Influence of fine aggregate on some properties of gypsum mortars

M Doleželová¹, J Krejsová¹ and A Vimmrová¹

¹ Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova 7, 166 29 Prague, Czech Republic

Email: magdalena.dolezelova@fsv.cvut.cz

Abstract. The importance of gypsum as a building material increases nowadays because of its environmental friendliness, excellent fire properties and good workability. Since the gypsum is mostly used in the form of the paste, the influence of aggregates on properties of gypsum mortars is not investigated very often. The article deals with some basic and mechanical properties of gypsum mortars with different aggregates and compares them with the properties of gypsum paste. Type of the aggregates influences the properties of gypsum mortars significantly. The presence of aggregate does not worsen the adhesive strength of the gypsum material in comparison with gypsum paste and the adhesive strength was even improved for some type of aggregates.

1. Introduction

The research of gypsum increases recently, because it can be produced from secondary materials and also because it has very low temperature of calcination (in comparison with the lime or cement production). The gypsum-based pastes were studied mostly and the research of gypsum mortars (e.g. materials with aggregates) is at the beginning nowadays. Gypsum mortars are used to the lesser extent than gypsum pastes, since the presence of aggregates in gypsum is not necessary from the point of view of shrinkage (gypsum does not shrink during setting), however aggregates are used in gypsum mainly for economic reasons. The use of aggregates in gypsum is mostly based on the historical experience or on the knowledge obtained from the study of other binders, especially of cement [1], but the real behaviour of gypsum mortars with common aggregates was not studied sufficiently yet. The authors studied mostly the possibility of waste products utilization [2, 3]. The adhesive strength is determined mostly for the commercial type of mortars [4]. The ready-mixed clay plasters were studied in [5]. Authors found that the procedure, described in EN 1015-12 is not suitable for this type of plasters. There was found only one paper, dealing with the adhesive strength of gypsum mortars [6]. In this study, the ladle furnace slag was used as filler and it was stated, that the adhesive strength decreased with increasing the amount of slag. The goal of the article was to describe the influence of different types of common siliceous aggregates on selected mechanical properties. The four aggregates of different origin, containing different types of particles were chosen for the design of gypsum mortars and the compressive, tensile and adhesive strength of mortars were tested. The results were compared with the properties of pure gypsum paste and they are discussed in this article. The research was performed in order to obtain some basic knowledge about the behaviour of gypsum composites, because it was found, that while the influence of aggregates on the properties and behaviour of cement-based materials was studied thoroughly [7, 8], no such research was performed for gypsum-based materials yet.
2. Used materials
Four gypsum mortars and one gypsum paste as a reference were tested. Gypsum (GYPSTREND Ltd.), four types of siliceous aggregates with different origin and particle shape and citric acid were used as dry components of the mixtures. The used gypsum was the common commercial product. The citric acid was used as the setting retarder. The amount of citric acid depends to the amount of gypsum. The specification of sands is in the Table 1. The grain surface roughness of all sands was determined by the confocal laser scanning microscopy (CLSM) as the three dimensional arithmetical mean roughness SRa [μm] [9].

| Sand - S2 | Sand - S3 | Sand - S4 | Sand - S5 |
|-----------|-----------|-----------|-----------|
| Producer  | Filtrační písky Ltd. | Sklopísek Strleč Inc. | KAMEN Zbraslav Inc. | TAPAS BOREK Ltd. |
| Origin    | Quarried | Quarried | Natural crushed | River bed, uncrushed |
| Surface   | smooth   | rather smooth | rough | smooth |
| SRa of grain surface [μm] | 0.235 | 0.361 | 0.771 | 0.215 |
| Remark    | Standardized sand (CEN, ČSN EN 196-1) | SiO₂ > 99 % | - | - |

Table 1. Materials specification.

2.1. Designation of mixtures
The final compositions of mixtures are in the Table 2. The components of mixtures were dosed by volume. The binder to sand ratio was 1:2. The amount of gypsum was the same for all mixtures. The amount of sand was calculated according to its bulk density to achieve the same volume of sand in each mortar. The volume of standardized sand was chosen as a default value. The detailed description of mixtures design was described in the previous article of authors [10].

