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RESEARCH ARTICLE

Behaviour of Cement Composites with Lightweight and Heavyweight Aggregates at High Temperatures

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Abstract

This paper presents new results of the research of the influence of individual input components done on the behaviour of cement based composite materials exposed to high temperatures. A new approach of this research lies in combining a fraction of basalt fine aggregate and an aggregate base on expanded clay for a composite material with lower volume weight and higher strengths and a resistance to high temperatures. In the field of dispersed reinforcement, new polypropylene fibers treated by low temperature plasma were tested. Degradation of the structure of designed mixtures was evaluated by comparing density changes, mechanical and acoustic properties before and after exposure to high temperatures. An optical microscope was used for determining the width of cracks caused by a thermal load. The contribution of this research was the use of non-destructive methods for diagnostics of quality and properties of designed concrete mixtures exposed to high temperatures.

Keywords

cement, high temperature, fire resistance, fibers, volumetric change,; mechanical properties, propagation of ultrasonic waves, modulus of elasticity, concrete

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1 Introduction

Fire safety of buildings has become more important in society recently because of more frequent acts of terrorism, arson and war operations as well as an increasing number of natural disasters and risks caused by industrial production and traffic accidents. Fires endanger human health, wildlife, personal and public property and cause incalculable environmental damage. Fire safety of buildings has to ensure a safe evacuation of people from the burning building and effective operations of a fire brigade; plus, to prevent the fire from spreading out of a building and between individual sections of a building. Therefore, an appropriate design of such structures of a building is very important, with respect to the use of optimal fire-resistant materials ensuring the maximum possible resistance of the structure to the negative impact of the fire. The aim is the development of optimal cement based composite materials applicable for the construction of buildings endangered by increased risk of possible fire: in particular road and railway tunnels, atomic power plants, high rise buildings and more and more frequently - car park buildings.

Concrete proves to be a suitable material from the point of view of fire resistance - it is non-flammable and it has low thermal conductivity. However, concrete which is not designed with respect to fire-resistance shows considerable explosive spalling caused by the combination of increasing pressure in inner pores and inner pressure tensions caused by different thermal expansion of input materials. As a result, the cross-section of the structure can be weakened, and, in the case of firereinforced concrete, even reinforcement bars can be exposed to temperatures higher than critical levels. This closely correlates to the loss of mechanical properties at higher temperatures, which is considerably affected by the selection of individual components of the concrete (type of aggregate, cement, admixtures or fibers).

The paper is focused on the research of the influence of individual components of cement based concrete on the behaviour of these materials exposed to a higher thermal load. In particular, the study of the influence of the selection of the type of cement, the aggregate and reinforcement fibers with the aim of the development of an optimal mixture of concrete resistant to fire. Knowledges of the behaviour of concrete with specific input materials are very important for the prediction and subsequent verification of the behaviour of concrete with modified composition, particularly with the addition of nanoparticles to the cement matrix. Experiments performed and described in this article are an important input for verifying the characteristics of cement composites using the same raw materials and the addition of nanoparticles, which are covered in our subsequent research works. The addition of nanoparticles can greatly increase the resistance of cement composites to high temperatures [1], [2].

2 Experimental preparation

Based on the determined aim of experimental research work, 9 mixtures of cement based concrete were designed with respect to the study of the behaviour of these materials exposed to a thermal load. The design of the composition of concrete was based on the experience with the problem of fire-resistant cement based composite materials, and on the results of literary search of technical literature [3], [4], [5], [6], [7], [8]

2.1 Selection of aggregate

An aggregate, just like almost all solid substances, expands with increasing temperature. Therefore, thermal expansion is a very important characteristic of an aggregate from the point of view of its reaction to increased temperature. Mineralogical composition decides on the total thermal distortion of the aggregate, because all minerals are different as regards to the value of thermal conductivity. Apart from thermal deformation, metamorphic changes of minerals can also occur. For example, quartz aggregate at the temperature of 573 °C and pressure 0.1 MPa increases its volume by as much as 4.7 %, carbonaceous aggregate decomposes CaCO₃ approximately about 800 °C (depending on the composition of aggregates) to CaO and CO₂, moreover, when CaO from the aggregate is cooled down, it can hydrate again and increase its volume by as much as 40 %. Based on this knowledge, the efforts aimed at using an aggregate with low thermal expansion and low thermal deformation, as is recommended in literature [9], [10], [11], [12]. Mineralogical composition and internal texture also affect the physical and mechanical properties of rocks which may also have an impact on thermal behaviour of natural aggregates [13].

