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Performance of ternary gypsum-based mortars in wet environment

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Abstract. Gypsum is one of the most environmentally friendly materials, but it could be used only in the dry environment. When gypsum is wet, its mechanical properties worsen substantially and namely its strength decreases significantly. Ternary gypsum-based binders, composed from gypsum, lime and pozzolan evince better resistance against the moisture, because water-resistant phases are created as products of pozzolanic reaction. The behaviour and properties of several gypsum composites with different types of pozzolans, exposed to the moisture, were investigated. Samples of ternary composites were stored in the water for 360 days and their properties were tested during the period and compared with the properties of gypsum mortar. While gypsum mortar degraded significantly, the ternary mortars retained its properties and the strength of some of them even increased.

1. Introduction

The gypsum is one of the most environmentally friendly materials and this is the reason why the gypsum is nowadays extensively studied. Wider use of gypsum in the construction process is restricted to its placement only in the interiors without the presence of moisture. The loss of strength caused by the wet environment is generally known [1]. One possibility how to improve the moisture resistance of gypsum is to combine the gypsum with pozzolan and ensure the activation of pozzolan in gypsum. Predominantly the ternary gypsum-based materials combining gypsum, lime or cement and different types of pozzolan are tested [2]. Fraire-Luna et al. [3] compared pure gypsum material with materials containing blast furnace slag and metakaolin, which have been stored under the water for 360 days and found out that the resistance of composed materials is better.

The solution to the problem of the loss of mechanical properties in the wet environment is thus the creation of ternary gypsum pastes. While the ternary gypsum-based pastes were studied extensively, the long-term durability of gypsum mortar has not yet been researched sufficiently. In general, we can say that the gypsum composites with any filler are described in the literature only rarely. Herrero et al. [4] focused on recycling by using the mechanical grinding of rubber particles from end-of-life tires as a filler in their article. The behaviour of the gypsum paste with a combination of the different types of sand was studied by authors in the previous article [4]. The next logical step is the description of the behaviour of ternary binders in combination with the filler. Therefore, the research of the ternary gypsum mortars, especially of their mechanical properties and also moisture resistance, is an important task.

2. Materials

Four gypsum-based composites and one gypsum mortar as a reference were tested. Gypsum, lime, three pozzolans were used as dry components of the mixtures. The specification of the used materials is in the table 1.

Table 1. Materials specification.

	Specifications	Producer
Gypsum	Gypsum binder G2 BII	Gypstrend s.r.o.
Lime	CL 90 S	Vápenka Čertovy schody a.s.
Crushed ceramic	-	HELUZ cihlářský průmysl v.o.s.
Silica fume	Stachesil S	STACHEMA CZ s.r.o.
GBS	Granulated blast furnace slag (SMŠ 400)	KOTOUČ ŠTRAMBERK, spol. s r.o.
Aggregates	Standardized sand (CEN, ČSN EN 196-1)	Filtrační písky spol. s.r.o. (Chlum)

The final compositions of tested mortars are in the table 2. The composites were composed of ternary or binary binder and sand. The gypsum mortar (G) was made as comparative material. This mortar contents only gypsum and sand. The binder to sand ratio was 1:2. The dosage of pozzolan in the ternary binders was calculated according to the amount of the amorphous phase (determined by XRD analysis; amorphous phase of crushed ceramic is 45.3%; silica fume - 90%; GBS - 87.7%) in order to achieve the same amount of amorphous phase in each mixture and therefore the results could be compared. The amount of lime was the same for all mixtures. The water/binder ratio was determined by the flow test for diameter 185 ±5 mm.

Table 2. Material compositions.

Designation of the mixtures	Pozzolan		Gypsum	Lime	Sand	Water/binder ratio
	[%] by mass					[-]
KC	Crushed ceramic	5.6	24.4	3.3	66.6	0.78
KM	Silica fume	2.8	27.2	3.3	66.6	0.82
KS	GBS	2.9	27.1	3.3	66.6	0.78
K	-		30.0	3.3	66.6	0.81
G	-		33.3	-	66.6	0.90

3. Methods

3.1. Basic properties

The basic properties were measured on the samples at the age of 28 days. The density ρ of tested materials was measured by helium pycnometry (Pycnomatic ATC, Thermo Fisher Scientific). The gravimetric method was used for determination of bulk density of samples. The porous structure was studied by mercury intrusion porosimeter Pascal 140 and 440 (Thermo Fisher Scientific).

The microstructure of all samples was studied by scanning electron microscopy (SEM) using a ZEISS Merlin electron microscope with Gemini II column at an acceleration voltage of 7-10 kV, probe current of 46–71 pA and working distance of 8–13 mm. The samples were dried and then covered with a carbon and they were not polished.

