost the same after 500 °C. After 700 °C, the intensity of peaks of asbestos minerals are lower and new phase is formed – ferrogedrite $(Fe_5Al_2(Si_6Al_2)O_{22}(OH)_2)$. At 900 °C, another degradation of asbestos takes place and next to asbestos minerals, quartz, ferrogedrite and lower content of illite and also hematite (Fe_2O_3) is formed. At 1,100 °C, asbestos minerals are fully degraded and sample contains magnetite (Fe_3O_4), hematite (Fe_2O_3), pyroxene enstatite $Mg_2Si_2O_6$, maghemite (Fe_2O_3) and quartz. Heat changes the colour of material. Oxidation of iron causes colour change from grey to russet.

Sample E - Results of XRD analysis show that the sample does not contain diffraction lines of chrysotile already at 700 °C. After dissolution in HCl that leads to concentration of inert components such as chrysotile, this fact is confirmed. The sample heated up to 700 °C does not contain any portlandite unlike the unheated one. Moreover, C_4AF (brownmillerite) appears. With increasing temperature of heat, calcite content decreases. Besides amorphous phase, samples dissolved in HCl contain only quartz, pyrite and feldspar. After exposure to 1,100 °C, the sample is formed only by quartz and periclase (MgO).

3.3 Preparation and properties of hydraulic binders

According to results of analysis it can be said that chemical and phase composition of asbestos cement roofing is close to composition of hydraulic mortars. Basic chemical parameters were counted from the results of chemical analysis (see Table 2):

$$LSF = \frac{100 \times CaO}{2.8 \times SiO_2 + 1.18 \times Al_2O_3 + 0.65 \times Fe_2O_3} \tag{1}$$

$$SR = \frac{SiO_2}{Al_2O_3 + Fe_2O_3} \tag{2}$$

$$AR = \frac{Al_2O_3}{Fe_2O_3} \tag{3}$$

$$HM = \frac{CaO}{SiO_2 + Al_2O_3 + Fe_2O_3} \tag{4}$$

Where:
- LSF – Lime saturation factor,
- SR – Silica ratio,
- AR – Alumina ratio,
- HM – Hydraulic modulus.

Table 2. Basic chemical parameters counted from the results of chemical analysis of sample E

Parameter	LSF	SR	AR	HM
Value	77.4	3.18	1.49	1.83

There are three categories of hydraulic limes based on value HM – feebly hydraulic lime (HM = 6 – 9), moderately hydraulic lime (HM = 3 – 6) and eminently hydraulic lime (HM = 1.7 – 3). According to this, burning of dangerous asbestos should lead to formation of eminently hydraulic lime.

This material, as proved by Ambrosius et al. [9], should be added into raw meal for clinker preparation in amount ~ 10 %. The problem is with manipulation and grinding of this dangerous material. It can release harmful fibres into the air. Burning of large pieces of asbestos-cement material in save flammable covers in shaft kiln would eliminate this problem.

Experimental burning of the sample E that simulates burning in shaft kiln shows that sample is stable and without traces of disintegration after burning. It is stable even when it went through slow cooling around critical temperature around 500 °C, when the transformation of β-C_2S (Ca_2SiO_4) into γ modification takes place. γ-C_2S (Ca_2SiO_4) is responsible for disintegration of clinker resulting in hydraulically inactive material.

From the burned sample E, polished section was prepared for observing in reflected light under polarization microscope. After etching of polished section by fumes of acetic acid, it is obvious that the sample contains mainly clinker minerals from original non-hydrated cement – alite (C_3S, Ca_3SiO_5) and belite (C_2S, Ca_2SiO_4), newly formed belite and also minerals of clinker melt C_3A ($Ca_3Al_2O_6$) a C_4AF ($Ca_2(Al,Fe^{3+})_2O_5$). Structure of the burned asbestos cement roofing is shown in Fig. 1 and 2. Quantitative phase composition of the burned material was evaluated by X-ray diffraction analysis and it is given in Table 3.

