IV.36
High Fired Gypsum Mortar for Screeds, Terrazzo and Masonry Repair on Historic Monuments. Production, Properties and Sample Applications

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Abstract This study presents the production, the composition and the resulting properties of a gypsum mortar for restoration work. It is manufactured in the tradition of the medieval gypsum materials used for masonry construction and sculptures. Application possibilities of the mortars will be discussed on the example of the historical city hall of Lüneburg and the New Museum building in Berlin, ranging from static strengthening of gothic brick masonry to ornamental floor screeds (terrazzo).

1 Introduction

Gypsum has served as a building material in Europe for a long time. As far back as the Roman Empire gypsum has been used as a joint mortar, masonry mortar or as interior and exterior plaster in the vicinity of natural gypsum deposits. Besides its use as building material a great number of sculptures, reliefs, and ornamental screeds were created using gypsum in medieval times. In many studies the binder of the historic gypsum mortars has been identified as high fired gypsum [1-7]. It was particularly valued because of its hardness and resistance to weathering in both outdoor and indoor conditions and differed in its material properties significantly from today’s gypsum materials.

Because of the application of modern hydraulic building materials knowledge of techniques for the production of gypsum mortars became less important in modern times and has therefore vanished.
However, as shown by a wide range of investigations, artificial as well as natural hydraulic components of repair mortars frequently lead to the formation of expansive minerals when in contact with historic gypsum mortars [8]. This has caused dramatic damage to historic objects leading to the loss of whole buildings.

In order to repair and restore historical buildings and ornaments in which gypsum mortars have been used, it is necessary to apply mortars that are not only adjusted to the visual appearance of the historical mortar but also possess compatible material and chemical properties.

Most gypsum mortars available on the building materials market proved not to be stable against environmental influences and are therefore not appropriate. However, there is a need for a durable repair material for restoration projects of historic monuments containing gypsum. At the suggestion of the preservation authorities a small enterprise at Hundisburg in Saxony-Anhalt has started to manufacture gypsum mortars according to traditional production procedures.

2 The Production

The knowledge of the historical firing technique has been reclaimed in the scope of the EU-LEADER project from 2005-2007. The involved test laboratories identified the influence of raw materials, firing conditions, mixing techniques, and after treatment, on the resulting gypsum mortars [9-11].

2.1 Raw material and firing process

For the production, two gypsum raw materials are currently used - one from the Zechstein period deposit in the Harz region (named A3) and one from the Keuper period deposit in Franconia (named Keuper).

The material from these quarries consists almost entirely of gypsum dihydrate. In addition minor constituents (mineral impurities) may be present:

A3: calcite (Fig.1), celestine (SrSO₄), anhydrite, Mg/Si-phases
Keuper: dolomite, dolomitic marl, celestine (SrSO₄), quartz, feldspar
In the past kilns of various types were used for firing gypsum raw material to produce mortars [1, 4]. The material described in the present article is fired in a kiln showing a square ground plan, a replication following one of the historical prototypes. The kiln is fired using wood and consists of a bigger chamber for the gypsum stones and a combustion chamber - separated from each other by a grate. Care is taken to minimize contaminated deposit horizons as raw material. After crushing and separating by size, the gypsum stones are stacked by hand (Fig. 2). The bigger stones (20-25 cm diameter) are located in the lower part of the chamber, and the smaller ones (5-10 cm diameter) are located on the top. A sufficient space between the stones has to be kept for the removal of the flue gases. Tree trunks are placed vertically between the stones for the same purpose. The firing time is 12-13 hours. Monitoring is done by measuring the firing temperature in the middle of the kiln (Fig. 3). Stacking and a continuous firing procedure are essential for the properties of the resulting material.
2.2 Phase content of the fired gypsum

This firing technique results in a mixture of varying degrees of calcined gypsum. The larger gypsum pieces are frequently well burnt in the outer areas, while unprocessed material can still be found in the nucleus. The combination of different mineral phases causes the relatively high initial strength and the typical dense micro fabric of the hardened mortar [1-6].

The mineralogical composition of the calcined gypsum is determined not only by the particular firing conditions but also by the geologically related minor constituents. XRD analysis and microscopic investigations show the following changes of the mineral content in the firing process:

- Gypsum (CaSO\(_4\) \cdot 2\text{H}_2\text{O}) is almost completely dehydrated to anhydrite (CaSO\(_4\)).
- Different types of anhydrite arise; microscopically two morphologies can be distinguished (Fig. 4, left).
  I. Compact to fibrous or strip-shaped anhydrite (called thermal anhydrite)
  II. Granular anhydrite, sometimes showing holes and melting marks (rounded edges, Fig. 4, right).
- With increasing firing temperature the proportion of granular anhydrite rises.
- Activation of Ca from dolomitic limestone or dolomitic marl particles.
- Loss of calcium leads to the formation of Mg-phases (probably brucite, Fig. 5).
- Calcite grains do not show any changes.
- Oxidation of Fe-containing minerals, leads to the typical brownish colour of these mineral grains.
- Non stoichiometric anhydrite or CaO thermally formed from gypsum, as indications for sulphur loss during firing at very high temperatures have not been observed.
3 Properties of high fired gypsum mortar

The high firing temperature, the resulting phase content and the coarse grinding (0 - 4 mm) lead to favourable processing properties. Contrary to other gypsum building materials mortar that has been manufactured by high fired gypsum achieves considerable strength and good durability over a hardening period of several weeks and is suitable for outdoor applications.