Two types of aggregate were selected for this experiment based on the available data and characteristics. It was basalt from Bilcice (fractions 0/4 mm and 4/8 mm), volume weight 3027-3035 kg.m⁻³, thermal conductivity 1.67 W.m⁻¹.K⁻¹, and light weight aggregate based on expanded clay with excellent thermo-insulating properties (fraction 0/4 mm and 4/8 mm), volume weight 925–1150 kg.m⁻³, specific mass 500–600 kg.m⁻³, thermal conductivity 0.09 W.m⁻¹.K⁻¹.

A lightweight aggregate was used with a view of application

for the reconstruction of load-bearing walls so that the load of the structure could be reduced. This line of research was followed based on previous experience and results of other specialists [14], [15], [16], [17].

2.2 Selection of cement

Heating of cement causes evaporation of free and chemically bound water present in the material. Water gradually releases; first the free water leaves through capillaries, then the chemically bound water. However, if mastic cement is heated in an enclosed and humid environment (so-called internal autoclaving) hydrothermal reaction can occur, which can cause considerable changes of the microstructure both physically and chemically [4], [10].

Concrete resists heat more if it is made from cement, which releases less $Ca(OH)_2$. Portlandite decomposes to water vapour H_2O and CaO when exposed to heat (500 °C).

$$Ca(OH)_2 \rightarrow CaO+H_2O\uparrow$$

When the fire is being extinguished, water is sprayed onto the surface of the structure, which causes expansive reaction (hydrating of lime), which damages concrete:

$CaO+H_2O \rightarrow Ca(OH)_2$

This causes an increase in the volume of the concrete, which spalls at the fire. The least appropriate hydraulic binder is pure Portland cement, which contains a high proportion of Portland clinker and decomposes after a loss of chemically bound water in hydration products. Therefore it is more advisable to use blended cements, which contain less Portland clinker and more additions based on fly ash. This is the effect of blending cement, which is proved in researches of Nasser and Marzouk and Collepardi [18, [19] and other researchers [20], [21], [22] showing lower production of calcium monoxide as a result of dehydration in the heat of fire, if fly ash and blast furnace slag were used. However, Nasser and Marzouk [18] also point out that these materials are not primarily used in practice for increasing resistance of concrete to high temperatures. Experimental work of Sobhan and Peterson [23] prove the fact, that concrete exposed to fire show better behaviour when cement with a low amount of portlandite Ca(OH), is used. Decomposition of portlandite can cause production of water vapour, which evaporates and calcium monoxide CaO is formed at the temperature of 512 °C. From this point of view, concrete with blast furnace slag, metakaoline or pozzolanic cement can work better than concrete with Portland cement. For example, professor Hertz [19] examined concrete containing silica fume with the aim of proving increased resistance of such concrete when exposed to an extreme thermal load. It was found that concrete with the content of 10% of silica fume by the total amount of cementitious components showed considerable improvement compared to concrete without this addition. However, opinions

of many specialists [18], [23] focusing on this topic imply, that it is not recommendable to rely solely on these studies, which are carried out mainly in laboratory furnaces only and real conditions of fires of concrete structures are not achieved.

For the purpose of this research, three different types of cement commonly available in the Czech Republic were used based on the above mentioned knowledge. These were Portland cement CEM II/B-S 32.5 R, which is characterized by increased resistance to aggressive environment, then Portland blended cement CEM II/B-M (S-LL) 32.5 R and blast furnace cement CEM III/B 32.5 N-SV, characterized by slow development of strength, slow development of hydration heat and high resistance to aggressive environment [25], [26].