3.2. Compressive and tensile strength

Production of samples for strength measuring was managed according to EN 13279-2 [6]. Two sets of test specimens were made for each period of measuring. The mechanical properties were measured after 7, 28, 90, 180 and 360 days. Each test set contained three prisms 160 × 40 × 40 mm. Samples were

stored in laboratory condition (marking A) and also under the water (marking W). Samples were not dried before the testing. Bending strength f_f [MPa] was determined by three-point flexural test by mechanical press FP 100 (VEB Industriewerk Ravenstein) and compressive strength f_c [MPa] was tested after the bending test. The strength tests were also performed according to standard EN 13279-2. Moisture by mass was determined immediately after the compressive strength test.

4. Results and discussion

The values of bulk density, density and porosity are shown in table 3. The density of samples stored in the laboratory condition is the same as density of samples stored under the water, Density of all materials is about 2550 kg·m^{-3}. The storage condition, however, had an effect on the bulk density and porosity of the samples.

The porosity of all materials was lower when the samples were stored under the water (W). The decrease of porosity of ternary composites (KC, KM, KS), stored under water is caused by the formation of CSH phases and ettringite, because wet environment is more favourable for pozzolanic reaction and therefore the faster transformation of the structure. The presence of these phases has been proven by scanning electron microscopy. The structure of ternary composites stored under the water (W) is distinctly finer, as depicted in figure 1, where the structure of composites with crushed ceramic after 360 days is shown. The presence of ettringite and CSH phases was observed e.g. by Fraire-Luna et al. [3] and Escalante-Garcia et al. [7][8].

The gypsum mortar (G) has the highest values of porosity. The slightly lower porosity of samples stored under the water is caused by the dissolution of gypsum.

Table 3. Bulk density, density and porosity at the age of 28 days.

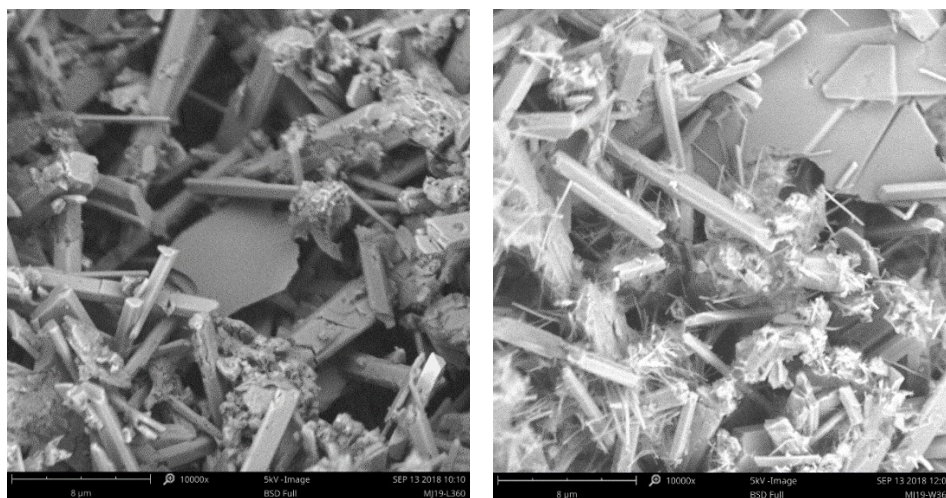| Designation of the mixtures	**A** - laboratory environment			**W** - stored under the water		
	ρ_V [kg·m^{-3}]	ρ [kg·m^{-3}]	p [%]	ρ_V [kg·m^{-3}]	ρ [kg·m^{-3}]	p [%]
KC	1627	2552	36.2	1709	2576	33.7
KM	1576	2520	37.5	1698	2551	33.5
KS	1629	2574	36.7	1747	2549	31.5
K	1529	2553	40.1	1716	2540	32.5
G	1459	2536	42.5	1511	2551	40.8

Figure 1. Structure of composite with crushed ceramic (*KC*): left - storage in the laboratory, right - storage in water.

The values of the compressive strength were evaluated by Dean-Dixon test [9]. The results of compressive strength are in figures 2 and 3. The moisture by mass (right axis) is added because the samples were not dried before the test. The values of moisture in the individual time period are connected by the line for better understanding.

The moisture by mass of composites stored in the laboratory environment (A) was in the range from 0.1 to 0.4%. The gypsum mortar (G) showed to hygroscopic behaviour, which is typical for the gypsum and therefore its moisture by mass is higher than the moisture of gypsum composites. The values are between 2.3 and 3.1%. The compressive strength of the materials stored in the laboratory environment (A) and its development in time are shown in figure 3. There is not any substantial loss of compressive strength for each material over time. That was expected because the gypsum was not exposed to increased moisture load.

