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The Influence of Mineralogical Composition Changes of Sandstone Cement on Physical-Mechanical Properties

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Abstract. The aim of the contribution is to present the results of research focused on cement mineralogical composition changes and their influence on physical-mechanical properties of sandstones. Three types of Czech sandstones were tested during this experiment – Hořice, Kocbeře and Božanov. The sandstone samples were treated in the climatic chamber in order to simulate weathering processes that are typical for winter period in Prague. The influence of road salts was also taken into consideration. For the purposes of mineralogical changes determination the sandstone cement was separated and subsequently analyzed using X-Ray diffraction a DTA/TG analysis. The physical-mechanical properties such as e.g. uniaxial compressive strength, water absorption and open porosity were determined before and after the climatic treatment to enable evaluation of the influence of weathering processes.

Introduction

Sandstones are consolidated clastic sedimentary stones which are formed of mineral grains (mostly quartz) and rock fragments. Cement with different character - e.g. calcareous, clayey, and ferruginous - binds these components. The action of weathering processes leads to the deterioration of sandstones. The weathering can lead to qualitative and also the quantitative changes of cement which can contribute to the negative influence on the original physical and mechanical properties of these stones.

The chemical weathering is influenced by the presence of water and dissolved chemical substances and gasses. Water may penetrate into the rock by different ways and during different conditions. The main influence on the rate of chemical weathering has temperature, humidity, presence of organic acids and dissolved carbon dioxide.

Subject and methods

Experimental material and procedure

Three types of cretaceous sandstone of Hořice, Božanov and Kocbeře, which have often been used in the Czech Republic as a building and sculpture material, were tested during this experiment. The cube samples (5 x 5 x 5 cm) were treated by an exposition in a climatic chamber. Based on the meteorological data measured in Prague during winter periods in years 1999 - 2008, the weathering simulation program was developed to simulate Prague winter climatic conditions [1]. The samples were treated by 56 freeze/thaw cycles in the temperature range from – 14°C to 14°C. The sandstone samples were soaked in distilled water and 2,5% NaCl solution for 24 hours before the beginning of the frost treatment and also after every seven freeze/thaw cycles. To determine the changes of cement mineralogical composition the methods of X-Ray diffraction and DTA/TG analysis were used before and after the climatic treatment. The level of deterioration process was evaluated using the physical-mechanical parameters such as uniaxial compressive strength, water absorption and open porosity measured before and after the climatic treatment.

Determination of mineralogical composition of cement

The methods of differential thermal analysis (DTA), thermogravimetric analysis (TG) and X-Ray diffraction were used for the purposes of the phases analysis of sandstone cement and matrix and of the changes initiated.

Sandstone fine-grained binding material was separated according to the procedure described by Ip *et al.* [2]. The cement was analysed before and after the climatic treatment.

The monitoring of sample weight changes during its heating is the main principle of thermogravimetric analysis. The dependence of weight change on the temperature is illustrated by the thermogravimetric curve, which provides the information on the composition of original sample, on his heat stability or heat disintegration but also on products of decay. The curve can be quantitatively evaluated in terms of stoichiometric calculation [3].

Differential thermal analysis (DTA) is a dynamic heat analysis in which the temperature effects of sample is monitoring regarding its physical or chemical changes during its continuous linear heating or cooling. The temperature differences between studied and reference (indifferent) sample are measured. These differences form during the simultaneous heating which is a linear time function. It is possible to detect every physical or chemical change of matter which is accompanied by the enthalpy which is reflected by endothermic or exothermic reaction [3].

The main principle of X-Ray powder diffraction is the utilization of the diffraction of monochromatic X-Ray after its impact on sample crystal structure based on the Bragg law. The formed diffraction record of studied sample is compared with diffraction records of minerals in databases to determine its phase composition. The analytical software X'Pert HighScore Plus (qualitative phase analysis) and the application MAUD (general diffraction/reflectivity analysis program mainly based on the Rietveld method) were used for the evaluation of gained diffractograms.

Determination of physical-mechanical properties

The assessment of basic physical-mechanical properties was done for the purposes of evaluation of studied material durability and rate of degradation. The samples were tested before and after the climatic treatment. The physical-mechanical properties were determined in accordance with the Czech national standard test methods.

Determination of uniaxial compressive strength was done following the procedure described in ČSN EN 1926 [4]. There were tested sets of ten experimental samples of size 5 x 5 x 5 cm. Water absorption was testing according to ČSN EN 13755 [5] and open porosity according to ČSN EN 1936 [6]. There were tested sets of six experimental samples of size 5 x 5 x 5 cm by both determinations.