2.3 Selection of appropriate fibers

Fibers are added into concrete generally for the purpose of changing and improving physico-mechanical properties. Their use limit formation of shrinking cracks and increases resistance of concrete in the event of high temperatures; in particular, it limits the process of explosive spalling [27], [28], [29], [30].

The formation of micro-cracks can be expected in any concrete, however, their number and size is very important. Classic steel reinforcement is capable of preventing formation of cracks only to a limited extent; fibers dispersed as reinforcement can help eliminate the problem partially. If the micro cracks are created, the fibers (in the direction across the crack) take tensile stress. Another positive effect of fibers is self-curing of concrete; most fine fibers bind mixing water, which is released in the course of time and concrete is gradually hydrated. Moreover, at the temperature of around 170 °C the polypropylene fibers melt out of the matrix of the cement based composite material. Evaporated fibers created a porous system inside the structure of the concrete, which helps escaping water vapour and explosive spalling caused by high water vapour pressure is eliminated. Cellulose fibers have a similar effect - high temperature causes decarbonation of fibers.

For the purpose of experimental work, the following types of fibers were used:

- Polypropylene fibers, length 12 mm, diameter 28 μm;
- Polypropylene fibers, length 12 mm, diameter 28 μm treated by low temperature plasma, length of action of discharge 5 s [29];
- Steel fibers from carbon steel, length 25 mm, diameter 0.5 mm.

The trend of using polypropylene fibers for fire resistant concrete is generally accepted, however, there are still areas of further research. The authors examined the trend of behaviour of polypropylene fibers with surface treated by low-temperature plasma (new technology). Moreover, many teams of scientists solve the problem of the necessary amount of these fibers (from 0.8 to 2 kg.m⁻³) [27], [28], [29], [31]. Another direction worth researching is the use of modified polypropylene fibers with a high Melt Flow Index MFI 2500. Polypropylene fibers with MFI 2500 start to melt in the range 147 °C - 150 °C. Standard polypropylene fibers have MFI 25 and start to melt at 160 °C [32], [33].

2.4 Preparation of concrete

Composition of individual mixtures is stated below in Table 1. Mixtures CEM III-B/Bas-PF to CEM III-B/Bas-PF-Plas are mixtures with different types of fibers; mixture CEM III-B/ Bas is reference for these mixtures. Mixtures CEM III-B-M/ Bas - CEM III-B and CEM III-B-M/LWA - CEM III-B/LWA are designed with various dosages and types of cement, various types and proportions of fractions of the aggregate.

For the manufacturing of the concrete, water from the mains was used and super plasticizer Mapefluid N200. Mapefluid N200 is a 40% water solution of active polymers, specific gravity 1.20 ± 0.02 kg.l⁻¹ at + 20 °C.

Concretes made from mixtures stated in Table 1 were mixed in a mixer with forced circulation. All input materials were weighed on scales with the exception of the lightweight aggregate, which was dosed by volume. After mixing the concrete, the test of consistency was carried out in accordance with the standard EN 12350-2 Testing of fresh concrete - Part 2: Slump-test. The concrete mix was placed in forms treated with a release agent and compacted on a vibration table. The next day, the test specimens were taken out of the forms and placed in a humid environment for 90 days.

The water cement ratio was identical for all test specimens with basalt aggregate and various types of fibers (CEM III-B/ Bas-PF, CEM III-B/Bas-SF, CEM III-B/Bas-PF-Plas, CEM II-B-M/Bas, CEM II-B-S/Bas, CEM III-B/Bas): w = 0.5. The water cement ratio was identical for all test specimens with a lightweight aggregate: w = 0.33. The lightweight aggregate was soaked with water before mixing. The differences of pore structure of test specimens with basalt aggregate and test specimens with lightweight aggregate are caused by the different production technology. The concrete with a lightweight aggregate is in practice used, for example, for reconstructions because of reduction load of the reconstructed structure. In such cases, it is advisable to evaluate and compare the behaviour of concrete with various types of aggregate. This line of research was followed based on a previous experience and results of other specialists [14], [15], [16], [17].

