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To cite this article: J Majerova and R Drochytka 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **385** 012034

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The influence of the addition of gypsum on some selected properties of lime-metakaolin mortars

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Abstract. The renovation and replacement of historical plasters is still a very current topic. Today's trend is for the production of dry mortar mixtures that can be used for the renovation of historic buildings. The article therefore focuses on the preparation of gypsum-modified lime-metakaolin mortar. An experiment consisted of two stages. In the first stage of the experiment, the basic technological properties of fresh and hardened mortars were examined (bulk density of fresh and hardener mortar, compressive and flexural strength). The second stage aimed at monitoring the hydration process in different environments. Environments should simulate real factors affecting the plaster (humidity, low and higher temperatures). To determine the phase composition, the X-ray diffraction analysis was used. The results showed that gypsum in the lime-metakaolin plaster caused an increase in compressive and flexural strength. The presence of gypsum also conducted to the formation of ettringite and this has caused volumetric changes.

1. Introduction

Almost all existing building materials (such as mortar, concrete and plaster mixtures) are designed using Portland cement. However, Portland cement is a modern binder and has been widely used only for the last hundred years, so it is not appropriate to use in the reconstruction of historical buildings. When reconstructing historic buildings, the materials used should be a combination of materials that best substitutes the original binders used [1].

The first lime paints and plasters date back to 7500 BC and come from the area of today's Jordan. In Syria, gypsum was used as a wall finishing from as early as 7000 BC. These plasters were often decorated with paintings. In some cases, pozzolanic admixtures have been added to the lime plasters because they speed up the hardening of fresh plaster and increase its strength. The beneficial effect of the addition of ash from volcanic areas has also been described by the Roman writer Marcus Vitruvius Pollio in his work Ten Books on Architecture [2-4].

In our region (Czech and Slovak Republic), limestone was for a long time the most commonly used material; this is due to the prevalent geological conditions in the area, especially the strong karst areas [5]. The first evidence of the use of lime comes from the remains of the furnaces used for lime burning that have been found in several Stone Age archaeological locations. It is known that lime was also used in the areas of Great Moravia and Bohemia, as a means of painting the walls. The lime-burning process was poorly supervised and even low-quality limestone was burned. This resulted in the presence of hydraulic oxides (Al_2O_3 , Fe_2O_3 , SiO_2), which formed the natural hydraulic lime. These limes are characterised by good plasticity and higher strengths than found in present-day lime. The plaster was added to the mortar mostly to accelerate the hardening process or to achieve higher strengths.



2. Materials

2.1. Metakaolin ($\cdot Al_2O_3 \cdot 2SiO_2$)

Metakaolin is one of the most reactive pozzolans produced by the calcination of kaolin, kaolinitic clays, shales and other suitable raw materials containing the mineral kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$). The raw materials are calcinated at temperatures of 600 – 900°C. At optimum activation temperatures between 725°C and 850°C, the dehydroxylation of the kaolinite occurs, forming a highly reactive amorphous structure of pentacoordinated atoms Al(V). At the same time, the temperatures are still low enough to form the crystalline phase. At temperatures above 900°C, the formation of mullite ($3Al_2O_3 \cdot 2SiO_2$) and cristobalite (SiO_2) occurs and causes a decrease of pozzolanic activity. The burning is followed by grinding to increase the specific surface of metakaolin [6]. Metakaolin reacts with $Ca(OH)_2$ to form the C-S-H and C-A-H phases of C_4AH_{13} , C_2ASH and C_3AH_6 [7-11].

The metakaolin used here, commercially sold as Mefisto K05, is one of the products by the company České Lupkové Závody a.s.

2.2. Calcium hydrate ($Ca(OH)_2$)

Calcium hydrate CL 90-S was used in this experiment. It is a white, slaked lime which is used to produce mortars, plasters and stucco. According to the standard EN 459 – 1, the CaO + MgO content must be at least 90%. The apparent density of the lime hydrate is $425 \text{ kg} \cdot \text{m}^{-3}$.

2.3. Gypsum ($CaSO_4 \cdot 1/2 H_2O$)

In addition to the above, normally hardening building gypsum was used.

2.4. Filler

Natural non-sorted aggregate from Bratčice with the fraction 0 – 4 mm was used as the filler. In the experiment, a grain fraction of 0 – 2 mm was used, so the sand was sifted through a 2 mm-mesh standard sieve and only the sieved part was used.

3. Methods and data

The study was carried out in two stages. The first stage was based on the modification of reference mortar mixture that was developed in the previous research conducted at the Institute of technology of building materials and components FCE VUT in Brno. This reference mixture was modified by the addition of plaster regarding its influence on basic technological properties of the material. In the second stage, the hydration process of the binder stored in various conditions was monitored.