Table 2. Mixture compositions.

| Mixture designattion | Fine aggregate (%) | Gypsum (%) | Citric acid (%) | Water/binder ratio (+) |
|----------------------|--------------------|------------|-----------------|------------------------|
| G                    | -                  | 99.92      | 0.08            | 0.45                   |
| GS2                  | 66.65              | 33.32      | 0.03            | 0.53                   |
| GS3                  | 65.3               | 34.67      | 0.03            | 0.53                   |
| GS4                  | 67.15              | 32.82      | 0.03            | 0.53                   |
| GS5                  | 66.92              | 33.05      | 0.03            | 0.53                   |

3. Measuring methods

3.1. Compressive and tensile strength
Production of samples for strength measuring was managed according to ČSN EN 13279-2 [11]. One set of test specimens was made from each mixture. Each test set contained three prisms 160 × 40 × 40 mm. Samples were stored in laboratory for 7 days and then they were dried at 50°C to constant weight before the testing. Tensile strength ft [MPa] was determined by three-point flexural test by mechanical press FP 100 (VEB Industriewerk Ravenstein) and compressive strength fc [MPa] was tested after the bending test. The strength tests were also performed according to standard ČSN EN 13279-2.

3.2. Adhesive strength
Adhesive strength tests were carried out according to ČSN EN 1015-12 [12]. Aerated autoclaved concrete blocks were used as a substrate. They were wetted and manually coated by mortars/plasters according to the conventional construction techniques. Five circles were cut into the fresh mortar/plaster to form circular test areas of 50 mm in diameter (Figure 1). The day before testing, circular pull-head plates made of stainless steel were glued to the circular test areas with two-component epoxy resin.
Adhesive strength tests were carried out 7 days after plastering using a measuring device COMING plus a.s. Tensile load was applied to the circular test area through the pull-head plate. The measuring assembly is shown in Figure 2. Failure load $F_u$ [N] was recorded and fracture pattern was described. Adhesive strength $f_u$ [N·mm$^{-2}$] was calculated as:

$$f_u = \frac{F_u}{A} = A^{-1}$$

where $A$ [mm$^2$] is circular test area.

Five values of adhesive strength were calculated for each mixture and evaluated using DDtest (Dean - Dixon test). Each fracture pattern was described as one of the following types or their combination:

a) adhesion fracture at the interface between mortar/plaster and substrate,
b) cohesion fracture in the mortar/plaster itself,
c) cohesion fracture in the substrate.

In the case of fracture patterns b) or c), calculated value of adhesive strength could be considered as lower value than the real value of adhesive strength.

4. Results and discussion

The bulk density of all gypsum mortars is around 1780 kg·m$^{-3}$. The bulk density of gypsum paste is lower (1311 kg·m$^{-3}$), because it does not contain any aggregates. The compressive strength and tensile strength can be seen in Figure 3. The samples with roughest particles (i.e. with natural crushed sand GS4) have the highest strength of all materials (22.2 MPa). The strength of gypsum paste is 18 MPa. The strength of all samples with the smooth particles (GS2, GS3, GS5) is lower than the strength of gypsum paste G. The lowest value has the mortar with standardized sand (GS2), which has the smoothest surface of particles from all tested aggregates. It is evident, that the quality of the surface aggregates plays similar role in gypsum as in the cement-based materials [13]. While the aggregate with smooth particles affects the strength of mortars negatively, the aggregates with rough particles cause the increase of the mortars strength.
Figure 3. Mechanical properties [MPa].

Table 3. Adhesive strength at the age of 7 days.

| Mixture designation | G    | GS2   | GS3   | GS4    | GS5    |
|---------------------|------|-------|-------|--------|--------|
| Adhesive strength [MPa] | 0.29 ± 0.03 | 0.3 ± 0.2 | 0.5 ± 0.1 | 0.50 ± 0.05 | 0.3 ± 0.1 |

The values of adhesive strength (with the standard deviation) are in Table 3. The adhesive strength was measured five times for each mortar/paste. No measuring had to be eliminated for the fracture between mortar/paste and the circular pull-head plates. The results of adhesive strength are in Table 3 and the evaluation of fracture pattern is shown in Table 4. The real value adhesive strength could be obtained only when the type a) of the fracture occurs (i.e. at the interface between mortar/plaster and substrate). This type of fracture pattern predominated for the gypsum mortars. The samples without the sand (G) were mostly fractured in the paste (type b) itself, so the tensile strength of paste was lower than its adhesive strength.

