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# EXPERIMENTAL EVALUATION OF MECHANICAL PROPERTIES OF CEMENT AND CALCIUM SULPHATE MINERAL MATRIX

BY

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Abstract. The obtained results of an experimental program concerning the mechanical properties and Young modulus of a mineral matrix are presented. The composite material, which is based on mineral matrix, can be reinforced with glass fibres in order to obtain structural elements with improved behaviour under loading. To assess the optimal mixture for composite structural elements, a good knowledge of mechanical properties is needed.

The experimental program was conducted at the "Gheorghe Asachi" Technical University of Iaşi, Faculty of Civil Engineering, Composite Materials Laboratory. Different mixtures have been tested during a period of six months. The testing methodology is in accordance with standard SR EN 196-1:2006.

It was observed that the compressive and flexural strengths, as well as the modulus of elasticity, have slightly lower values compared to Ordinary Portland Cement (OPC) mineral matrix. However the workability of the mineral matrix with the new binder was highly improved compared to that of OPC mineral matrix.

Key words: mineral matrix; Portland cement; calcium sulphate.

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#### **1. Introduction**

Development of new structural systems is an important and current goal in building industry. A starting point is the composite materials which were designed based on the research conducted since the 1960s. They have been continuously improved and may lead to the development of completely new systems that meet current structural performance requirements (Singh & Garg, 2001, 2005, 2007; Țăranu et al., 2009; Singh, 1993).

### 2. General Overview

Composite materials are the key for many problems. Based on the structural behaviour aspects and those relating to technology, composite materials successfully solve the adoption of advanced structural systems. These are necessary to develop the built environment, especially in rural areas of Romania. Composite materials are the result of combining two or more constituent materials. The result of combining two or more components is a material with new properties different from those of the components and with higher, improved, characteristics (Lungu et al., 2010; WO 2010/003827 A1).

In this paper mineral matrix glass fibre reinforced composite materials are presented as a viable solution in the development of structural elements. To achieve a mineral matrix that lead to obtaining load bearing structural elements, it is necessary to use sand. Portland cement and calcium sulphate  $\beta$ -anhydride. This combination of binder has an important advantage compared to each component. The above mentioned advantage is that it can be cast into different shapes with a minimum thickness of 5 mm.

### **3. Experimental Tests**

# 3.1. Test Set Up

Materials used in the mixtures are the basic materials used in masonry mortars. Different mixtures were obtained by combining Portland cement, CEM II/A-S 32.5 R, playing the role of the activator, with sand 0...0.3 mm and calcium sulphate (insoluble anhydride) with different percentages. Anhydrous calcium sulphate in the β-anhydrite III' form is a hydraulic sulphated binder obtained exclusively from industrial waste, mostly non-recycling materials, stored in huge deposits above ground (phosphogypsum, lactogypsum, citrogypsum and Flue Gas Desulfurization gypsum).

Moreover, the production process runs at low temperatures, under 700°C, and is  $CO_2$  emissions free. It is entirely recyclable when the storage time expires, without generation of any waste products (WO 2010/003827 A1). The purpose of using this binder is to reduce the amount of Portland cement and to

improve the workability of mixtures. Table 1 presents the percentages used in the mixtures, while Table 2, the variation of water/total ratio in a single mixture.

Mixtures of Mineral Mairix						
Spec. no.	Label	Sand %	Portland cement %	Calcium sulphate %	Water/total ratio	
1	NCK 60/40/0	60	40	0	0.40	
2	NCK 60/33/7	60	33	7	0.40	
3	NCK 60/30/10	60	30	10	0.40	
4	NCK 55/35/10	55	35	10	0.40	
5	NCK 50/40/10	50	40	10	0.40	
6	NCK 50/25/25	50	25	25	0.40	
7	NCK 50/35/15	50	35	15	0.40	

Table 1Mixtures of Mineral Matrix

 Table 2

 Water/Binder Ratio Variation in NCK 60/25/15 Mineral Matrix

Spec. no.	Label	Sand %	Portland cement %	Calcium sulphate %	Water/total ratio
1	NCK 60/25/15-0.45	60	25	15	0.45
2	NCK 60/25/15-0.41	60	25	15	0.41
3	NCK 60/25/15-0.34	60	25	15	0.34
4	NCK 60/25/15-0.32	60	25	15	0.32
5	NCK 60/25/15-0.30	60	25	15	0.30