3.1. Stage I

Mix design of raw material powder and amount of mixing water. In the first stage, the plaster was added to the standard mixture to substitute 5 or 10% of the dry mortar mix (including fine aggregate). The amount of mixing water was carefully determined so that all samples exhibited a constant consistency ($175 \pm 10 \text{ mm}$) corresponding to determination of consistence of fresh mortar (by flow table) according to CSN EN 1015 – 3. The value is characterised by the water coefficient w (ratio of the amount of water to the dry mortar mix). The mix composition of the historic mortars is presented in table 1.

The bulk density of fresh mortar. The fresh mortar bulk density test was carried out according to CSN EN 1015 – 6.

The bulk density of hardened mortar, compressive strength and flexural strength. The fresh mortar was poured into moulds to form prismatic specimens of $40 \times 40 \times 160 \text{ mm}$. After 24 hours, the samples were demoulded and stored in the laboratory conditions. The bulk density, compressive strength and flexural strength of hardened mortars were analysed after 7 and 28 days.

Table 1. Mix design for historic mortars.

	S0	S5	S10
Fine aggregate 0 – 2 mm Bratčice (%w)	82	77.9	73.8
Metakaolin Mefisto K05 (%w)	10	9.5	9
Lime hydrate (%w)	8	7.6	7.2
Gypsum (%w)	0	5	10
Water ratio (water/dry mortar mix)	0.26	0.24	0.23

3.2. Stage II

The entry raw materials that form the mortar (lime hydrate, metakaolin and gypsum) were mixed according to proposed recipes from the Stage I. The materials were homogenised and mixed with water to form a paste of standard consistency (according to Vicat). After 24 hours we made test microsamples of all mixtures and stored them in various moisturising environments. These environments simulated the effects of atmospheric exposure on the plaster (humidity, low and higher temperatures, etc.):

- L/20: Exposure in the laboratory environment,
- C/5: Exposure in water saturated with CO₂ at 5°C,
- C/40: Exposure in water saturated with CO₂ at 40°C,
- V/5: Storage in water at 5°C,
- V/40: Storage in water at 40°C.

From each of the environments described above, sampling was performed after one, three, seven, 14, 21, and 28 days. To prevent any further hydration, the samples were washed several times in isopropyl alcohol, then dried and milled. The phase composition of all samples was determined using X-ray diffraction analysis.

4. Results and discussion

4.1. Stage I

It is clear from the results that, in order to ensure a uniform mortar consistency, it is necessary to use the largest quantity of mixing water in the reference mixture S0 (i.e., the mixture without the addition of gypsum). Furthermore, it can be assumed that the higher value of the water ratio in the S0 sample (table 1) results in lower values in the bulk density of the fresh mortar and in lower strength values (table 2).

When the gypsum content is increased, bulk density decreases after seven days. This is as a result of the more intensive drying process used to remove the mixing water. After 28 days, the bulk densities were virtually equal.

The compressive strengths and flexural tensile strengths of samples containing gypsum were already quite high after only seven days of hydration. After 28 of hydration, the strength increase in these samples is not so significant. The opposite phenomenon can be observed in the reference sample S0 (which is without gypsum content). The high initial strengths of samples containing gypsum can be attributed to the hydration of calcium sulphate hemihydrate to gypsum and, apparently, to a moderate increase in sample densities caused by the formation of primary ettringite. It can be assumed that, in the presence of sufficient water, the strength of the reference sample S0 will grow significantly in the long term. This will be caused by the pozzolanic reactions between metakaolin and lime hydrate during the formation of C-S-H products.

Table 2. The technological properties of fresh and hardened mortar.

	S0	S5	S10
Bulk density of fresh mortar ($\text{kg}\cdot\text{m}^{-3}$)	1,970	1,990	2,010
Bulk density of hardened mortar after 7 days ($\text{kg}\cdot\text{m}^{-3}$)	1,760	1,710	1,670
Bulk density of hardened mortar after 28 days ($\text{kg}\cdot\text{m}^{-3}$)	1,650	1,670	1,660
Compressive strength after 7 days ($\text{N}\cdot\text{mm}^{-2}$)	0.6	6.7	8.6
Compressive strength after 28 days ($\text{N}\cdot\text{mm}^{-2}$)	2.4	8.3	8.8
Flexural strength after 7 days ($\text{N}\cdot\text{mm}^{-2}$)	0.3	1.5	1.6
Flexural strength after 28 days ($\text{N}\cdot\text{mm}^{-2}$)	0.7	1.7	1.9

4.2. Stage II

X-ray diffraction analysis has proven the presence of residues of inert mineral, specifically kaolinite, quartz, and portlandite from lime hydrate. From the new hydration products, the following minerals were identified: gehlenite octahydrate (C_2AH_8), C-A-H phase-type C_2AH_8 , carbonate complex ($\text{C}_3\text{A}\cdot\text{CaCO}_3\cdot 12\text{H}_2\text{O}$), gypsum ($\text{CaSO}_4\cdot 2\text{H}_2\text{O}$), ettringite ($\text{C}_3\text{A}\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$), monosulfate ($\text{C}_3\text{A}\cdot\text{CaSO}_4\cdot 12\text{H}_2\text{O}$) and calcium carbonate (CaCO_3) in the form of vaterite, aragonite and calcite.

