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Design and basic properties of ternary gypsum-based mortars

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Abstract. Ternary mortars, prepared from gypsum, hydrated lime and three types of pozzolan were designed and tested. As a pozzolan admixture crushed ceramic, silica fume and granulated blast slag were used. The amount of pozzolans in the mixtures was determined according to molar weight of amorphous SiO₂ in the material. The samples were stored under the water. The basic physical properties and mechanical properties were measured. The properties were compared with the properties of material without pozzolan. The best results in the water environment were achieved by the samples with silica fume.

1. Introduction
Gypsum is "airy" binder and therefore it could be used only in the indoor environment. Moisture causes deterioration of the mechanical properties, especially the loss of compressive strength [1]. Production from secondary raw materials, unpretentious production process, recyclability and good behavior in fire are reasons why better moisture resistance of gypsum is tried to be solved nowadays. Combination of gypsum and pozzolan is one possible solution. The pozzolan needs the alkaline environment and presence of calcium hydroxide to activate the pozzolanic reaction and the formation of hydraulic products (CSH phases and ettringite), therefore hydrated lime is added to the mixture of gypsum and pozzolan. The mixture thus contains three binders and it is denoted as ternary binder. The authors have already studied ternary gypsum-based binders and improvement of moisture resistance was demonstrated [2]. Adding of fine sand to the ternary gypsum binder could extends the possibility of using this binder in the buildings. The type of the sand has effect on the workability of mixtures and the properties of samples as stated in the article [3].

2. Materials and mixtures
Studied mortars were composed of ternary gypsum-based binders and filler. As a filler the standardized sand (ČSN EN 196-1 [4]) was used. It was supplied by the company Filtrační písky, Ltd in Chlumec, Czech Republic. The sand has the high chemical purity (99% of SiO₂) and it has low content of iron oxide.

Ternary binders were composed of gypsum, hydrated lime and three different types of pozzolan (silica fume, crushed ceramic and granulated blast slag). The main component of all binders was gypsum and it was represented more than 50 % in the composition of binders. The used gypsum is common commercial product (producer Gypstrend Ltd, Czech Republic). Hydrated lime was used as the alkaline activator of pozzolanic reaction in the mixture. Used lime hydrate CL-90-S is commercially available product of producer Carmeuse CR.
Silica fume is the commercial product also (producer Stachema, CR), it is sold under the name Stachesil S and it contains 90 % of amorphous SiO₂. Used crushed ceramic is a waste material from the production of brick blocks. This material was sifted before using in the mixture so it doesn’t contain grains larger than 250 µm. Granulated blast slag (GBS) is produced by Kotouč Štramberk under the name SMŠ 400. Chemical composition of crushed brick and granulated blast slag is stated in the table 1. The phase composition and the amount of amorphous SiO₂ were determined by X-ray diffraction analysis.

Table 1. Chemical composition of used materials.

|          | Crushed ceramic | GBS  |
|----------|-----------------|------|
| SiO₂     | 51.0            | 36.0 |
| Al₂O₃    | 20.0            | 9.0  |
| Fe₂O₃    | 6.1             | 0.3  |
| CaO      | 11.6            | 43.5 |
| MgO      | 4.7             | 8.3  |
| K₂O      | 3.2             | 0.5  |
| Na₂O     | 1.3             | 0.5  |
| TiO₂     | 0.8             | 0.3  |
| SO₃      | 1.0             | 0.5  |

2.1. Designed of mixtures

Composition of each mixture was designed with requirement to compatibility of the achieved results and therefore dosage of pozzolan was designed according to amount of silicon in the pozzolan. All mixtures contain the same amount of reactive phase and alkali activator, therefore we can compare results of measurements. The default condition was that the ratio of the molar amount of calcium (n₉₅) and silicon (nₛ₁) was 1.5 (1).