Table 3. Phase composition of burned sample E determined by XRD analyses in wt. %

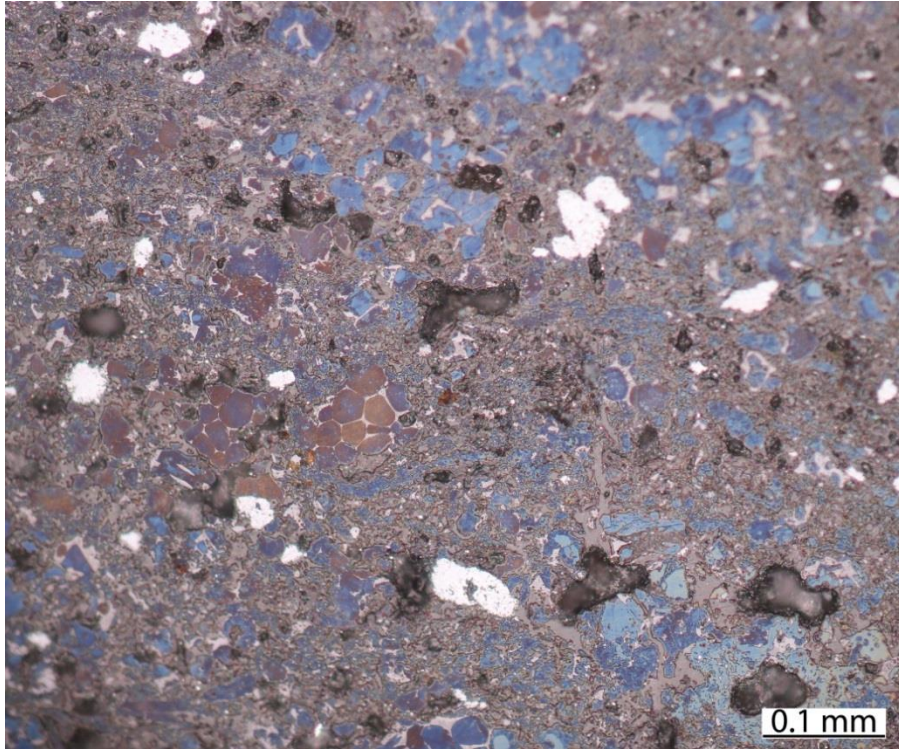| Phase	β-C_2S	γ-C_2S	C_3S	C_3A	C_4AF	CaO free	Periclase
Content	72.8	0.8	1.6	5.5	14.2	0.0	5.1

Fig. 1 Microstructure of burned asbestos cement roofing with obvious fractions of original non-hydrated cement in left and upper part. Blue crystals – alite, brown grains – belite, light areas – clinker melt; in the middle and bottom part – particles of secondary formed fine-grained belite (reflected light, polished section etched with acetic acid fumes)
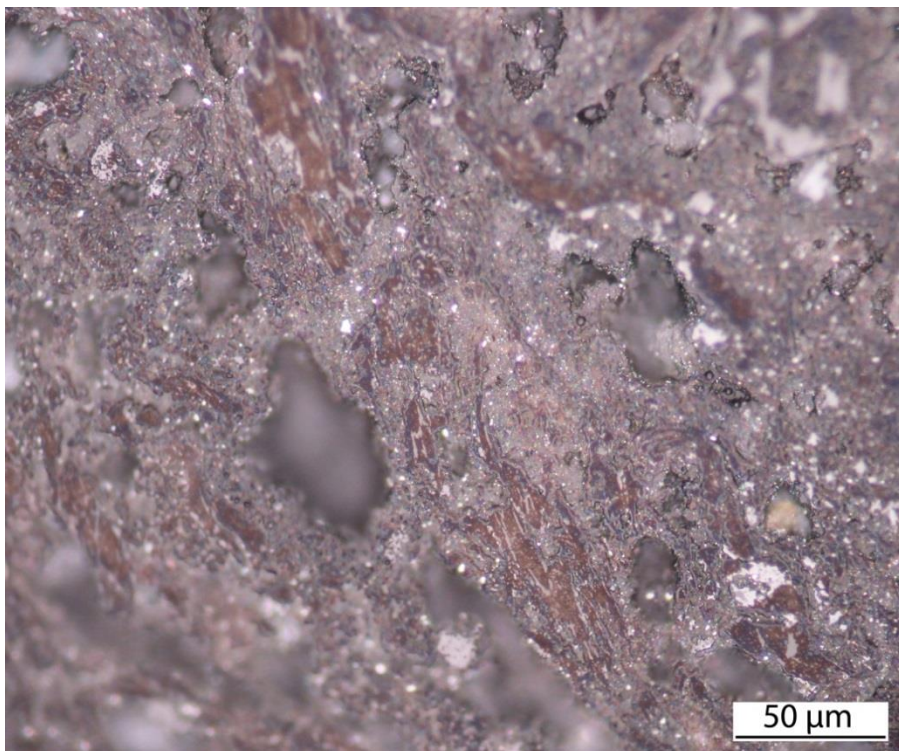