The area of fracture in the mortar (type b) was in the range from 14% to 36% for mortars. Fracture in the substrate (type c) occurred only rarely. The area of fracture c) was maximally 12%. It can be supposed that the measured values of samples GS2, GS3 are very close to the real adhesive strength, because the fractures were predominantly in the interface between gypsum mortars and substrate. The adhesive strength of mortar with crushed sand (GS4) could be mildly higher in reality, considering the type of fracture. The samples of gypsum paste without sand (G) account the biggest inaccuracy. The paste has lowest measured adhesive strength (0.290 MPa), but it could be considered as a minimal value and the real value of adhesive strength of gypsum paste is probably higher.

The material with the roughest aggregates (GS4) has the highest adhesive strength, similarly as the compressive and tensile strength, while the mortars with smoothest particles (GS2, GS5) have the calculated value of adhesive strength nearly the same as gypsum paste. But because the real adhesive strength of gypsum paste is probably higher than calculated value, the real adhesive strength of gypsum mortars with smooth particles is in reality lower than adhesive strength of gypsum paste. The higher adhesive strength of mortar GS3 can be attributed to the higher amount of fine particles in this type of sand, which was confirmed by the laser diffraction analysis [9]. The conclusion could be drawn, that the presence of smooth aggregates slightly worsens the adhesive strength of gypsum material, but utilization of aggregates with rough particles could improve it.
Table 4. The evaluation of fracture pattern.

| Fracture pattern             | G  | GS2 | GS3 | GS4 | GS5 |
|------------------------------|----|-----|-----|-----|-----|
| at the interface between plaster and substrate | a) | 18% | 80% | 72% | 56% | 67% |
| in the plaster               | b) | 82% | 14% | 16% | 36% | 27% |
| in the substrate             | c) | 0%  | 6%  | 12% | 8%  | 6%  |

5. Conclusions
The influence of the surface quality of the aggregate particles on some mechanical properties of gypsum mortars was investigated. Four gypsum mortars with different types of sand and pure gypsum paste were tested and compared. The results confirm that the quality of aggregate surface affects the mechanical properties of gypsum materials in similar way as cement materials. The aggregate with smooth particles causes the decrease of mechanical properties of mortars and on the contrary, the strength of mortar with rough particles is higher than the strength of gypsum paste. The values of adhesive strength were evaluated considering the type of fracture. The gypsum paste has the lowest value of the adhesive strength, but the result does not express the real value accurately, because the fracture occurred mostly in the paste, while the values of adhesive strength of gypsum mortars with smooth particles corresponds with reality. Also the real value of adhesive strength of mortar with rough particles is higher than the measured value. Therefore it can be said, that the presence of smooth particles worsens the adhesive strength of gypsum material while the rough particles improves it. These results are in accordance with the conclusions, obtained for cement-based materials.

Acknowledgement: This research was supported by the Czech Science Foundation, Project No. 16-01438S and by the project SGS16/199/OKH1/3T/11.

References
[1] Scrivener K, Crumbie A and Laugesen P 2004 Interface Sci. 12 pp 411–21
[2] Jiménez Rivero A, De Guzmán Báez A and Navarro J G 2014 Constr. Build. Mater. 55 pp 146–52
[3] Adamopoulos S, Foti D, Voulgaridis E and Passialis C 2015 BioResources 10 pp 5563–72
[4] Feria P, Santos T and Aubert J E 2015 J. Mater. Civ. Eng. 28 (1)
[5] Delinière R, Aubert J, Rojat F and Gasc-Barbier M 2014 Build. Environ. 80 pp 11–7
[6] Rodriguez A, Gutiérrez-González S, Horngies M and Calderón V 2013 Mater. Des. 52 pp 987–94
[7] Ollivier J, Maso J and Bourdette B 1995 Adv. Cem. Based Mater. 2 pp 30–38
[8] Tasong W, Lynsdale C and Cripps J 1998 Cem. Concr. Res. 28 pp 1453–1465
[9] Krejsová J, Doleželová M and Vimmrová A 2017 Key Eng. Mater. 760 pp 245–50
[10] Doleželová M, Krejsová J and Vimmrová A 2016 Key Eng. Mater. 722 pp 337–42
[11] ČSN EN 13279-2:2017 Gypsum binders and gypsum plasters. Test methods (Prague: UNMZ)
[12] ČSN EN 1015-12:2000 Methods of test for mortar for masonry - Part 12: Determination of adhesive strength of hardened rendering and plastering mortars on substrates Test methods (Prague: UNMZ)
[13] Mindess S, Francis J and Darwin D 2003 Concrete Prentice Hall (Upper Saddle River, NJ)