Utilization of the waste from the marble industry for application in transport infrastructure: mechanical properties of cement pastes

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Abstract. This article is focused on the mechanical testing of cement-based samples containing a micronized waste marble powder used as replacement of standard binders. Tested materials consisted of cement CEM I 42.5 R (Radotín, Czech Republic) and three different amounts of the marbles (25, 50 and 70 wt. %). Standard bending and compressive tests of the prismatic samples having dimensions equal to 40 × 40 × 160 mm were done in order to reveal an influence of marble amount on flexural and compressive strength, respectively. Moreover, the dynamic modulus of elasticity and dynamic shear modulus were examined and compared after 7 and 28 days of mixture curing.

1. Introduction

Recycled materials are commonly used as an alternative to non-renewable resources. Unfortunately, these materials have significant problems with variability. As a result of this drawback, the usage of these materials is limited only to the fields of transport infrastructures [1]. The utilization of waste or recycled materials have been spelled out throughout the different researches, as showed e.g. in [2, 3]. Unlike the mentioned research works, we focused directly on the usage of waste marble sludge. The sludge is generated during the block cutting or the finishing process of stone materials. Generally, the production of waste may cause a wide range of environmental problems, for example as follows: air pollution, water pollution, visual impacts and loss of flora and fauna [4]. The amount of this waste was estimated for the area of The Shaq El Thoaban (Egypt) to one million tons per year [5] and for area of Estremoz Anticline (Alentejo, Portugal) to 600 thousand tons per year [6]. Both mentioned areas have approximately the same production of marble stone. On the other hand, the technical, operational and management practices in the industry are less developed in area Shaq El Thoaban. Moreover, they have contributed significantly to greater waste generation. Small amount (from 10 to 20 %) of waste generated by marble industry is used in construction. Remaining part is refused and accumulated in mounds of marble waste close to the extraction quarries [7].

The marble sludge is the most interesting part of the waste because of its almost nil usage. There are several works dealing with the use of marble sludge in cement composites. It was found that
adding of small amount of marble sludge into the cement composites may improve their mechanical properties [8]. As pointed out in [9], compressive strength of cement samples (w/c ratio 0.50) containing 5.0, 7.5, 10 and 15 % of marble dust as cement replacement was increased by 7 %, 4 %, 5 % and 14 %, respectively.

Grains of marble sludge clump together into lumps and thus grains of marble sludge are not homogeneously distributed through the mixture. The use of high-speed mill from Lavaris Ltd. May be an option to limit or eliminate this effect. The resulting product is micronized marble powder, which has better mechanical properties than marble sludge [10]. High-speed milling process separates all the grains of marble sludge. The high-speed milling process has been successfully used for micronization of recycled concrete [11].

2. Materials and samples
The tested mixtures consisted of Portland cement CEM I 42.5R (Radotín, Czech Republic) and micronized marble powder with fractions 0 – 40 µm. The micronized marble powder had an average grain and mean grain size of 9.74 and 4.59 microns, respectively. 90 wt. % of grains were smaller than 29.67 microns. The micronized marble powder was obtained from the processing of limestone marble in the Czech Republic as unsorted waste (Beroun, Jež Ltd.). Micronized marble powder was micronized using a high-speed mill from Lavaris Ltd. (Libčice, Czech Republic). The mixtures were designed to maintain the same workability as the fresh reference mixture. The consistency of the mixtures was determined by means of a flow expansion test after 10 (d10) and 20 (d20) impulses (lift up several centimeters and then dropped), (Table 1). The water ratio is defined as water to cement and marble powder weight ratio. Cement was replaced by the waste micronized marble powder by the amount of 25, 50 and 75 wt. percent (Table 1).

Mixtures prepared according to Table 1 were cast in the rectangular molds having inside dimensions equal to 40 × 40 × 160 mm. After casting, these specimens were kept in the molds for 24 h at room temperature of 22 °C. Each set consisted of 6 samples made of the same mixture. After demolding, these specimens were stored in a laboratory environment at 22 ± 1 °C and relative humidity 50 ± 2 % for 28 days.

| Set/ Material | Cement (g) | Marble powder (g) | Water ratio (-) | Flow expansion test d10 (mm) | Flow expansion test d20 (mm) | Bulk density (kgm⁻³) |
|---------------|------------|-------------------|-----------------|-----------------------------|-----------------------------|----------------------|
| C100          | 3000       | 0                 | 0.35            | 160                         | 190                         | 1919 ± 9             |
| C75           | 2250       | 750               | 0.31            | 157                         | 190                         | 1902 ± 4             |
| C50           | 1500       | 1500              | 0.31            | 155                         | 190                         | 1760 ± 15            |
| C25           | 750        | 2250              | 0.32            | 162                         | 195                         | 1668 ± 10            |

3. Experimental methods
In the initial stage of research, the marble powder was examined from the perspective of material composition by means of microanalysis. The tests were performed using a ZEISS Merlin electron microscope with EDS at the University Centre for Energy Efficient Buildings CTU in Prague. The output of the EDS was spectrum frequency of X-ray signal in the individual energy windows characterized by peaks (Figure 1). Individual peaks corresponded to the individual elements, whose height was proportional to the concentration of the sample element.
The values of unit weight, dimension and dynamic modulus of elasticity of the specimens were determined before the destruction test (the compressive and the flexural strength test) at the 7th and the 28th day. The non-destructive resonance method was used for the determination of the dynamic shear modulus and the dynamic modulus of elasticity. Six specimens having dimensions equal to $40 \times 40 \times 160$ mm were tested. Measurements of the dynamic modulus were carried out using the measurement set from Brüel & Kjær. By using measuring assembly and software PULSE LabShop, the basic natural frequencies were evaluated. As the next step, the standard equations for the prismatic sample were used in order to find out the dynamic modulus of elasticity and dynamic shear modulus.