Results

Mineralogical composition

DTA/TG analysis

Hořice sandstone

Two mineralogical phases – *kaolinite* and *goethite* - were determined in the samples of cement and matrix of Hořice sandstone using the method DTA/TG. These minerals were determined in all samples of this sandstone (also after the climatic treatment).

Kocbeře sandstone

The presence of *kaolinite* or *halloysite* (eventually mixture of both minerals) was determined in the samples of Kocbeře sandstone using the DTA/TG analysis. It is necessary to clarify the exact mineral identification using another analytical method. The presence of *quartz* was also determined. These mineralogical phases were determined in all samples of this sandstone (also after the climatic treatment).

Božanov sandstone

There was detected a phyllosilicate composed of tetrahedral and octahedral nets with mica sheets in the samples of Božanov sandstone, most likely from a *smectite group*. It is necessary to clarify the exact mineral identification using another analytical method. This mineral was determined in all samples of Božanov sandstone (also after the climatic treatment).

X-Ray powder diffraction

The sandstone samples were analysed using X-Ray powder diffraction before and after 56 freeze/thaw cycles in combination with distilled water and with solution NaCl as well. The summary of qualitative phase analysis of powder cement and matrix samples is given in following table (Table 1). The same mineralogical phases were identified in every sandstone sample of the same petrographical group before and also after the climatic treatment using the distilled water and also NaCl solution.

Table 1. Summary of qualitative phase analysis of powder cement and matrix samples (K – Kocbeře sandstone, H – Hořice sandstone, B – Božanov sandstone).

Group	Mineralogical Phase
K	*quartz* SiO_2
	kaolinite $Al_2Si_2O_5(OH)_4$
	muscovite $KAl_2(Si_3Al)O_{10}(OH)_2$
H	*quartz* SiO_2
	kaolinite $Al_2Si_2O_5(OH)_4$
	muscovite $KAl_2(Si_3Al)O_{10}(OH)_2$
	goethite α-FeOOH
	lepidocrocite γ-FeOOH
B	*quartz* SiO_2
	orthoclase $KAlSi_3O_8$

Physical-mechanical properties

Water absorption at atmospheric pressure and the open porosity was determined as arithmetic mean of measured values of six samples and the uniaxial compressive strength was determined as arithmetic mean of measured values of ten samples by all Czech sandstone types. The parameter was determined before and after the climatic treatment in the combination with distilled water and also NaCl. The results are summarized in Table 2.

Table 2. The average values of water absorption at atmospheric pressure [% w.], open porosity [% vol.] and uniaxial compressive strength [MPa], the percentage change (in brackets).

	Hořice sandstone			Božanov sandstone			Kocbeře sandstone		
	Untreated samples	F/T + H_2O	F/T + NaCl	Untreated samples	F/T + H_2O	F/T + NaCl	Untreated samples	F/T + H_2O	F/T + NaCl
Water absorption	7.35	7.19 (- 2.24)	7.04 (- 4.26)	4.57	4.37 (- 4.34)	4.49 (- 1.75)	3.97	3.97 (0.21)	3.20 (-19.51)
Open porosity	14.96	14.62 (- 2.27)	14.24 (- 4.81)	9.96	9.58 (- 3.82)	9.76 (- 2.01)	8.73	8.75 (0.23)	7.08 (-18.90)
Uniaxial compressive strength	51	54.9 (7.65)	44.6 (-12.55)	75.2	73 (- 2.93)	68.7 (- 8.64)	87.6	76.9 (-12.21)	92.4 (5.48)

Conclusion

As it is obvious from previous chapter, the physical-mechanical properties changed after the climatic treatment. The changes were different according the applied mode of treatment, i.e. use of distilled water or the solution of NaCl. All sandstones reacted differently to the same way of treatment.

One way how to explain the changes of physical-mechanical properties after the climatic treatment of these stones was to focus on changes of mineralogical composition of their cement. The distilled water simulated the role of rainwater in the weathering process. This water is called "hungry" for its properties. We supposed that it can cause the changes of mineralogical composition of cement. The same assumption was by the use of NaCl solution. But as it is obvious from the results of mineralogical analysis of cement powder, the change of chemical composition and mineral phases composition as well was not detected.

In conclusion, the changes of cement mineralogical composition after the climatic treatment were not detected. To sum up, the change of cement mineralogical composition does not explain the physical-mechanical changes of studied sandstone samples. The changes of physical-mechanical properties can be explained by the change of internal structure. This assumption should be explain be another tests.

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