# 3.2 Testing Methodology

All components have been mixed in dry condition while water was added later using the mixer dosimeter. For 30 s the mixer run at 140 rpm (first stage), and then for 90 s at 285 rpm (second stage). The fluid mix has been cast in the three-cavity moulds especially designed for three specimens to be cast. In order to determine the mechanical properties, the mixtures were tested at different ages. Testing ages were 24 h, 48 h, 72 h, 7 days, 14 days, 21 days and 6 months. Tests consisted of determining the mechanical strengths both in bending and compression according to the SR EN 196-1:2006 normative prescriptions. The three point loading test was performed on prisms with the size of  $160 \times 40 \times 40$  mm. After the bending test, the resulting half-prisms were tested to compression. The testing equipment is semi-automatic, with a load rate in bending of 50N/s until failure. In case of uniaxial compression, the specimen is subject to a loading rate of 2,400 N/s continuously until failure (SR EN 196-1, 2006; ASTM Design. C61-94).

Cylinders having a height/diameter ratio of 2/1 were selected to accomplish the mechanical characterization of the mineral matrix, focusing on the compressive strength as well as in the Young's modulus assessment. After grinding the top and bottom surfaces, average dimensions of 50 mm diameter and approximately 110 mm height were used for all the specimens. All the specimens were tested under monotonic compressive loading at an age of 7 and 14 days (Tăranu, 2009).

# 4. Results

#### 4.1. Tensile and Compressive Strength

The results obtained at the terms established are presented comparatively in Figs. 1 and 2. The tensile strength of mineral matrix is shown in Fig. 1 while the compressive strength is shown in Fig. 2. In order to clearly observe the trends of the tensile and compressive strength values, comparative curves are required. In Figs. 3 and 4 strength *versus* age curves are presented.



Fig. 1 – Comparative chart of the flexural strengths.



Fig. 2 – Comparative chart of the compressive strengths.



Fig. 3 – Comparative curves of flexural strength.



Fig. 4 - Comparative curves of compressive strength.



Fig. 5 - Comparative chart of flexural strengths of different W/B ratio.



Fig. 6 - Comparative chart of compressive strengths of different W/B ratios.

Water/binder ratio has a very important role on the strength of a mineral matrix. The effect of the amount of water was studied by varying it for a single mixture named NCK 60/25/15. In Figs. 5 and 6 the comparative values of strengths for each W/B ratio are presented.

The effect of freezing was observed by testing mixtures NCK 60/33/7 and NCK 60/30/10 at a period of 6 months with 60 freeze-thaw cycles.

The flexural strength in case of NCK 60/33/7 was about 6.20 MPa and in the case of NCK 60/30/10 was 4.93 MPa. The compressive strength in case of NCK 60/33/7 was 32.55 MPa and 16.32 MPa in case of NCK 60/30/10.

#### 4.2 Young's Modulus Determination

In this stage two mixtures have been chosen to determine the longitudinal modulus of elasticity in compression. The chosen mixtures were NCK 50/25/25 and NCK 50/35/15. The stress-strain envelope curve in compression of NCK 50/25/25 specimen is presented in Fig. 7 while Fig. 8 shows the corresponding envelope curve for NCK 50/35/15 specimen.



50/25/25; 7 days cure period.



Fig.  $9 - 160 \times 40 \times 40$  mm prism tested in bending.



Fig. 10 – Cylinder tested in compression.

In Tables 3 and 4 are presented the average values of modulus of elasticity, maximum load and displacement at maximum load and the variation coefficient for the two mixtures tested.