3.1 Hardened mortar properties

Through the contact with water a binder matrix of well-linked dihydrate crystals is formed (Fig. 6), including bigger grains of the firing product. Crystal
shape and size are different from those obtained by producing gypsum mortars from a hemi-hydrate (Fig. 7).

The microscopic studies show that the fibrous or strip-shaped anhydrite preferably dissolves and crystallizes as dihydrate. The granular anhydrite remains unchanged. Even a treatment with additional water, which simulates a regular moisture input under weathering conditions, does not lead to a hydration of this type of anhydrite. It follows that this anhydrite is considered insoluble.

On the grains of the firing product the reactive part of the anhydrite will dissolve, sometimes resulting in cavities. The typical fibrous strip-crystal forms of anhydrite are kept pseudomorph in the conversion of dihydrate (Fig. 8); at the same time dense sintering rims along the borders of the firing product grains may form.

In particular, the microscopic examinations enable the origin of structure characteristics typical for historic gypsum mortars to be traced.

The aforementioned water treatment of the hardened mortar under laboratory conditions leads to the increased hydration of the remaining available reactive anhydrite, thereby contributing to the growth of the existing larger dihydrate crystals. The effect of after treatment on the material characteristics can be summarized as follows:

- An increase in the gross density
- Reduction of the true density
- Reduction of the porosity and water absorption
- An increase in the strength and the dynamic modulus of elasticity

**Fig. 6** Left: Typical micro fabric of the hardened mortar [Thin sections in transmitted light, x-pol.]; Right: The matrix is formed by well-linked dihydrate crystals. Some anhydrite crystals (bright grains) and the capillary pore spaces (black) are visible [SEM-BSE-images on thin sections]
Fig. 7 Left: High fired gypsum; Right: Gypsum mortar from hemi-hydrate. Comparing the images, taken under the same imaging conditions (sample preparation, magnification, SEM-detector) it becomes clear that high fired gypsum shows larger and better linked crystals leading to a high strength and good durability [both: SEM-SE-images on fractured surfaces]

Fig. 8 Left: firing product grain with dense sintering rim along the border; Right: Detail from the center of the grain in the left image. Dihydrate pseudo morph to fibrous anhydrite, bright grains are granular anhydrite [both images: SEM-BSE-images on thin sections]

The relevant properties of the mortars manufactured from the raw materials A3 and Keuper are compared in Table 1.
Table 1  Properties of high fired gypsum mortars manufactured at ZIEGELEI Technisches Denkmal Hundisburg e.V., Saxonia-Anhalt, Germany. Analysis: Bremen Institute for Materials Testing (MPA)

<table>
<thead>
<tr>
<th>Storing conditions before testing: 2 days 20°C / 95% RH.; 26 days 20°C / 65% RH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 1 Properties of high fired gypsum mortars manufactured at ZIEGELEI Technisches Denkmal Hundisburg e.V., Saxonia-Anhalt, Germany. Analysis: Bremen Institute for Materials Testing (MPA)</td>
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</tbody>
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<table>
<thead>
<tr>
<th>Properties</th>
<th>A3</th>
<th>Keuper</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh mortar properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water/gypsum ratio</td>
<td>0.30</td>
<td>0.31 – 0.32</td>
</tr>
<tr>
<td>Flow table test</td>
<td>146 mm</td>
<td>145 mm</td>
</tr>
<tr>
<td>Beginning of setting</td>
<td>80 min</td>
<td>68 – 105 min</td>
</tr>
<tr>
<td>End of setting</td>
<td>1545 min</td>
<td>313 – 325 min</td>
</tr>
<tr>
<td>pH-value</td>
<td>11.3</td>
<td>11.1 – 11.2</td>
</tr>
<tr>
<td>Hardened mortar properties (28 d)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gross Density [kg/dm³]</td>
<td>1.71</td>
<td>1.69 – 1.74</td>
</tr>
<tr>
<td>Water absorption [M-%]</td>
<td>12.2</td>
<td>9.9 – 11.9</td>
</tr>
<tr>
<td>Open porosity [Vol-%]</td>
<td>20.8</td>
<td>17.3 – 20.2</td>
</tr>
<tr>
<td>pH-value</td>
<td>10.8</td>
<td>n.d.</td>
</tr>
<tr>
<td>Dynamic E-Modul [kN/mm²]</td>
<td>8.3</td>
<td>12.2</td>
</tr>
<tr>
<td>Flexural strength [N/mm²]</td>
<td>4.2</td>
<td>4.6 – 4.9</td>
</tr>
<tr>
<td>Compressive strength [N/mm²]</td>
<td>17.8</td>
<td>19.7 – 22.3</td>
</tr>
<tr>
<td>Expansion / Shrinkage [mm/m]</td>
<td>n.d.</td>
<td>-0.20 – 0.30</td>
</tr>
</tbody>
</table>