Fig. 2 Microstructure of burned asbestos cement roofing – detail with particles of secondary formed fine-grained belite (brown areas) (reflected light, polished section etched with acetic acid fumes)

3.3.1   Preparation of binders for technological tests

In the following process of solution, 2 samples of asbestos waste were provided in bigger amount – waste W1 – asbestos cement cladding boards and W2 – asbestos cement roofing.

Asbestos minerals chrysotile ($Mg_3(Si_2O_5)(OH)_4$), riebeckite ($Na_2Fe_5(Si_8O_{22})(OH,F)_2$) and amosite $Fe_7Si_8O_{22}(OH)_2$ were identified in the sample W1 by X-ray diffraction analysis. In the sample W2, only chrysotile was identified.

Both samples were burned by process described in chapter 2 that simulates burning of lime or hydraulic lime in shaft kiln. Also mixtures of sample W2 with pure crashed limestone (LL) in ratio 3 : 2 and 2 : 3 were burned by the same process.

Using electron microscopy by imaging in secondary electrons supported by EDAX analysis, complete conversion of hazardous asbestos fibers was demonstrated. These results are promising in the light of previous findings [11], where authors claim that the presence of $CaCO_3$ (the case of sample W2) prevents the asbestos to decompose according to the known decomposition reactions and leads to the formation of calcium-silicate compounds. An example of the structure prior and after burning the sample W2 is shown in Figures 3 to 8.
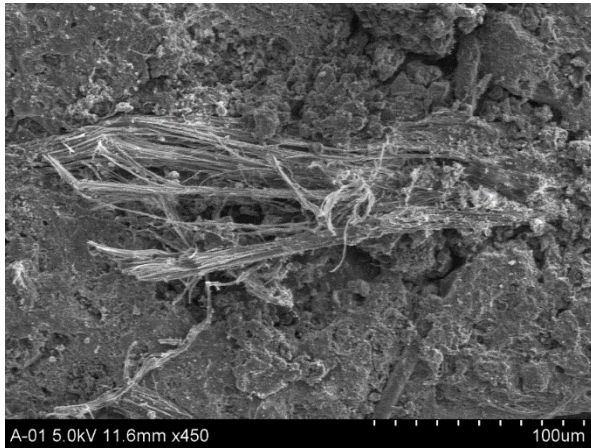

Fig. 3 A bundle of asbestos fibers (chrysotile) indicates in the original sample
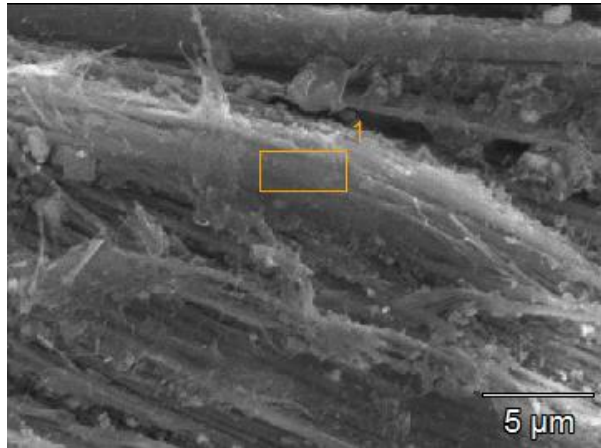

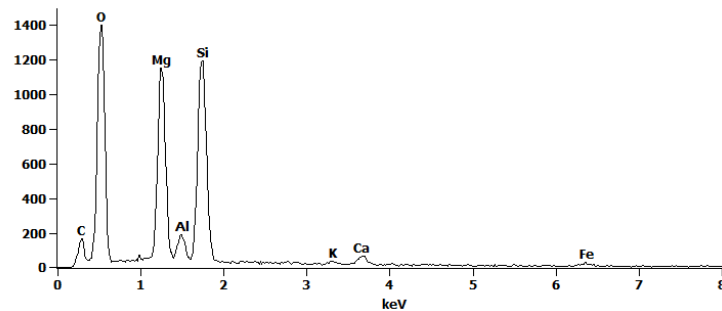**Fig. 4** Detail of chrysotile fibres, yellow border the place of the analysis

