MECHANICAL PROPERTIES OF LOW TEMPERATURE HYDRAULIC BINDERS

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ABSTRACT

The local mineral deposits – clay and dolomite are widely used for production of building materials in Latvia. Investigations of new materials and energy saving methods are today`s topicality. One of the possibilities is to reduce production expenses using lower firing temperature, and, in addition, providing desired strength characteristics required for building applications (Lindina et.al., 2011). For obtaining new materials the deformation and strength properties have to be analysed and taken into account.

It is known, that a great part of architectural monuments in Latvia were built using low-temperature natural cement – dolomitic Roman cement as a binder and now it is time to start their restoration. As the production of dolomitic Roman cement stopped 60 years ago, it is urgent to develop a method on how to obtain low temperature binder from dolomite and clay similar to the historic type, from natural dolomitic marl produced Roman cement.

At first, the composition as well as physical properties of hydraulic binders were investigated (Barbane et.al, 2012). In order to study the mechanical properties, a range of uniaxial loading tests with samples were performed using the testing equipment "Instron 5985". For comparison ready-mixed calcitic Roman cement „Prompt" samples were previously tested. Analysis of the results shows stable mechanical property values. The resulting binder's mechanical parameters could be used for further improvement of the historic building restoration works.

Key words: Roman cement, clay and dolomite, mechanical properties

INTRODUCTION

Roman cements are well-known low temperature hydraulic binders. Historically Roman cements were produced and used for construction of buildings up to the middle of the 20th century, when they were replaced by Portland cement. Roman cement "rebirth" started 10 years ago (ROCEM project) due to the necessity to renovate century-old buildings of historic significance. Logically, the restoration is targeted at the use of similar materials and technologies which had been applied in restored buildings.

In Central Europe calcitic Roman cements were used as a binding agent, and extensive research work has been carried out within international projects since 2003 (Hughes et. al., 2009). It should be noted that dolomitic Roman cement has not been included in these projects because calticit marl is dominant as a raw material in the main part of Europe. In Latvia, contrary to other parts of Europe, dolomitic Roman cement was produced as there were rich resources of dolomitic marlstone. A new method has been worked out on how to synthesize hydraulic binders for historic arhitectural renderings.

In this paper the most important mechanical properties of proposed binders are analyzed on the experimental background. The main attention is paid to the effects of chemical composition and hardening time of the binder, as well as the influence of other factors in order to search the areas of effective application of binders.

MATERIALS AND METHODS

Materials

Mixtures from two types of clay and dolomite in powder state were synthesized. Samples were prepared by mixing the raw materials, semidry pressing and firing at 800°C temperatures. Thermochemical processes in dolomite-clay mixtures depending on the production temperature and clay type were compared by using XRD analysis and full chemical analysis (Barbane et.al, 2012).

Two compositions from Devonian clay and dolomite with a clay content of 13%, 24% (A1, A2 respectively) and one composition using Quaternary clay with a clay content of 24% (U2) were synthesized. The chosen dolomite-clay mass ratio closely conforms to the chemical composition of natural dolomitic marl – a traditional raw material of dolomitic Roman cement used in Latvia. Temperatures of 800-850^{o}C were chosen as optimal for the synthesis of the hydraulic binder from the

mixture of clay and dolomite similar to natural dolomitic Roman cement.
For determination of mechanical properties the cylindrical samples (the ratio of the diameter-height - 1:2) of synthesized binders A1, A2 and U2 were made by adding 60% water and holding them in wet-curing conditions for 7 days (Fig.1). Tests with samples were performed after 7 and 28 days. Before the test samples were dried at 80°C for one day, they were weighed for the determination of density. Parallel, for comparison ready-mixed calcitic Roman cement "Prompt" samples were made and tested.

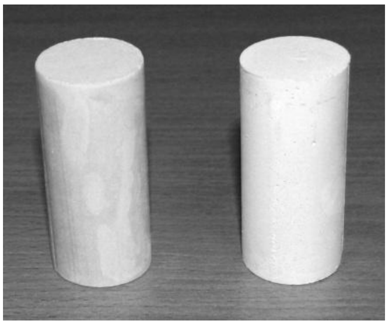

Figure 1. Cylindrical samples of synthesized binders

Methods

Testing method, sample shape and dimensions (in comparison with LVS EN 196-1) is chosen based on two considerations:

- compression of short cylinders of prismatic shapes is the most general testing method for the preliminary determination of strength of mineral materials with unknown properties;
- one of the main objectives of this investigation is to search the areas of an effective application of new material, therefore a general testing scheme was applied.