Composition / Mixture	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
CEM III/B 32.5 N-SV	350	350	350	_	_	350	_	_	375
CEM II/B-M (S-LL)32.5 R	_	_	_	350	_	-	375	_	-
CEM II/B-S 32.5 R	-	_	_	_	350	_	_	375	-
Aggregate Basalt Bilcice 0/4	1070	1070	1070	1070	1070	1070	500	500	500
Aggregate Basalt Bilcice 4/8	1050	1050	1050	1050	1050	1050	_	-	-
Light weight aggregate 0/4	-	_	_	_	_		120	120	120
Light weight aggregate 4/8	-	_	_	_	_		240	240	240
Polypropylene fibers *	1	_	_	_	_	_	_	_	-
Steel fibers **	-	50	_	_	_	_	_	_	-
Polypropylene fibers*, plasma 5sec	_	_	1	_	_	_	_	-	-
Super plasticizer Mapefluid N200	3.5	3.5	3.5	3.5	3.5	3.5	4.5	4.5	4.5
Water	175	175	175	175	175	175	125	125	125

*The polypropylene fibres constitute 0.11% of the total volume of concrete. **The steel fibres constitute 0.64% of the total volume of concrete.

3 Experimental methods

3.1 Mode of thermal load

Concrete test specimens after 90 days were loaded at temperatures 200 °C, 400 °C, 660 °C and 800 °C. Thermal loading was carried out in muffle furnaces in accordance with standardized thermal curve ISO 834 with following thermal development: Rate of thermal increase was set at 11 °C.min⁻¹, thermal dwell 30 minutes at the set temperature and then gradual cooling down of test specimens to the temperature of the laboratory.

3.2 Parameters observed on test specimens before and after thermal load

Concrete test specimens after 90 days and after thermal load were subjected to the following tests:

- Compressive strength of concrete in accordance with EN 12390-3 Testing hardened concrete – Part 3: Compressive strength of test specimens;
- Flexural strength in accordance with EN 12390-5 Testing hardened concrete - Part 5: Flexural strength of test specimens;

- Density in accordance with EN 12390-7 Testing hardened concrete - Part 7: Density of hardened concrete;
- Ultrasonic pulse velocity and dynamic Young's modulus of elasticity (see chapter 3.3);
- Formation of cracks on the surface of test specimens (see chapter 3.4);
- Determination of maximal width of cracks after thermal load (see chapter 3.5).

The aim was to observe the level of degradation of properties of concrete with selected types of cement, aggregate and fibers exposed to high temperatures.

3.3. Determination of ultrasonic pulse velocity and dynamic Young's modulus of elasticity

Ultrasonic pulse method is a non-destructive acoustic method used for testing materials. It is used for construction materials mainly for determining the propagation velocity of the ultrasonic pulse and dynamic Young's modulus of elasticity. These parameters are used for evaluating the elasticity characteristics of the material being examined as well as for evaluating changes in its structure. For testing concrete with the ultrasonic pulse method, probes with natural frequency from 40 kHz to 150 kHz are used. The method is based on the emission of repeated ultrasonic pulses by a transducer into the material and receiving pulses on the other side of the specimen; the time of passage of the ultrasonic pulse through measured specimen is measured. Failures in the inner structure of the material (micro-cracks, pores and other imperfections) prolong the time of passage of the ultrasonic pulse [34], [35].

Determining the propagation velocity of the ultrasonic pulse was based on the provision of EN 12504–4 [36] and Young's elasticity modulus was determined in accordance with the procedure stated in CSN 731371 [37].

