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The influence of different aggregates on the behavior and properties of gypsum mortars

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ABSTRACT

The structure, behavior and properties of gypsum mortars with different types of fine aggregates were studied. Investigation by scanning electron microscopy shows that the presence of aggregate particles significantly influences the shape and size of gypsum crystals in the interfacial transition zone. The porosity of the gypsum matrix in mortars is greater than the porosity of pure gypsum paste and the pore size distribution shifts towards smaller pores. The shape and size of crystals in the transition zone depends on the surface roughness of the aggregate particles. The grain surface roughness was measured by confocal laser scanning microscopy. The properties of mortars also depend on the grain surface roughness. The workability, setting times and thermal conductivity decrease with increasing grain surface roughness, while the strength and water vapor resistance increase.

1. Introduction

Although the significance and use of gypsum, as one of the most environmentally friendly binders, is currently increasing, the influence of aggregates on the behavior and properties of gypsum-based materials has not been studied adequately. The presence of aggregates in cement-based materials is necessary because they provide better dimensional stability and wear resistance [1]. Gypsum does not shrink during setting; to the contrary it expands slightly [2] and therefore gypsum could also be used, unlike cement, in the form of a pure paste. Nevertheless, aggregates are often used in gypsum, mainly to improve workability and for economic reasons, and they could also improve the behavior of gypsum based materials in fires [3].

The use of aggregates in gypsum is mostly based on historical experience, on the knowledge obtained from the study of other binders and recently also on attempts to utilize waste products [4–6] or to improve the thermal insulating properties of the gypsum material [7].

The influence of the aggregate on the properties of cement-based mortars and concretes was thoroughly studied in recent decades and it is generally accepted that the size, shape and texture of the aggregate particles influence the properties of the fresh mixtures and hardened material. The properties of the aggregates affect e.g. the workability of the fresh mixture, the mechanical properties and the durability of the hardened mortars or concretes [1]. The interfacial transition zone (ITZ) between the aggregate particles and cement paste has been described and investigated [8–10]. The interfacial transition zone usually has greater porosity than the bulk paste [11,12] and its properties and thickness depend on the composition of the aggregate particles and their physical properties [13,14]. Recently mostly the influence of different types of recycled aggregates on the cement-based materials [15,16] or the influence of standard aggregates on the advanced cementitious materials (e.g. high performance concretes [17,18] or engineered cementitious composites [19,20]) is studied.

However, no information was found about the influence of commonly used aggregates on gypsum-based materials. It is generally assumed that the role of aggregates in gypsum is similar to their role in cement-based materials; however, gypsum is chemically a different material and the processes governing its setting and hardening differ significantly from the processes in cement or lime. Therefore, it seemed useful to carry out a more detailed study of the influence of different types of natural aggregates on the properties and behavior of gypsum mortars.

2. Experimental

2.1. Materials

The gypsum binder employed was a commercial product (Gypstrend ltd., Czech Republic), produced from a mixture of natural gypsum and titano-gypsum. It consists mostly of calcium sulfate hemihydrate

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Table 1
Chemical composition of gypsum.

Oxide	SiO_2	CaO	Al_2O_3	Fe_2O_3	SO_3	K_2O	MgO	TiO_2	MnO	P_2O_5
wt %	6.93	41.81	2.50	0.96	45.50	0.42	0.70	0.30	0.03	0.03

Table 2
Mineral composition of gypsum.

Mineral	bassanite	anhydrite	calcite	quartz	muscovite
	$CaSO_4{\cdot}1/2H_2O$	$CaSO_4$	$CaCO_3$	SiO_2	$KAl_2(AlSi_3O_{10})\ (OH)_2$
wt %	85.6	3.6	0.9	3.4	6.5

(basanite). The chemical and mineral compositions of the employed gypsum (obtained by XRF and XRD analysis) are listed in Table 1 and Table 2, respectively.

Four different types of fine aggregates were used as fillers. The aggregates were chosen according to their different origin and shape. CEN standard sand according to EN 196–1 [21] was used as a reference material. Another three types of aggregate consisted of uncrushed sand from an open sand pit, crushed rock aggregate and uncrushed river sand. The origin, appearance and mineral composition of all the aggregates are summarized in Table 3.

Because of the short setting times of the gypsum samples, citric acid monohydrate (Inchema Ltd., Czech Republic) was added as a setting retarder.