In the case of samples with the addition of gypsum (S5, S10), the same minerals formed. Thus, from now on only the results from mixtures without gypsum (S0) and the mixture with the 5% gypsum content (S5) will be reported.

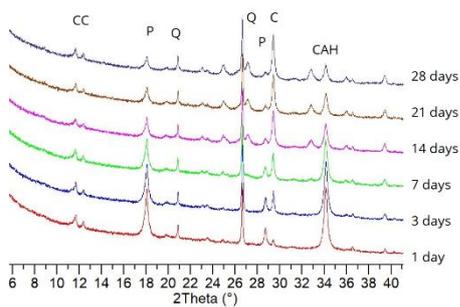


Figure 1. L/20 sample S0 (note: CC – carbonate complex; P – portlandite; Q – quartz; CAH – C-A-H phase; C – calcite).

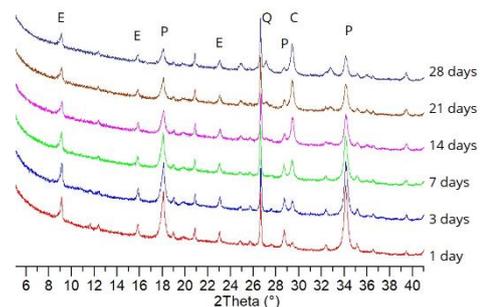


Figure 2. L/20 sample S5 (note: C – calcite; E – ettringite; P – portlandite; Q – quartz).

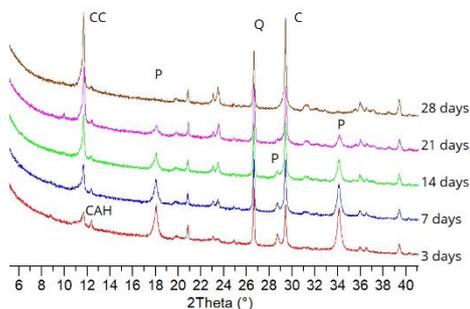


Figure 3. C/5 sample S0 (note: CAH – C-A-H phase; C – calcite; CC – carbonate complex; Q – quartz; P – portlandite).

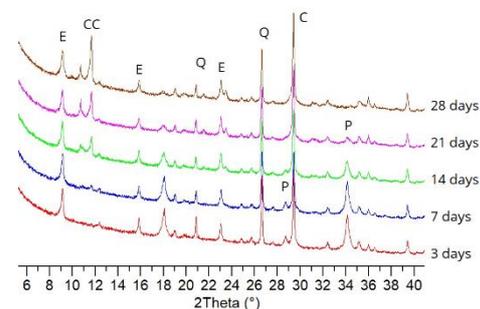


Figure 4. C/5 sample S5 (note: C – calcite; CC – carbonate complex; E – ettringite; P – portlandite; Q – quartz).

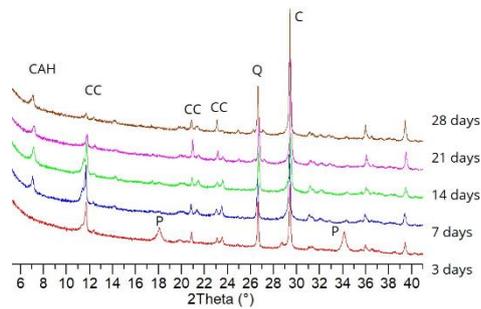


Figure 5. C/40 sample S0 (note: CAH – C-A-H phase; CC – carbonate complex; C – calcite; P – portlandite; Q – quartz).

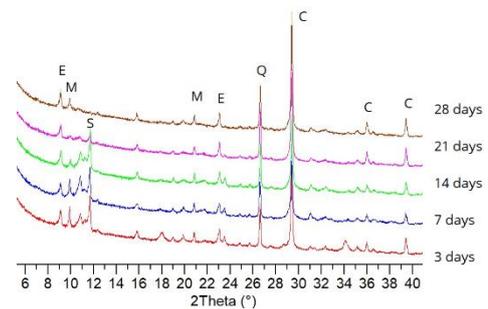


Figure 6. C/40 sample S5 (note: C – calcite; E – ettringite; M – monosulfate; Q – quartz; S – gypsum).