Really interesting is the slight decrease in compressive strength at the age of 180 days. The decrease in samples with pozzolan was more pronounced than the decrease in samples without pozzolan. The decrease is caused by crystallization changes in the structure with the result in loss of strength. Similar decrease of compressive strength was observed by Fraire-Luna et al. [3] and Magallanes-Rivera and Escalante –Garcia [11]. The compressive strength at the age of 360 days increased again. In a further study of these systems, it would be appropriate to perform scanning electron microscopy supplemented by EDS analysis which could help to clarify these changes of compressive strength at the 180 days.

The more fundamental are results of samples stored under the water (W) (figure 3). The compressive strength of gypsum mortar decreased during the time, as expected [1]. The strength of gypsum mortar is significantly lower than the strength of samples, stored in the laboratory condition (A). The moisture by mass was higher, between 17.1 and 22.3%. The maximum moisture by mass of all composites was 14.6%. The decrease of strength and higher value of moisture by mass were accompanied by an increase of volume, see figure 4. The composite without pozzolan (K) had lower values of compressive strength than the other composites. The values were constant in the time.

The compressive strength of composites with pozzolan (KC, KM, KS) was growing over the time. The compressive strength of composite with crushed ceramic (KC) was increasing smoothly gradually. Compressive strength at the age of 360 days was three and a half times higher than the strength at the age of 7 days. The development of strength of samples with GBS (KS) was similar but the increase of strength was less than twofold. The increasing of strength of the materials with pozzolan was proven by other authors [3][7][8][12], while other authors the use of ternary gypsum based materials in wet environment do not recommend [13], [14].

The composites with silica fume (KM) achieved the highest value of compressive strength at all times. Its strength was 6.04 MPa at the age of 360 days and it was more than threefold than the value at the age of 7 days. The most significant increase took place up to 90 days. If we compare the composite with silica fume stored in the laboratory environment (KM – AU) with the same composite stored under the water (KM – WU), we can see that the compressive strength of samples stored in the laboratory was relatively constant at all time. On the contrary, the strength of samples stored under the water increased gradually. The compressive strength of samples at the age of 7 days was more than three times lower when the samples were stored under the water.