2.2. Experimental methods

The chemical composition of the raw materials was evaluated by X-ray fluorescence analysis (XRF) using a Spectroscan MAKC GVII (Spectron Optel, Russia) apparatus. The **mineral composition** of the raw materials was characterized by X-ray diffraction (XRD) analysis using a PANalytical X'PertPRO diffractometer with $Cu_{K\alpha}$ radiation source. The data were evaluated by the HighScorePlus software package (version 3.0.5) and JCPDS PDF2 database.

The microstructural morphology of the gypsum mortars, gypsum paste and surfaces of the aggregate grains 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 thin layer of carbon, which acted as a conductor. The samples were not polished.

2.2.1. Testing of aggregates

The **bulk density** ρ_v [kg/m^3] of the aggregates was determined according to Czech technical standard ČSN 72 1171 [22] by measuring the volume of a known mass of aggregate sample in a graduated cylinder containing water. The bulk density was calculated according Eq. (1), where m [kg] is the mass of the dry aggregate and V [m^3] is the volume of the aggregate. Because the aggregates are not porous, the value of bulk density is equal to the value of the **specific mass** ρ [kg/m^3].

$$\rho_v = m/V. \tag{1}$$

Sieve analysis. The particle size distribution was determined according to EN 933–2 [23]. Standard sieves with apertures of 0.063 mm, 0.09 mm, 0.125 mm, 0.25 mm, 0.5 mm, 1.0 mm, 2.0 mm and 4.0 mm were used.

The **fine particles distribution** was determined by the Analysette 22 MicroTec laser diffraction analyzer (Fritsch GmbH, Germany).

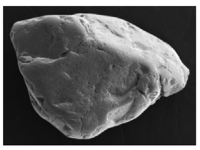
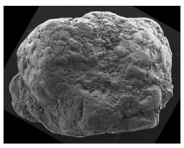
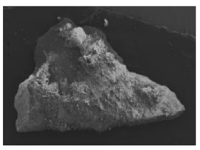
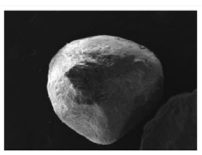
The **grain surface roughness** was determined by confocal laser scanning microscopy (CLSM) as the **three-dimensional arithmetical mean roughness** value *SRa* [μm]. The three dimensional roughness is an analogy to the arithmetical mean deviation of the profile *Ra* (two dimensional roughness) according to ISO 468 [24]. Geometrically, the three dimensional roughness is an indication of the average height of the roughness. In this paper, *SRa* can be defined as the height of the prism, whose base is equal to the field of view and whose volume is equal to the volume of the body bordered by the roughness and middle plane. The employed software automatically modifies the scanned surface before it performs the calculation – the tilt is taken into account and waviness is filtered off according to the set limiting wavelength. *SRa* is usually used for surface roughness quality evaluation of precise engineering products (made by CNC machines etc.). *SRa* can also be used for mortars and concrete surface evaluation (e.g., for mold release agent evaluation [25]). The *SRa* value of the typical grain surface of each aggregate was determined according Eq. (2).

$$SRa = \frac{1}{l_1 l_2}\int_0^{l_2}\int_0^{l_1} |f(x, y)|dxdy, \tag{2}$$

where f (x,y) is height of the assessed profile and l_1, l_2 are measured lengths.

Confocal laser scanning microscopy LEXT OLS3000 (with resolution 0.12 μm) was used. Specifically, an objective with 10× magnification (the magnification on the monitor was 1150×), working distance

Table 3
Aggregate types.

Aggregate	A1	A2	A3	A4
Origin	open pit	open pit	rock quarry	river bed
Processing	mined, uncrushed	mined, uncrushed	quarried, crushed	mined, uncrushed
Remark	CEN standard sand (EN 196–1)			
Appearance				
Typical grain				
Mineral composition	Q	Q	Q, A, MC, C, M, CC	Q, A, MC, M

Q: quartz (SiO_2), A: albite ($NaAlSi_3O_8$), MC: microcline ($KAlSi_3O_8$), C: calcite ($CaCO_3$), CC: clinochlore ($Mg_5Al(AlSi_3O_{10})\ (OH)_8$), M: muscovite $(KF)_2(Al_2O_3)_3(SiO_2)6(H_2O)$