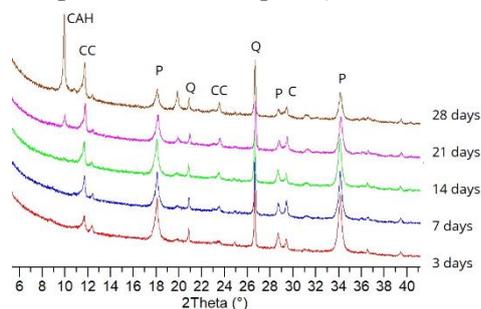


Figure 7. V/5 sample S0 (note.: CAH – C-A-H phase; CC – carbonate complex, C – calcite P – portlandite, Q – quartz).

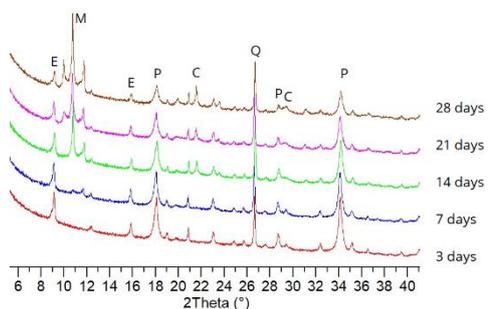


Figure 8. V/5 sample S5 (note: C – calcite; E – ettringite; M – monosulfate; P – portlandite; Q – quartz).

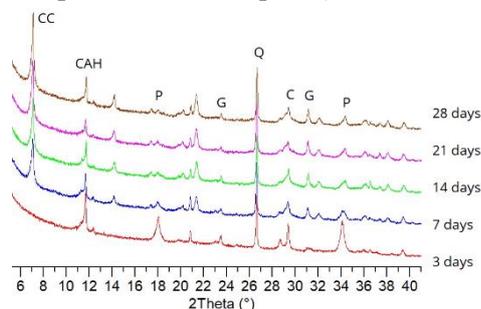


Figure 9. V/40 sample S0 (note: CAH – C-A-H phase; CC – carbonate complex; G – gehlenite octahydrate; P – portlandite;; Q – quartz).

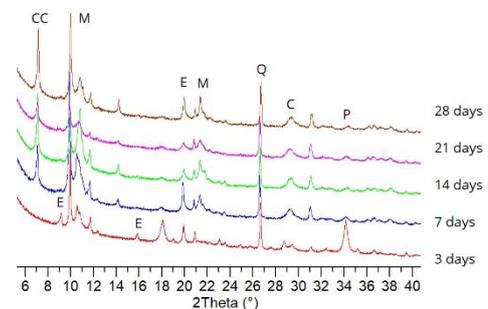


Figure 10. V/40 sample S5 (note: CC – carbonate complex; C – calcite; E – ettringite; M – monosulfate; P – portlandite; Q – quartz).

By following the phase composition in various hydration environments, it has been discovered that, in pure lime-metakaolin mortars, new hydration products from the C-A-H phase are formed (figures 1, 3, 5, 7, 9); these then gradually transform into an AFm phase of the carbonate complex type. This transformation takes place also partly at lower temperatures. Higher temperatures help create gehlenit octahydrate in a water environment (figure 9). When replacing a part of the lime-metakaolin mortar with gypsum, the ettringite is present from the beginning figures 2, 4, 6, 8, 10); this is unstable and transforms into monosulfate at 40 °C (figure 6 and figure 10). In the long run, this process may have a negative effect on the volume constancy and, consequently, on the technological properties of the hardened mortars

5. Conclusion

The aim of the experiment was to research the influence of plaster on some properties of lime-metakaolin mortar intended for the preparation of historic plasters. It was discovered that replacing 5% of the original reference mixture by gypsum positively affects both the compressive and the flexural strength. The compressive strength after 7 days was ten times higher than the compressive strength of the reference sample. The flexural strength after 7 days was five times higher than the flexural strength of the reference sample. This is principally caused by the hydration of calcium sulphate hemihydrate to gypsum and the formation of primary ettringite. However, the presence of sulphates and water in the plaster leads to the formation of ettringite, which can transform to monosulfate at temperatures of 40 °C and above. This transition is characterised by volumetric changes and can, in extreme cases, lead to a defects within the plaster.

6. Acknowledgment

This paper has been worked out with financial support from the budget under the project FAST-S-18-5406, “Study of advanced silicate composites structure with emphasis on the characterization of secondary raw materials influence” and under the project No. LO1408 "AdMaS UP - Advanced Materials, Structures and Technologies", supported by Ministry of Education, Youth and Sports under the “National Sustainability Programme I”.

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