Measurements were carried by means of direct sounding along the length of the test specimen with the apparatus TICO with natural frequency of probes 150 kHz (natural frequency of probes was selected so that the ratio of wavelength (λ) to the smallest dimension in the direction of sounding (d_{min}) < 1, so that possible reduction of the velocity of the ultrasonic pulse was eliminated. Time of passage of ultrasonic pulse was measured with accuracy 0.1µs.

On the basis of transit time and previously known length of measuring base, there will be the calculation of the ultrasonic pulse velocity according to relation (1) of EN 12504-4 [36]:

$$V = \frac{L}{T} \tag{1}$$

Where:

- V ultrasonic pulse velocity [km.s⁻¹]
- L length of measuring base [mm]
- T transit time $[\mu s]$.

On the basis of the ultrasonic pulse velocity and density of the hardened concrete, there will be a calculation of dynamic Young's modulus of elasticity related to the tested material according to relation (2) from CSN 731371 [37]:

Where:

 E_{u} dynamic Young's modulus of elasticity [MPa]

$$E_U = V^2 . D . \frac{1}{k} \tag{2}$$

V ultrasonic pulse velocity [km.s⁻¹]

D density of hardened concrete [kg.m⁻³]

k ambient dimensionality ratio.

3.4 Determination of occurrence of cracks on the surface of test specimens

The surfaces of test specimens after a thermal load were photographed with a digital camera. The images were transferred into the AutoCAD software. Cracks in the images were zoomed to the maximal size resolution and then the cracks on the surface of test specimens were marked red as individual areas. By means of the AutoCAD software, the cumulative area of cracks related to the total area of test specimen was calculated. This method is based on the knowledge from paper by Xing et al. [12]. In this paper, the authors described a study of the area of cracks and measuring their widths in concrete with various types of aggregate [12]. Because cracks were observed only on specimens loaded at 660 °C and 800 °C, this method was applied only on specimens loaded at these temperatures. Another method is monitoring the presence of cracks inside the concrete using micro X-Ray Computed Tomography. Micro X-Ray Computed Tomography is more exact but requires expensive equipment and is time consuming [38].

3.5 Determining maximum width of cracks after thermal load

Determining the maximum width of cracks was carried out on test specimens after thermal loads at the temperatures of 660 °C and 800 °C. After a thermal load at lower temperatures, no measurable cracks formed on the surface of the concrete. Cracks on test specimens were observed and photographed by means of optical microscope Nikon with TV camera Sony. The picture was transmitted into PC by means or software equipment LIM Elements (LUCIA). Test specimen was placed under optical microscope with digital output and by means of a builtin scale; maximum widths of cracks on the surface of test specimens were read. The specimens were magnified 100x.

4 Results and discussion

Test specimens with dimensions 40 mm x 40 mm x 160 mm were subjected to determination of above mentioned characteristics before and after the thermal load. These values were compared and changes of individual characteristics of tested cement based composites were calculated according to Eq. (3).

$$\Delta PROP = \frac{\Pr_{ti} - \Pr_{ref}}{\Pr_{ref}}.100$$
(3)

Where:

 $\Delta PROP$ property difference [%]

Pr_{ti} value of property after thermal load

 Pr_{ref} value of property before thermal load.

4.1 Influence of thermal load on density of hardened concrete

The density of hardened concrete before and after the thermal load and evaluation of their changes are stated in Table 2 below and in Figure 1.

Table 2 Determined density before and after thermal load [kg.m ⁻³]										
Mixture	Thermal Load [°C]	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
al 3]	200	2440	2450	2440	2560	2540	2560	1700	1640	1700
therm cg.m ⁻⁶	400	2450	2420	2450	2520	2540	2560	1690	1640	1680
fore 1 ad []	660	2420	2430	2470	2550	2540	2550	1650	1690	1680
Be	800	2440	2420	2460	2540	2540	2550	1720	1670	1630
]]]	200	2390	2410	2390	2470	2480	2490	1583	1520	1560
herma (g.m ⁻	400	2360	2360	2370	2420	2441	2450	1540	1450	1430
fter tl oad []	660	2330	2360	2350	2430	2440	2440	1470	1470	1470
A	800	2350	2340	2330	2410	2450	2450	1500	1500	1460