Sample size – a diameter of 28.5 mm was adopted due to a limited amount of research material.
Uniaxial compression tests were performed using the loading equipment Instron 5985, assuming the loading rate of 0.2 mm per second (Fig. 2). Conditions of uniaxial compression were provided by using of pinned joint base plates at the top and the bottom side.
As a result, data for Excel were obtained and load-extension, as well stress-deformation curves were drawn. The samples of each composition (on average five per setting group) A1, A2 and U2 were tested and the mean values assessed for sample ages of 7 and 28 days respectively.
Compression deformation modulus of the synthesized compositions is calculated using equation:

$$E = \frac{\Delta F \cdot h}{A \cdot \Delta w}, \qquad (1)$$

Where:
ΔF_u and Δw – load and extension component on the linear part of the curve;
A, *h* – cross section area and height of the sample respectively.

Figure 2. Uniaxial compression tests

Breaking compression stresses are determined and the main attention is focused on the influence of the essential factors - chemical composition, hardening time as well failure modes of the samples, water-cement ratio (W/C ratio) and the density of the binder.
Along with breaking stress and E-modulus the deformation energy is an important material characterizing factor, because of its correlation with impact resistance and fracture toughness of material (Chamis et. al., 1971). The critical deformation energy is the amount of elastic energy, accumulated in the material volume at the critical deformation or critical stress:

$$U_{cr} = \int_0^{\Delta h} F\, dh = \int_0^{\Delta h} E A \frac{\Delta h}{h} dh = \frac{E A \Delta h^2}{2h}$$

$$\text{or} \quad U_{cr} = \frac{1}{2} E \varepsilon_{cr}^{2} A h \qquad (2)$$

The critical deformation energy per unit volume:

$$\frac{U_{cr}}{V} = u_{cr} = \frac{E \varepsilon_{cr}^{2}}{2} = \frac{\sigma_{cr} \varepsilon_{cr}}{2} \qquad (3)$$

$$\text{or} \quad u_{cr} = \frac{\sigma_{cr}^{2}}{2E} \qquad (4)$$

Mainly two failure modes were observed: 1 - crushing of the top part of the samples; 2 – crushing over the whole length or in the middle of the samples (Fig.2 and 3). The first mode lead to a reduced load capacity due to the influence of air inclusions at the top part of the samples and these results were excluded from further analysis.

Figure 3. Two failure modes of the samples

Experimental results

An overview of test results is given in the diagrams below (Fig.4–7).

Ready-mixed calcitic Roman cement „Prompt" samples were tested previously (Fig.4). The methodology was accepted and first result analysis worked out during these tests.

It can be seen, that the general level of the load bearing capacity is determined by the chemical compositions of the samples. In addition other factors affect the stress and strain properties. Hardening time has a very significant impact; only composition A1 makes an exception. In our opinion, it is caused by low clay content.

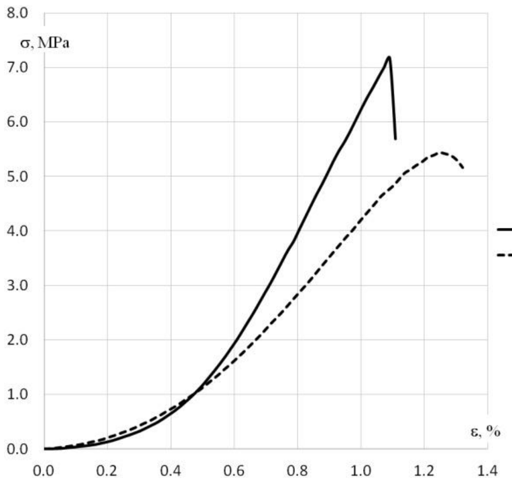

Figure 4. Test results of Roman cement "Prompt" samples: 1, 2 – hardening time of two and one week respectively

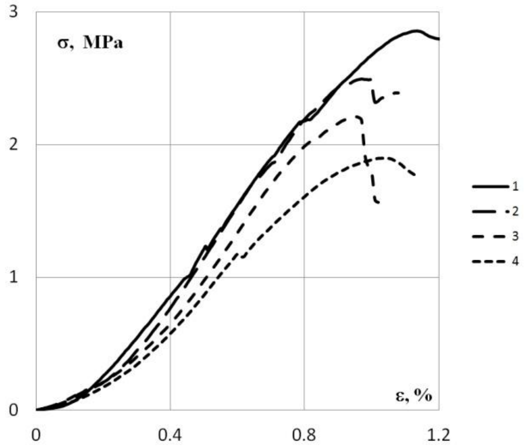

Figure 5. Test results of composition A1 samples: 1, 2 – hardening time of four weeks; 3, 4 – hardening time of one week

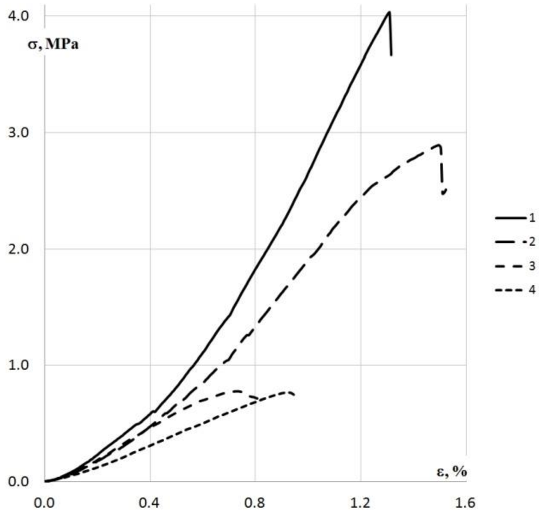

Figure 6. Test results of composition A2 samples 1, 2 - hardening time of four weeks; 3, 4 – hardening time of one week

