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Effect of high temperatures on gypsum-based composites

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HIGHLIGHTS

- Gypsum-based pastes and mortars were exposed to the temperatures up to 1000 °C.
- Volume changes of mortars were three times smaller than volume changes of pastes.
- Pure gypsum demonstrated the higher residual compressive strength at 1000 °C.
- Gypsum-lime material featured the least favourable behaviour at high temperatures.
- Properties of gypsum-lime-silica fume binder are sufficient for common use.

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ABSTRACT

The behaviour of several gypsum-based materials at high temperatures was investigated. Three gypsum-based binders, pure gypsum, binary gypsum-lime binder and ternary gypsum-lime-silica fume binder were exposed to temperatures up to 1000 °C. From each binder paste (binder and water) and mortar (binder and mortar and silica sand) were prepared. The basic physical and mechanical properties of the materials after heating were measured. With the exception of gypsum-lime paste, all the materials performed well at temperatures up to 400 °C. Gypsum paste achieved the best residual strength at 1000 °C (about 30% of the original value). The volume changes could be effectively reduced by adding silica sand, but the strength of the mortars was about 10% lower than the strength of the pastes. The volume changes in mortars were three times less than the volume changes of the pastes. The presence of aggregates also prevents the disintegration of gypsum-based materials. The best resistance against high temperatures was found for pure gypsum materials. The ternary materials have similar residual strength to cement-based pastes and mortars, while the behaviour of the binary materials was significantly worse.

1. Introduction

Safety in case of fire is one of the basic requirements for structures [1] and therefore it is important to obtain detailed knowledge of the fire behaviour of every building material that is specified in the construction design. One of the materials which can be considered to be fire-resistant is gypsum, which is often used for fire protection of other materials. Gypsum plaster has been extensively used as fire protection of load-bearing structures since medieval times [2]. However, knowledge of fire protective properties of gypsum was based mostly on the experience of builders. The first scientific results appeared only in the last century, where the first thermal analyses by West and Sutton [3] and Khalil et al. [4] were performed and also the first fire tests of gypsum were carried out [5].

The utilization of gypsum as a building material is currently increasing because gypsum is one of the most environmentally friendly binders. New applications are sought for gypsum and new materials are being designed and tested [6,7]. In order to use these materials sensibly in buildings, their fire-resistance properties must be known. It was found that, while the mechanism of action of pure gypsum as a fire protection is described sufficiently [8,9], there is a lack of knowledge about the behaviour of other gypsum-based materials. Recently, research has been focused mainly on the fire-resistant properties of gypsum boards, because gypsum plasterboards are the most commonly used gypsum-based building products today. The thermochemistry of common gypsum board at elevated temperatures was described by Kontogeorgos and Founti [10]. The thermal properties of gypsum boards at elevated temperatures were described e.g. by Thomas [8] and by Park et al. [11] and thermal models were proposed by Thomas [6], Lázaro et al. [12] Weber [13] and Mehaffey et al. [14].

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Gypsum exposed to high temperature exhibits significant shrinkage, which could be reduced by a suitable filler. Vermiculite is commonly used as an additive to mitigate shrinkage at high temperatures in commercial products, but vermiculite significantly decreases the mechanical properties of gypsum. Martias et al. [15] found, that the addition of vermiculite in proportions between 5 and 25 w% leads to a decrease of 80% of the Young modulus. Féjean et al. [16] achieved good results by the addition of an inorganic filler, an unspecified industrial by-product. Material with 60% of inorganic filler had four times smaller shrinkage at 200 °C than pure gypsum. Common aggregates, such as silica sand, are also used in gypsum-based mortars (e.g. in plasters, gypsum blocks or self-levelling floors), mainly for economic reasons, because gypsum does not shrink at normal temperatures. Nevertheless, no information about the behaviour of gypsum-based materials with this type of aggregates at high temperatures was found in the literature.

There is also a lack of information about the behaviour of composed gypsum-based binders at high temperatures. Some composite gypsum-based binders were used traditionally (e.g. gypsum-lime binders) and new materials are being intensely tested. These new materials are designed to eliminate the main disadvantage of gypsum, which is its loss of the mechanical properties in a wet environment. A wide range of composed gypsum-based materials with improved resistance against moisture have been studied. The best results were achieved with ternary materials containing, in addition to gypsum, a pozzolan material and activator of the pozzolanic reaction [6,17–19]. While it was found that cement-based ternary materials performed well up to 450 °C [20], the behaviour of ternary gypsum-based materials at higher temperatures has not yet been systematically tested.

The aim of our research was to provide lacking information about the behaviour of some less-studied gypsum-based composites at high temperatures in order to obtain more complex knowledge about modern gypsum-based materials. The obtained information could contribute to broader utilization of this interesting and environmentally very promising material.