Statistic Data NCK 50/25/25					
NCK 50/25/25 n = 6	E <sub>mod</sub> MPa	F <sub>max</sub> N	$dL$ at $F_{max}$ mm		
Average value	5,900	19,000	0.3		
Standard deviation	207	161	0.0		
Coefficient of variation	3.51	1.03	2.07		

Table 3Statistic Data NCK 50/25/25

Statistic Data NCK 50/35/15					
NCK 50/35/15 n = 6	E <sub>mod</sub> MPa	F <sub>max</sub> N	$dL$ at $F_{max}$ mm		
Average value, x	6,160	21,300	0.2		
Standard deviation, s	197	325	0.0		
Coefficient of variation	3.19	1.93	3.02		

 Table 4

 Statistic Data NCK 50/35/15

# 5. Disscusion

Flexural strengths obtained on eight different mixtures have values between 2.5 and 4.5 MPa. All the mixtures exceeded the 2 MPa value, which is the minimum considered to be achieved.

The compressive strengths are situated between 14 and 24 MPa. These values are reasonable considering the components of mixtures. From all of the mixtures, NCK 60/30/10 is the only one that doesn't satisfy the requirement to obtain a minimum compressive strength value of 20 MPa.

In relation to the reference mixture NCK 60/40/0, in which anhydrous calcium sulphate in the  $\beta$ -anhydrite III' form is completely missing, the mechanical strengths in both flexure and compression are higher. The strength gain was between 15% and 30% compared to the reference specimen.

By reducing the amount of water relative to the binder mixture there is an important increase in strength both in tension and compression. Inside effect of reducing the amount of water is significantly reducing the workability of mixtures.

Regarding the elastic modulus value, average values were situated between 5,900 MPa in case of NCK 50/25/25 and 6,160 MPa in case of NCK 50/35/15.

### 6. Conclusions

The period of 7 days is very important to obtain a reasonable strength because the time of execution of structures is less than 7 days. Therefore in order to be able to use the building soon, the material used must fulfil the minimum requirements which consist in strengths of 20 MPa in compression and 2...5 MPa in tension. Among the eight mixtures tested experimentally it was found that only few are suitable for structural performance requirements.

The solution regarding the combination between Portland cement and anhydrous calcium sulphate in the  $\beta$ -anhydrite iii' form could be a good choice. The effect of calcium sulphate is the significant change in workability of binder.

This new quality of material makes it possible to achieve different types of structural elements. The obtained mixture is like a fluid which can be cast in small spaces, and to cover well the reinforcement.

To establish an optimum mixture of mineral matrix eight different formulas were tested. From all the eight mixtures only two can be considered to be efficient both in economical costs and structural performance. The main mixtures are NCK 50/25/25 and NCK 50/35/15 which were tested to determine the modulus of elasticity.

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# EVALUAREA PROPRIETĂȚILOR MECANICE PE CALE EXPERIMENTALĂ A MATRICILOR MINERALE PE BAZĂ DE CIMENT PORTLAND ȘI SULFAT DE CALCIU

### (Rezumat)

Se prezintă rezultatele unui program de încercări experimentale privind determinarea rezistențelor mecanice, respectiv a modulului de elasticitate. Materialul încercat reprezintă o matrice minerală a unui material compozit armat cu fibre de sticlă din care se pot obține elemente structurale ce au o bună comportare la acțiunile exterioare. Pentru a obține un amestec optim în vederea execuției de elemente structurale, este necesară determinarea caracteristicilor mecanice.

Încercările s-au desfășurat în cadrul Laboratorului de Materiale Compozite al Facultății de Construcții și Instalații, Universitatea Tehnică "Gh. Asachi" din Iași. Pe o perioadă de 6 luni au fost testate mai multe rețete ale amestecurilor. Determinarea rezistențelor mecanice s-a realizat conform prescripțiilor standardului privind determinarea rezistențelor mecanice pe cimenturi SR EN 196-1:2006.

În concluzie rezultatele experimentale au condus la observații precum reducerea rezistențelor la compresiune și întindere din încovoiere și a valorii modulului de elasticitate în comparație cu amestecul de referință realizat din ciment Portland și nisip. Principala calitate a amestecului pe bază de sulfat de calciu în forma  $\beta$ -anhidridă III' o reprezintă lucrabilitatea sa ridicată, ceea ce face ca amestecul sa poată fi turnat în diverse forme și grosimi.