The compressive strength of composites with silica fume was the same for both types of storage (around 6 MPa) after 360 days. Use of silica fume as the pozzolan addition in the ternary gypsum-based composites seems therefore very appropriate.
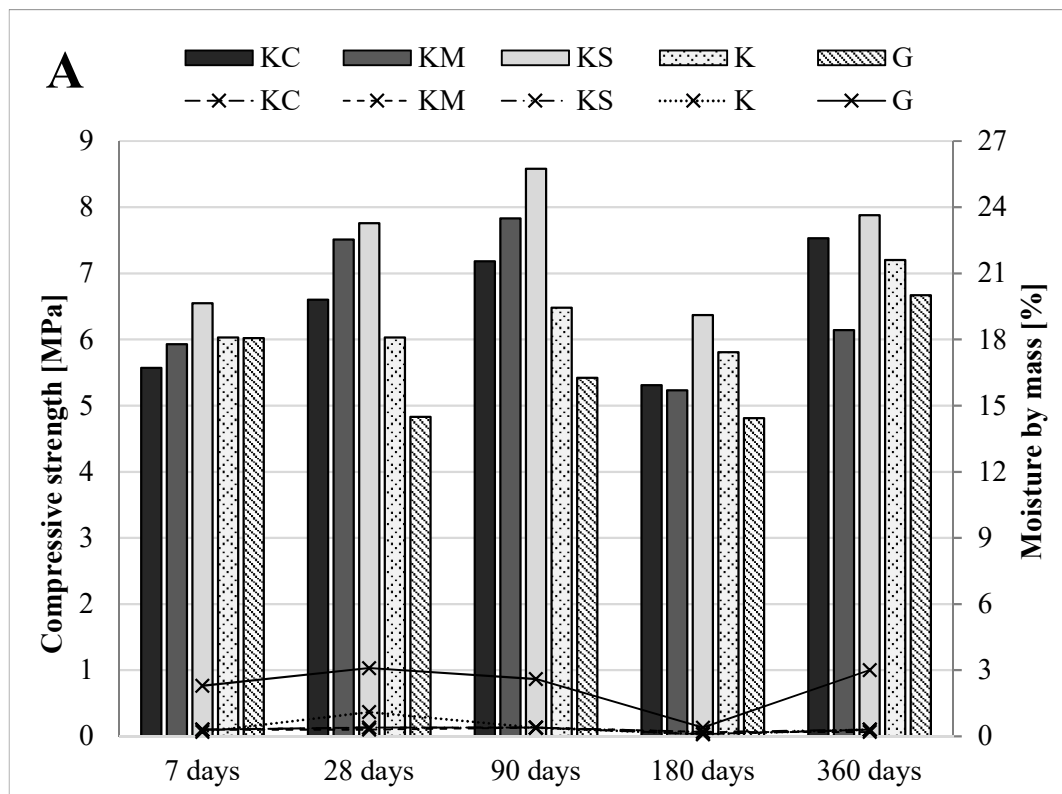
Figure 2. Mechanical properties and moisture by mass of samples stored in the laboratory (A).
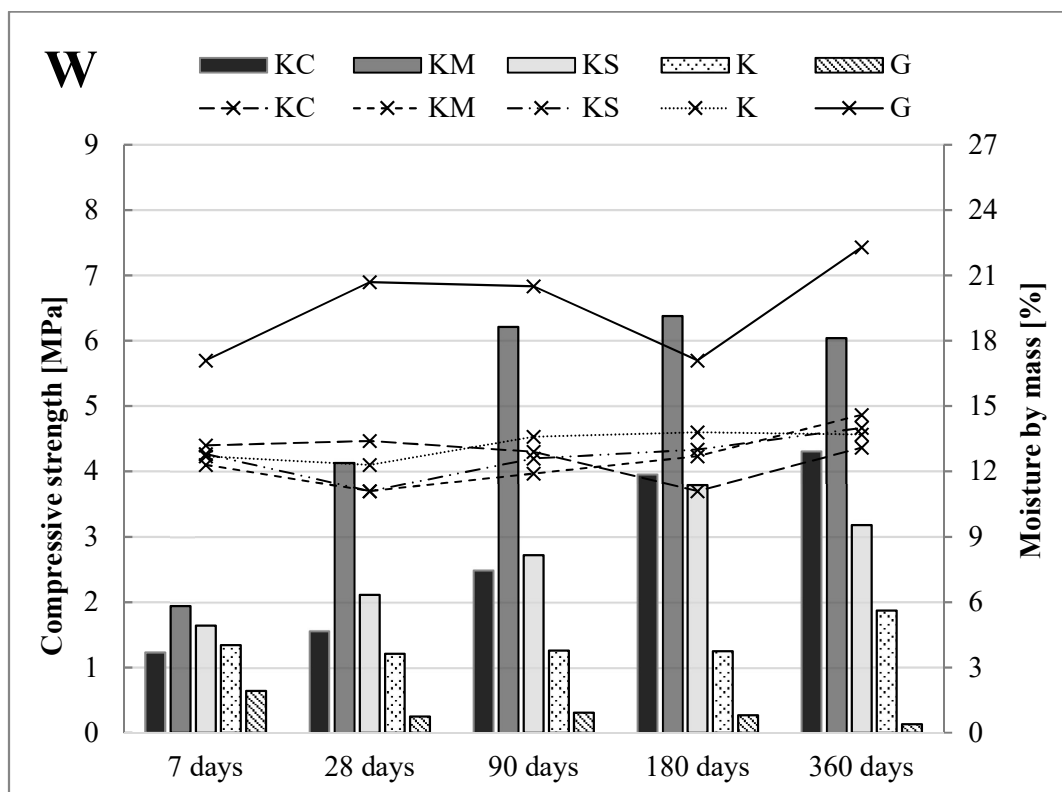

Figure 3. Mechanical properties and moisture by mass of samples stored in the water (W).

Figure 4. Photo of the gypsum-based composites and gypsum mortar stored under the water (W), 360 days old; from the left: crushed ceramic-KC, Silica fume-KM, GBS-KS, without pozzolan-K, gypsum mortar-G.

5. Conclusions

The influence of the storage on the mechanical properties of gypsum composites and gypsum mortar was investigated. The samples were stored in two environments, in the laboratory and under the water. The compressive strength of all materials was measured at 7, 28, 90, 180 and 360 days. The basic properties, i.e. bulk density and porosity depend on the storage conditions. The values of compressive strength of all materials stored in the laboratory conditions were over 4.8 MPa, regardless of the age. The storage under the water had a more fundamental impact on the values of compressive strength. The gypsum mortar had a negligible compressive strength at the age of 360 days. The binary composite did not lose strength during the storage under the water, the values were up to 1.87 MPa. The compressive strength of ternary composites was increasing during the time. The ternary composite with silica fume proved the best results. Its compressive strength at the age of 360 days was the same for both types of storage. This combination is therefore considered as a good solution if we want to improve the resistance of gypsum against wet environment.

Acknowledgements

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