Fig.1 Comparison of changes of density depending on the composition of the concretes and the extent of the thermal load

In general, it can be stated that as the temperature increased, the decrement of density was larger. The density of various concrete specimens differed depending on aggregate used. The density of concrete with basalt aggregate after 90 days was 2420–2560

kg.m⁻³, density of concrete with lightweight aggregate after 90 days was 1630–1720 kg.m⁻³. Concrete with dispersed reinforcement and concrete with basalt aggregate showed after the thermal load the lowest density changes. Mixture CEM III-B/Bas-SF with steel fibers showed the lowest density change: 3.3 %. Concrete with a lightweight aggregate showed the highest loss of density; this was caused by the fact that the aggregate was added into the mix saturated with water and measured by volume.

4.2 Evaluation of influence of the effect of the thermal load on strength of concrete

The results of determination of compressive strength of test specimens before and after thermal load (200 °C, 400 °C, 660 °C and 800 °C) are in Table 3 and Figure 2 below.

Table 3 Determined compressive strength before and after thermal load [MPa]

Mixture	Thermal load [°C]	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
1 ⁻²]	23	37.7	27.7	27.7	31.6	30.2	30.5	33.0	26.8	28.8
sive I.mm	200	47.2	40.1	39.5	39.0	38.3	44.4	28.0	24.8	24.1
th [N	400	34.7	30.5	31.3	30.9	34.5	37.8	21.7	20.0	21.1
Con	660	26.2	20.8	20.7	27.9	27.7	23.8	16.0	18.2	16.4
stı	800	22.0	15.9	15.2	17.5	17.4	14.7	12.0	12.1	10.1



Fig. 2 Comparison of changes of compressive strength depending on the composition of the concretes and the extent of the thermal load

Based on the analysis of the results of measurement of compressive strength depending on the thermal load, the following facts were found:

 After thermal load of 200 °C, compressive strength of concrete with basalt aggregate and various types of cement increased (CEM III-B/Bas-PF, CEM III-B/Bas-SF, CEM III-B/Bas-PF-Plas, CEM II-B-M/Bas, CEM II-B-S/Bas, CEM III-B/Bas).

- According to Hager, Zhang et al., and Wittmann [40], [41], [41] this is caused mainly by evaporation of free and partly physically bound water, which contributes to partial increasing of strength, and by non-hydrated nuclei of cement, which have positive influence on compressive strength. The results were the same as stated by Zhang and their team, who observed an increase of compressive strength after thermal load [41], [41].
- As the temperature of thermal load increased, compressive strength gradually decreased. The most marked changes of compressive strength were observed on recipes CEM II-B-M/LWA to CEM III-B/LWA mixtures with a lightweight aggregate. On the contrary, the lowest changes of compressive strength 800 °C were observed on recipe CEM III-B/Bas-PF: 41.6 %.

Then, flexural strength of concrete exposed to the thermal load was observed. Test results are given in Table 4 and Figure 3 below.

Table 4 Overview of flexural strength before and after the thermal load [MPa]

Mixture	Thermal load [°C]	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
1 ⁻²]	23	6.2	5.6	4.8	6.3	6.8	5.1	7.4	6.5	6.4
al I.mn	200	6.6	6.0	5.3	6.4	6.2	5.6	5.7	5.2	6.0
exur th [N	400	4.8	4.6	3.9	4.2	5.8	3.7	4.8	3.5	4.2
F] reng	660	3.3	2.4	2.2	2.5	3.6	2.1	1.6	3.1	3.4
sti	800	3.0	2.0	1.8	1.7	2.5	1.5	0.7	2.4	0.3

Based on the analysis of the results of the flexural strength measurement, depending on the thermal load, the following facts were found:

After a thermal load of 200 °C, the development of flexural strength of concrete was similar to the development of compressive strength. Concrete with a lightweight aggregate shows more considerable reduction of strength after exposure to 200 °C. This corresponds to more expressed changes of density of the concrete with a lightweight aggregate. This type of concrete contains a higher amount of water because of different production technology compared to standard concrete.