Fig. 5 The energy spectrum of the fiber areas indicated by the yellow border in Figure 4
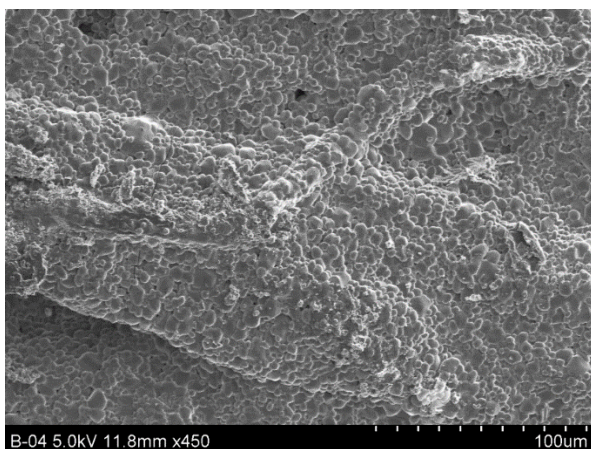

Fig. 6 The thermally converted bundle of asbestos (chrysotile) of W2 sample after firing. point.
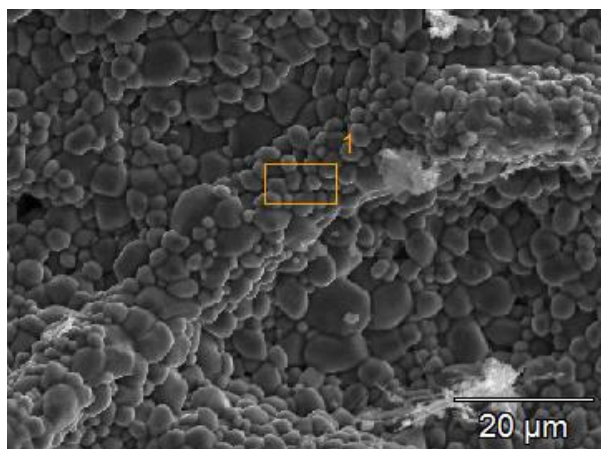

Fig. 7 Detail of decomposed chrysotile fibres (from Fig. 6), yellow frame indicates the place of the analysis
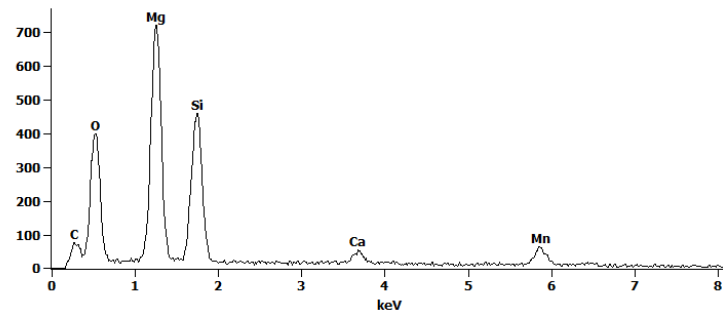
Fig. 8 The energy spectrum of the transformed fibers marked with the yellow border in Fig. 7

Burnt samples were ground in laboratory ball mill until specific area of approx. 450 m^2/kg was achieved and their phase composition was evaluated by XRD Rietveld refinement without internal standard (Table 4). Italian authors [4] previously prepared similar larnite/rich material as the product of high temperature transformation of cement-asbestos proposing it as the possible candidate for substituting a fraction of cement in many building materials and innovative green cement productions. However, this high temperature treated material contains MgO (periclase), an undesirable phase in PC because of the expanding properties of its hydration product $Mg(OH)_2$ (brucite) [1].