The values needed for the calculation are summarized in the table 2. The percentages of molar amount of silicon (Mₛ₁) in the molar amount of amorphous SiO₂ (Mₛ₂) were calculated at the first step. The percentages of molar amount of calcium (M₉₅) in the molar amount of Ca(OH)₂ (M₉₅) were estimated in the same way. These values and the values in table 2 were used to calculate amount of Si in each pozzolan. The amount of Si in each pozzolan and amount of Ca in lime hydrate are in the table 3. By substitution the equation (2) into the equation (1), we obtain the weight ratio of silicon and calcium (3). The pozzolans and lime contain also other ingredients than the silica oxide and calcium hydroxide (table 2) and therefore the weight ratio of the silica oxide and calcium hydroxide was calculated to the weight ratio of each material.

\[ n_{Ca} n_{Si}^{-1} = 1.5 \]  
\[ n = m M^{-1} \]  
\[ m_{Ca} m_{Si}^{-1} = 2.14 \]

n ..... molar amount [mol]; m ..... weight [g]; M ..... molar weight [g.mol⁻¹]
Table 2. The amount of amorphous SiO$_2$ and Ca(OH)$_2$ in the materials.

|                | Silica fume | Crushed ceramic | GBS   | Lime |
|----------------|-------------|-----------------|-------|------|
| **Amorphous SiO$_2$** | 90 %        | 45.3 %          | 33.6 %| -    |
| **Ca(OH)$_2$**    | -           | -               | -     | 95 % |

Table 3. The amount of Si and Ca in the materials.

|                | Silica fume | Crushed ceramic | GBS   | Lime |
|----------------|-------------|-----------------|-------|------|
| **Si**         | 42 %        | 11 %            | 15.7 %| -    |
| **Ca**         | -           | -               | -     | 51.4 %|

Compositions of designed mixtures are in the table 4. The alkaline activator (lime hydrate) was added in the same amount in all mixtures. The amount of pozzolan was added per unit of quantity of lime according to a previous calculations (1), (2), (3). The sand contained three fractions (0 - 0.5 mm, 0.5 - 1mm and 1 - 2 mm), which were mixed in the ratio 1:1:1. The ratio of ternary gypsum-based binders and sand was 1:1.35. The amount of water in the mixture was determined by flow test. The overflow was established to be between 185 to 190 mm with respect to the workability of mortars. The amount of water is calculated as a ratio between mass of water and mass of ternary binder in the mixture. Composition of designed mixtures are in the table 4. The mixture marked COM is the reference mortar, prepared from gypsum and lime without pozzolan.

Table 4. Composition of designed mortars.

| Mixture | Composition [%] | Water/binder ratio |
|---------|-----------------|--------------------|
|         | Pozzolan | Gypsum | Lime | Sand |             |
| **SF**  | Silica fume   | 3.6    | 32.6 | 6.4  | 57.4 | 0.8        |
| **CC**  | Crushed ceramic | 12.8  | 23.4 | 6.4  | 57.4 | 0.7        |
| **GBS** | GBS          | 9.6    | 26.6 | 6.4  | 57.4 | 0.75       |
| **COM** | -            | -      | 36.2 | 6.4  | 57.4 | 0.73       |

2.2. Procedure of preparing and testing

All the designed mixtures were prepared in the same way. The dry binder components (gypsum, lime, pozzolan) were weighed and were mixed thoroughly together. Thereafter the water was metered in the graduated cylinder and it was put in the bowl together with dry binder components. This mixture was mixed mechanically in the standard mixer for 60 s at low speed, than sand was added and material was mixed for 30 s at high speed, than wiped of manually and mixed again for 60 s at high speed. The samples were unmolded 2 hours after production. The samples were stored in the laboratory for one day and thereafter they were put into the water and stored there until the time of testing.

Bulk density was determined by gravimetric method from dimensions and mass. Pore size distribution was measured by a mercury porosimetry using apparatus Pascal 140 + 440 (Thermo Electron). Tensile and compressive strength were measured on set of prisms 160 × 40 × 40 mm according the standard ČSN EN 13279-2 [5]. Samples were taken out of the water and they were dried on the surface immediately before testing. The testing of mechanical properties was carried out by mechanical press FP 100 (VEB Industriewerke Ravenstein).
3. Results and discussion