Three gypsum-based binders were tested – pure gypsum as a reference material, binary gypsum-lime binder and ternary gypsum-lime-silica fume binder. A paste (mixture of binder and water only) and mortar (mixture of binder, water and silica sand) was prepared from each binder. The materials were heated to temperatures up to 1000 °C and their basic physical and mechanical properties were tested and compared.

2. Thermo-chemistry of used materials

Calcined gypsum (commonly known as a Plaster of Paris) in powder form consists mainly of calcium sulphate hemihydrate $CaSO_4{\cdot}1/2H_2O$, which is prepared by dehydration of calcium sulphate dihydrate $CaSO_4{\cdot}2H_2O$ (natural gypsum rock or an industrial by-product, e.g. from flue gas desulphurization of coal power plants or from phosphoric acid production).

During heating, hardened gypsum (i.e. calcium sulphate dihydrate) undergoes two endothermic decomposition reactions in which water of crystallization is removed. Initially, gypsum is converted to calcium sulphate hemihydrate (1). This reaction starts at about 60 °C [21]. Then calcium sulphate hemihydrate is converted into calcium sulphate anhydrite (2), usually at temperatures below 200 °C. The temperatures of both conversions could vary significantly, because they are influenced by a great many factors – e.g. the type of gypsum, origin, presence of impurities, rate of heating and partial water vapour pressure [10,14,16,22]. Gypsum dehydration therefore takes place in a relatively large temperature range between 60 °C and 300 °C [10,23].

$$CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 \cdot 1/2H_2O + 3/2H_2O \quad (1)$$

$$CaSO_4 \cdot 1/2H_2O \rightarrow CaSO_4 + 1/2H_2O \quad (2)$$

After the second dehydration, different forms of anhydrite are formed at different temperatures. First anhydrite III is formed, and this is then transformed into anhydrite II at a temperature over 200 °C. Anhydrite II is transformed from a slowly soluble to an insoluble form (between 300 and 600 °C) and then to Estrich gypsum (above 600 °C). Anhydrite I is usually formed at temperatures above 900–1200 °C [24]. Finally, the calcium sulphate decomposes to CaO and SO_3 (3) [23].

$$CaSO_4 \rightarrow CaO + SO_2 + 1/2O_2 \quad (3)$$

The reactions of other materials, which were contained in tested materials, also have to be considered. The type of aggregate plays an important role in gypsum-based mortars. Standardized silica sand was used in our experiments; this material is chemically stable below 1000 °C, but is volume-unstable at temperatures below 1000 °C. The volume instability is caused by the phase transformation of SiO_2. First a significant volume change occurs at 573 °C, when inversion between α- and β-quartz occurs. This phenomenon is accompanied by a 1.4% increase in volume. At 870 °C tridymite is formed and the volume of the aggregate increases again by 14.8% [25–27].

Hydrated lime and silica fume were also used in the binary and ternary materials. The behaviour of lime at higher temperatures could be described by two reactions – dehydration of calcium hydroxide (4), which occurs at about 450 °C [28], and calcium carbonate (formed by carbonation) decomposition (5), which occurs at about 800 °C [29].

$$Ca(OH)_2 \rightarrow CaO + H_2O \quad (4)$$

$$CaCO_3 \rightarrow CaO + CO_2 \quad (5)$$

Silica fume contains mostly amorphous SiO_2, which is chemically stable at temperatures under 1000 °C; however the decomposition of C-S-H phases, which were created by the pozzolanic reaction with calcium hydroxide, could be observed at the temperatures between 100 and 200 °C [30].

The gypsum employed also contained some amount of muscovite $KAl_2(Si_3Al)O_{10}(OH)_2$ (see Table 3), whose dehydroxylation has been reported between 650 and 850 °C [31].

3. Experimental

3.1. Materials and compositions

Six gypsum-based materials (three pastes and three mortars) were tested. Three pastes were prepared: pure gypsum paste (GP) as a reference material, binary gypsum-lime paste (GLP) and ternary gypsum-lime-silica fume paste (GLSP). Three mortars (GM, GLM, GLSM) with silica sand were prepared using the same binders. In composite binders, the amount of gypsum was significantly greater than amounts of the other dry components (lime and silica fume). The compositions of all the tested materials are given in Table 1.

The grey gypsum employed is a commercial product (producer Gypstrend, Czech Republic), which complies with EN 13279-2 [32]. In the binary binder, commercial white hydrated lime (producer Lhoist, Czech Republic) was used as a second binder., In addition to gypsum and lime, silica fume (commercial name Stachesil S, producer Stachema CZ, Czech Republic) was also added to the ternary binder. The elemental composition of all the binders, measured by a X-ray fluorescent scanning spectrometer SPECTROSCAN MAKC-GV, is shown in Table 2.