Table 4. Phase composition of burned sample W1 and W2 determined by XRD analyses in wt. %

Phase	W1	W2	W2 : LL = 3 : 2	W2 : LL = 2 : 3
β-C_2S	69.0	57.2	48.7	40.1
γ-C_2S	0.6	1.4	1.2	1.0
C_3S	3.6	4.1	3.5	2.9
C_3A	1.8	4.3	3.6	3.0
C_4AF	15.3	15.5	13.2	10.9
CaO free	0.1	0.0	15.1	29.9
Periclase	6.7	4.6	3.9	3.2
Quartz	3.0	0.7	0.5	0.5
Bredigite α'-C_2S	-	3.9	3.3	2.7
Akermanite $Ca_2MgSi_2O_7$	-	6.2	5.2	4.3
Maghemite Fe_2O_3	-	1.1	0.9	0.8
Pyrite FeS_2	-	1.0	0.9	0.7

In the first phase, evaluation of setting time was done, according to EN 196-3, on pastes with normal consistency and without addition of setting regulator. Setting was very fast and material has bad workability. Addition of 2, 4 and 6 wt. % of gypsum did not lead to expected results. Because of that, it was decided to use different type of setting regulator. Tartaric acid (t.a.) can be used for hydraulic lime. The dose was stated 0.1, 0.2 and 0.4 wt. %. Waste burned together with limestones has quite a high amount of free lime and they had to be slaked before evaluation of setting time. After that, their setting time is long and no regulator needs to be added. Results of setting and hardening time are stated in Table 5.

Table 5. Setting times of prepared hydraulic mortars according to EN 196-3

Samples	Start of setting [hour:min]	End of setting [hour:min]
W1	0:10	0:20
W2	0:10	0:20
W2 + 6 wt. % gypsum	0:10	0:20
W2 + 0.1 wt. % tartaric acid	1:40	2:30
W2 + 0.2 wt. % tartaric acid	4:20	6:00

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W2 + 0.4 wt. % tartaric acid	2:00	2:50
W1 + 0.1 wt. % tartaric acid	0:20	0:30
W2 : LL 3 : 2	3:50	26:00
W2 : LL 2 : 3	4:20	38:00

Test of soundness of these mortars meets the requirements from standard EN 196-3. From the samples of mortars without and with different addition of setting regulator, test elements 4 × 4 × 16 cm were prepared according to standard EN 196-1 for evaluation of compressive and flexural strength. Because of low initial strength, demolding was done after 2 days of hydration and test elements were stored in humidity chamber (see standard EN 459-2), that provided temperature 20 °C and humidity higher than 90 %. Results of strength development of prepared hydraulic mortars are given in Table 6.

Table 6. Compressive (c. s.) and flexural (f. s.) strength in MPa according to EN 196

Samples	7 days		28 days		90 days	
	f. s.	c. s.	f. s.	c. s.	f. s.	c. s.
W1 + 0.1 wt. % t.a.	0.8	1.9	3.1	10.0	6.0	40.3
W2	0.2	0.8	1.9	3.4	3.3	11.2
W2 + 0.1 wt. % t.a.	-	0.6	0.6	1.6	2.5	14.1
W2 + 0.2 wt. % t.a.	-	0.6	0.3	0.8	0.3	1.5
W2 : LL 3 : 2	-	-	0.4	1.8	2.1	10.2
W2 : LL 2 : 3	-	-	0.4	2.4	1.6	8.4

Higher addition of setting regulator has a positive effect on prolonging of working time, but it also causes lower resulting strength. When limestone is added before burning, binder with great workability and good strength is formed after initial slaking. According to the results, it is obvious that the binder has sufficient strength for the use as a hydraulic binder into plaster mixtures or other applications.

Conclusions

In terms of literature information and also our own research, it is evident that solution for recycling of asbestos waste from building and dumps is very actual and necessary.

The research on asbestos-cement products was conducted and the way of possible liquidation proposed. It was found that this dangerous waste can be thermally decomposed and used as hydraulic binder.

Redesign can be done by burning of the waste in original state without any mechanical changes with prospective addition of bulk limestone in shaft or tunnel kiln at temperatures from 1,100 to 1,250 °C.

Burned product ground to proper specific surface with or without addition of setting regulator forms a hydraulic mortar.

This material has quality technological parameters appropriate for various applications in building industry. The material can be used as supplementary cementitious material. It could be used in the production of blended cements, dry plaster mixtures or as an admixture in concrete.

Acknowledgement

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