3.1. Basic properties
The basic properties were measured on the samples at the age of 28 days. The mass moisture and bulk density of samples are in the table 6. The bulk density was measured on the dried samples. Values of bulk density are similar for all samples (between 1506 - 1586 kg·m⁻³). The mass moisture was measured because the samples were not dried before the measuring of mechanical properties. The samples without pozzolan (COM) contained more moisture than the samples with pozzolan. The crushed ceramic (CC) achieved the biggest moisture among the samples with pozzolan. The lower moisture content in the samples with pozzolan is caused by the finer porous structure of these samples. On the figure 1 we can see the distribution of pores in the mixtures. The mixtures with pozzolan have the greatest amount of pores between 0.1 - 1 μm, while the samples without pozzolan have the greatest representation of pores in the range from 1 to 10 μm. The thermogravimetric analysis was proved the development of CSH phases and ettringite. Finer structure of samples with pozzolan is determined by the formation of these phases.

| Material | Moisture by mass (%) | Bulk density [kg/m³] |
|----------|----------------------|----------------------|
| SF       | 20.88                | 1586                 |
| CC       | 22.13                | 1525                 |
| GBS      | 20.82                | 1547                 |
| COM      | 25.27                | 1506                 |

![Figure 1. Distribution of pores in the mixtures.](image-url)

3.2. Mechanical properties
The mechanical properties were measured at the age of 7, 28 and 90 days. The measured values of mechanical properties are given in the table 6 and table 7. The moisture content has big influence
on the values of strength. The values of compressive strength are especially important for use in the building practice.

The values of the compressive strength are plotted in figure 2. We can observe that the highest compressive strength have the samples with silica fume (SF). These samples have also the highest increase of compressive strength in time, the compressive strength increased more than three times in the period from 7 to 90 days. The samples with granulated blast slag (GBS) have the similar behaviour but the increase of strength in time is not as significant as for the samples with silica fume (SF). The gradual increase of strength is caused by the formation of CSH phases and ettringite. The samples without pozzolan (COM) show the typical decrease of compressive strength influenced by moisture. Worst values of compressive strength have the samples with crushed ceramic (CC). Decrease of compressive strength at the age 90 days was caused by the volume changes, which were the largest of all materials and caused severe cracking on the surface of the samples (figure 3). However, the integrity of the samples with crushed ceramic was preserved.

| Table 6. Tensile strength [MPa]. |
|----------------------------------|
| 7 days  | 28 days  | 90 days |
| SF      | 1.1  | 2.7  | 3.6  |
| CC      | 0.6  | 0.6  | 0.7  |
| GBS     | 1.0  | 1.6  | 1.9  |
| COM     | 0.9  | 0.9  | 0.7  |

| Table 7. Compressive strength [MPa]. |
|-------------------------------------|
| 7 days  | 28 days  | 90 days |
| SF      | 2.1  | 5.3  | 7.1  |
| CC      | 1.1  | 1.4  | 0.8  |
| GBS     | 1.8  | 3.1  | 3.4  |
| COM     | 2.0  | 1.9  | 1.5  |

![Figure 2. Compressive strength of samples in time.](image)
4. Conclusions
The ternary gypsum-based mortars were designed with the main aim to obtain the comparable amount of amorphous SiO$_2$ in three different ternary gypsum-based mortars. Basic physical and mechanical properties of the mortars, prepared according to the design, were tested.

We can say, on the basis of obtained results, that some pozzolan additives are suitable for the use in the gypsum-based mortars in wet environment. The mortars with silica fume (SF) and granulated blast slag (GBS) have higher compressive strength than the mortar without pozzolan (COM). The compressive strength of these mixtures increases in time, while reference mortar without pozzolan loses its strength gradually, when stored in water. Mortar with silica fume (SF) has more than four and half times higher compressive strength at the age 90 days than the mortar without pozzolan (COM) at the same age. The crushed ceramic seems to be unsuitable for the use in gypsum-based mortars because of unacceptable volume changes and low strength of the material.

Development of strength at longer time intervals is studied presently. In the next step further properties of these materials will be investigated, e.g. other physical, thermal and moisture properties. The ternary mortars with improved resistance against humidity can be used similarly as lime or cement based mortar, e.g. as exterior plasters.

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