Dynamic modulus of elasticity was determined by measuring the basic longitudinal natural frequency of the samples. The acceleration transducer was located in the center of the vertical side perpendicular to the length of the sample and the impact hammer strikes were carried out in the middle of the opposite side. The sample was supported by a soft and elastic pad at the midpoint of its length, which is the fundamental longitudinal nodal point.

The measurement of the dynamic modulus of elasticity was conducted by measuring of basic flexural natural frequency of the samples. In this case, the acceleration transducer was placed on the edge of the top side of the sample and the impact hammer strikes were carried out on the edge of the same side of the sample. The sample was supported by two soft and elastic pads at the fundamental bending nodal lines, which were located at the distance of $0.224 \times \text{length of the sample}$ from each end of the sample.

Dynamic shear modulus was examined by measuring of basic torsional natural frequency of the sample. The acceleration transducer was located in the corner of the vertical side parallel to the length of the sample and the impact hammer strikes were carried out diagonally on the same side. The sample was supported in the same manner as in the case of measuring of longitudinal natural frequency of the samples.

The values of the compressive and the flexural strength of the specimens were determined at the 28th day. Six specimens for each series were used for the determination of the flexural strength on beams of dimensions equal to $40 \times 40 \times 160$ mm and the uniaxial compressive test was performed on the broken halves of the specimens with effective dimensions of $40 \times 40 \times 40$ mm. The load was applied at a constant rate of 0.1 mm/s in the case of the three-point bending test and 0.3 mm/s for the compressive test by using the Heckert loading frame, model FP100. The distance between supports for the three-point bending test was equal to 100 mm.

Figure 1. EDS spectrum of the sample.
4. Results and discussion
The microscopic analysis determined the elemental composition of the waste material (micronized marble powder). The results were slightly distorted due to the carbon presence sputtered onto the sample surfaces (necessary technological step needed for the analysis). The results showed that the samples were composed of 98 wt. % CaCO$_3$. Remaining 2 wt. % consisted of minor components, of which the most important was MgCO$_3$. According to the composition, waste micronized marble can be used as an additive to blended cement.

The dimensions of all rectangular molds were measured before casting the mixture. These values were used to calculate approximate shrinkage. Samples C25 and C50 exhibited approximately the same shrinkage, concretely 0.40 ± 0.02 % and 0.37 ± 0.05 %, respectively. On the other hand, C75 and C100 reached shrinkage equal only to 0.21 ± 0.06 % and 0.19 ± 0.06 %, respectively. Increased shrinkage of samples was caused by the addition of fine particles of micronized marble powder characterized by the high specific surface area. This negative effect of powder can be eliminated by using plasticizers.

The results from non-destructive testing (Figure 2 and Figure 3) showed differences between values of modulus at the 7$^{th}$ day and 28$^{th}$ day. Differences between the values were in the size of the standard deviations. It can be stated that between 7$^{th}$ and 28$^{th}$ days, a minimal change of the dynamic modulus of elasticity and dynamic shear modulus occurred. The samples C75 had the largest value of the dynamic shear modulus, namely 7.82 ± 0.13 GPa (Figure 2) and dynamic modulus of elasticity, namely 19.19 ± 0.23 GPa (Figure 3).

The results from the destructive tests are shown on Figure 4 and Figure 5. Samples C50 reached the largest value of flexural strength, namely 3.91 ± 0.41 MPa (Figure 4). The other samples had approximately the same value of flexural strength. Differences between the values did not exceed the size of the standard deviations. Similar trends as the dynamic modulus of elasticity were observed in the compressive strength (Figure 5). The samples C75 had approximately the same value of compressive strength as reference samples (C100), namely 74.52 ± 9.14 MPa.

![Figure 2. Comparison of the dynamic shear modulus (with standard deviation).](image-url)
Figure 3. Comparison of dynamic modulus of elasticity (with standard deviation).

Figure 4. Comparison of flexural strength (with standard deviation).

Figure 5. Comparison of compressive strength (with standard deviation).

5. Conclusions
This work was focused on the influence of the micronized waste marble powder on mechanical properties of cement pastes. The researched cement pastes were composed of Portland cement and micronized marble powder in an amount of 0, 25, 50 and 75 weight percent. Based on the results, it can be concluded that:

- The average value of dynamic modulus of elasticity and dynamic shear modulus stayed unchanged between 7 and 28 days.
- For 50 wt. % of the waste micronized marble powder, flexural strength increased by approximately 50 %. For 25 wt. %, compressive strength decreased by approximately 3 %, probably due to its microfiller properties (filling pores by grains of micronized marble...
powder). For 75 wt. %, compressive strength decreased by approximately 75 %, probably due to small amount of portland cement.

- The compressive strength of cement pastes made with 25 % of micronized marble powder and cement was found to be either comparable or less than reference samples.

It can be clearly said that the investigated materials have the potential for economic savings because of their almost zero material cost. Therefore the authors consider them to be very promising for the construction of transport infrastructure. In the future, the authors will focus on determining properties such as freeze-thaw resistance and resistance to deicing chemicals.

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