Recipe CEM III-B/Bas-PF also showed the lowest percentage of change of flexural strength after thermal load of 800 °C, -51.6 %. On the contrary, the highest change of flexural strength was observed on mixture CEM III-B/LWA -95.3 %.



Fig. 3 Comparison of changes of flexural strength depending on the composition of the concretes and the extent of the thermal load

4.3 Evaluation of the influence of the thermal load on ultrasonic pulse velocity and dynamic Young's modulus of elasticity

Test results of measurements by ultrasonic pulse method are stated in Tables 5 and 6. Apart from the values of ultrasonic pulse velocity and dynamic Young's modulus of elasticity determined for individual types of concrete, the differences of the values before and after thermal load calculated according to formula (3) are also stated in percentages. The Figure 4 and Figure 5 give an evaluation of differences of ultrasonic pulse velocity and dynamic Young's modulus of elasticity before and after the thermal load.

Table 5 Ultrasonic pulse velocity before	(V_{ref}) and after thermal load (V_{ti})
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Mixture	Thermal load [°C]	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
	200	4.21	3.70	3.83	4.37	4.12	4.23	3.74	3.61	3.72
m.s ⁻¹	400	4.17	3.75	3.91	4.25	4.17	4.15	3.68	3.50	3.67
/ _{ref} [k	660	4.12	3.83	3.97	4.28	4.12	4.19	3.65	3.66	3.67
-	800	4.16	3.80	3.94	4.20	4.10	4.06	3.73	3.60	3.68
_	200	3.93	3.47	3.57	3.88	3.75	3.70	3.43	3.39	3.41
n.s ⁻¹	400	3.22	2.50	3.08	3.28	3.37	2.86	2.82	2.64	2.83
V _u [kı	660	2.71	2.27	2.43	2.65	2.75	2.26	2.04	2.47	2.31
<u> </u>	800	2.72	2.23	2.41	2.26	2.28	1.84	1.78	2.11	1.91



Fig 4 Comparison of changes of ultrasonic pulse velocity depending on the composition of the concretes and the extent of the thermal load

Table 6 Dynamic Young's modulus of elasticity before $(E_{u,re})$ and after the thermal load $(E_{u,r})$

Mixture	Thermal load [°C]	CEM III-B/Bas-PF	CEM III-B/Bas-SF	CEM III-B/Bas-PF-Plas	CEM II-B-M/Bas	CEM II-B-S/Bas	CEM III-B/Bas	CEM II-B-M/LWA	CEM II-B-S/LWA	CEM III-B/LWA
	200	43.1	33.6	35.8	49.0	43.1	45.8	23.8	21.3	23.5
GPa	400	42.6	34.1	37.5	45.6	44.2	44.1	22.9	20.1	22.6
u,ref	660	41.1	35.6	38.9	46.6	43.1	44.7	22.0	22.6	22.6
щ	800	42.3	35.0	38.2	44.9	42.7	42.1	23.9	21.6	22.1
	200	36.9	29.0	30.5	37.3	34.9	34.1	18.6	17.4	18.1
GPa	400	24.5	14.8	22.5	26.1	27.8	20.1	12.3	10.1	11.4
E _{u,ti} [660	17.0	12.1	14.6	17.1	18.4	12.4	6.1	8.9	7.8
	800	17.4	11.6	13.5	12.3	12.8	8.3	4.7	6.7	5.3



Fig. 5 Comparison of changes of dynamic Young's modulus of elasticity depending on the composition of the concretes and the extent of the thermal load

Based on the analysis of the measurement results by ultrasonic pulse method, the following facts were found:

Parameters measured by ultrasonic pulse method decrease proportionally to increasing temperature to which the concrete was exposed. The reason for this decrease is changes in the internal structure of the concrete.