1) determined by DIN 1168-2, chapter 2.5.2.2 respectively 2.5.2.5, footnote 1
2) taken from Richter [10]
3) high pH values probably caused by the fired impurities dolomite and marl

4  Sample applications

In recent years high fired gypsum has been been successfully used in the following traditional applications:

- Screed and Terrazzo
- Mortar for constructing masonry
- Plaster
- Stucco

Typical examples for the use of high fired gypsum in restoration work are shown in Fig. 9. The floor of the church in Eschenbergen was reconstructed based on historical findings. The white areas between the ceramic tiles are renewed screed made of high fired gypsum. On the steeple of a church in Wernigerode consolidation of joint mortars and plasters has been done; again, in accordance with the historical findings high fired gypsum was used. At the church of Kehmstedt the outer walls have been plastered using this material.
4.1 City Hall Lüneburg

The medieval city hall of Lüneburg was built using gypsum as a masonry mortar (Fig. 10). For structural reasons, in the basement of the building several supporting pillars had to be rebuilt [12]. Preliminary studies have shown that a moderate humidity and salt stress in the pillars allowed the use of gypsum mortar [13]. With a view of the compressive strength required, high fired gypsum was chosen not only because it matched the historical material but because it ensured an optimum material compatibility. Due the relevance of static, it was necessary to ensure that the gypsum mortar, even in contact with moisture, achieved the strength required by regulations [13]. Table 2 shows that proof of this could be provided successfully; even in water-saturated conditions and after 7 days stored underwater, the high fired gypsum mortar maintained more than 50% of its strength (Fig. 11). Gypsum mortars based on hemi-hydrate show a much greater loss in strength under these conditions.
Fig. 10 Pillars in the basement of the city hall Lüneburg. Left: Situation before start of repair work. The masonry consists of brick and gypsum mortar. The pillars are safeguarded by steel profiles; Right: Pillars in final state, rebuilt using high fired gypsum mortar.

Fig. 11 Strength of high fired gypsum mortar related to moisture content of the mortar.

Table 2 Requirements and test results of high fired gypsum mortar in water-saturated conditions. Analysis: Bremen Institute for Materials Testing (MPA)

<table>
<thead>
<tr>
<th>Requirement by standards 1)</th>
<th>Test result</th>
<th>Assessment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength [N/mm²] ≥ 5</td>
<td>11.8 ± 1.0</td>
<td>passed</td>
</tr>
<tr>
<td>Compressive strength in joint [N/mm²] ≥ 5 (method II)</td>
<td>16.6 ± 2.2</td>
<td>passed</td>
</tr>
<tr>
<td>Bond shear strength [N/mm²] ≥ 0.2</td>
<td>0.4 ± 0.2</td>
<td>passed</td>
</tr>
</tbody>
</table>

1) standards: DIN 1053-1; DIN EN 998-2; DIN V 18580; DIN V 20000-412
4.2 New Museum Berlin

The building of the new museum in Berlin was heavily damaged by the effects of WWII. Since the 1990s, extensive restoration work has been carried out [14]. The splendid terrazzo floors were reconstructed according to historical findings using high fired gypsum from Hundisburg (Fig. 12). For the very elaborate production of the floors in the New Museum, pigments and crushed stone were added as decorative elements, and the floor surface was ground in several steps, polished and sealed.

![Fig. 12 New Museum in Berlin: Overview and detail of the terrazzo floors made using high fired gypsum as a binder material (photos: Kaiser)](image)

5 Conclusions

The high fired gypsum produced in the building material factory TECHNISCHES DENKMAL HUNDISBURG e.V. is a material comparable to historic gypsum, in regards to the raw materials, firing process, grinding technology and properties. The raw materials are natural gypsum rocks that are processed with the typical variation in their composition. It is ensured that minimum contaminated deposit horizons are used and that production and application are free of chemical additives.

The mortar is characterised by a sufficient long processing time, high strength, and an optimal microstructure. According to the historical findings the material can be modified by the addition of pigments and coloured sands or stones.

Various surface treatments such as compaction by beating, grinding, polishing and smoothing to a shine are possible. The high fired gypsum can be used in all traditional applications of gypsum mortar.

The material has been extensively studied scientifically in recent years and is subjected to regular production control.
6 Acknowledgements

The presented research about the development and use of high fired gypsum was possible thanks to funding by the EU LEADER project. We also thank the preservation authorities of Saxony-Anhalt for the suggestions on this research and their support.

7 References