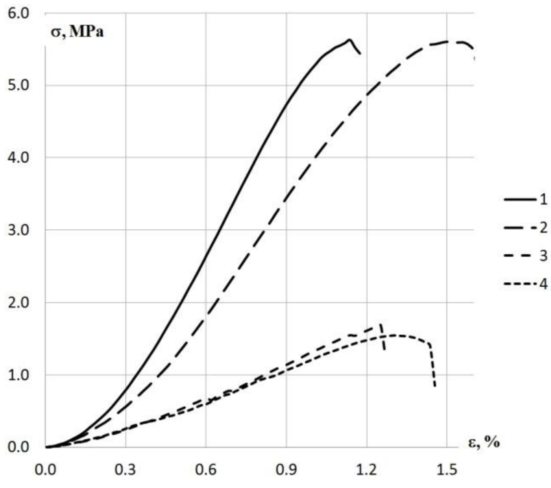

Figure 7. Test results of composition U2 samples: 1,2 – W/C ratio 60%; 3,4 – W/C ratio 70%

As mentioned before, the compositions were made by adding 60% water. It should be noted that due to rapid setting it is necessary to use a retarder – 0.6% of citric acid. The lowest possible W/C ratio is chosen to obtain a paste with sufficient workability. In order to determine the influence of W/C ratio, the additional samples of compositions U2 were made by adding 70% water. As a result a decrease in the density and strength properties can be observed (Fig. 7).

RESULTS AND DISCUSSION

The main physical and mechanical properties of the binder compositions are summarized in Table 1.

Table 1

Properties of binder compositions

Composition	No.	ρ	σ_{cr},	E	u_{cr},
		kg/m³	N/mm²	N/mm²	N*m/m³
Prompt	1``	1252	7.24	1143	22930
	2`	1190	5.44	685	21601
A1	1````	1075	4.64	354	30409
	2````	1100	4.06	387	21297
	3`	1095	3.59	349	18464
	4`	1112	3.08	259	18314
A2	1````	1200	4.03	460	17653
	2````	1160	2.89	290	14400
	3`	1190	0.76	110	2625
	4`	1200	0.78	150	2028
U2	1````	1190	5.60	600	26133
	2````	1210	5.67	720	22326
	3`	1122	1.69	180	7934
	4`	1113	1.54	160	7411

where apostrophes ` indicate hardening time (weeks)

The numerical calculation of u_{cr} was performed according to equation (4) taking into account E-modulus, obtained from the linear segment of stress-strain curves. The obtained values (max u_{cr} = 30409 N m/m^3) show that the tested specimen material critical deformation energy lays between these parameters for bulk glass (u_{cr} = ~17 000 N m/m^3) and solid clay brick (u_{cr} = ~46 000 N m/m^3) (Brencich et. al., 2001), (Bansal et.al., 1986).

As a result of the experimental tests the main mechanical properties of the synthesized compositions were assessed taking into account hardening time and density (W/C ratio) of the samples. Besides, crushing modes of the samples were also considered.

In addition it should be noted that sample properties are affected by various other factors:

- influence of air inclusions due to rapid setting (beginning in 2 or 3 minutes) observed during preparation and testing. Vibration and retarder (0.6 % citric acid) partly reduced this influence;
- imperfections in loading process induced by eventual deviations from a uniaxial scheme;
- strength property was often decreased due to material brittleness.

CONCLUSIONS

Compatible binder to historic romancement has been obtained from dolomite-clay compositions containing Quaternary and Devonian period clay. In order to study the mechanical properties, a range of uniaxial loading tests with samples were performed.

Mechanical properties (σ_{cr}, E, u_{cr}) relatively reflects the chemical composition characteristics.

Evaluating compositions with equal dolomite-clay ratio (A2 and U2): composition A2 shows variability of strength values due to great content of quartz; whereas composition U2 differs with stable strength properties and higher impact resistance (u_{cr}) which can be explained by higher amount of cement minerals.

Composition A1 shows less variability of compression modulus and hardening time influence. It might be caused by lower clay content.

Ready-mixed calcitic Roman cement “Prompt” (tested for comparison) has better strength properties in overall. In addition significant hardening time influence should be noted.

Given results could be used for prediction of the areas of application and further improvement of the historic building restoration works.

It is evident (Table 1) that a stable volume of compression modulus and the hardening rate should be taken into account in the choosing of the binder composition. Furthermore the rapid hardening materials (i.e. A1 compositions) have relatively lower strength and strain properties in comparison with the more clayey compositions A2, U2.

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