Level of decrease of ultrasonic pulse velocity and dynamic Young's modulus of elasticity was different and depended on the components of individual mixtures.

The influence of the components of the concrete on ultrasonic pulse velocity and dynamic ultrasonic pulse velocity and dynamic Young's modulus of elasticity was lowest at temperature 200 °C: differences of values of ultrasonic pulse velocity before and after the thermal load were between -6 % and -12 %, the differences for dynamic Young's modulus of elasticity were -14 % to -26 %. For the temperature 400 °C, the differences of values of ultrasonic pulse velocity before and after the thermal load were between -19 % and -33 %, the differences for dynamic Young's modulus of elasticity were -40 % to -57 %. After the thermal load of 660 °C, the differences of values of ultrasonic pulse velocity before and after the thermal load were between -33 % and -46 %, the differences for dynamic Young's modulus of elasticity were -40 % to -57 %. After the thermal load of 800 °C, the differences of values of ultrasonic pulse velocity before and after the thermal load were between -35 % and -55 %, the differences for dynamic Young's modulus of elasticity were -59 % to -80 %.

Influence of the components of the concrete (type of cement, dense natural aggregate, light-weight ceramic aggregate, various fibers) was marked particularly at higher temperatures ($T > 660^{\circ}$ C).

Concrete with basalt aggregate and with the addition of fibers showed the lowest decrease of ultrasonic pulse velocity and dynamic Young's modulus of elasticity.

4.4 Evaluation of presence of cracks and determination of their width

Based on the evaluation of the quality of the surface of test specimen after the thermal load it can be stated, that no cracks were formed after thermal load at 200 °C and 400 °C. Because visible cracks were formed on test specimens only after a thermal load at 660 °C and 800 °C, analysis was carried out only on these specimens.

It is clear from the images that mixture CEM II-B-M/Bas showed the lowest amount of cracks after thermal load 660 °C: 0.0 %, mixture CEM II-B-S/LWA showed the highest amount of cracks: 0.47 %. Mixture CEM II-B-M/Bas showed the lowest amount of cracks after thermal load 800 °C: 0.90 %, mixture CEM II-B-S/Bas showed the highest amount of cracks: 1.48 %.

Based on the evaluation it can be stated that mixture with a lightweight aggregate showed more cracks than mixtures with basalt aggregate from Bilcice. Corners of test specimens with lightweight aggregate flaked off after thermal load of 660 °C; this phenomenon was observed on test specimens with basal aggregate Bilcice only sporadically on one specimen of mixture CEM III-B/Bas after thermal load of 660 °C. Comparisons of formation of cracks on test specimens after 660 °C and 800 °C are given in Table 7 below.

Table 7 Comparison of formation of	cracks on test specimens / Area of cracks
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Mintura	Area of cracks [%]				
witxture	660 °C	800 °C			
CEM III-B/Bas-PF	0.012	0.037			
CEM III-B/Bas-SF	0.017	0.040			
CEM III-B/Bas-PF-plas	0.170	0.841			
CEM II-B-M/Bas	0.000	0.032			
CEM II-B-S/Bas	0.003	1.483			
CEM III-B/Bas	0.290	1.097			
CEM II-B-M/LWA	0.435	1.279			
CEM II-B-S/LWA	0.469	1.227			
CEM III-B/LWA	0.155	1.129			

Figure 6 below shows cracks of mixture CEM II-B-M/Bas after thermal load.



Fig. 6 Structure of mix-design CEM II-B-M/Bas after thermal load, determination of width of cracks

5 Conclusions

Research work focused on the study of the influence of individual components of cement based composite materials on the behaviour of these composites at higher temperatures. The aim was the development of a suitable composition of concrete which would be applicable for construction of structures endangered by higher risk of fire.

Prepared test specimens made from individual mixtures were subjected to tests in order to determine basic physical and mechanical properties before and after a thermal load. These measurements were complemented with non-destructive testing by means of ultrasonic pulse method and examination of the amount of cracks and their width by means of electron microscopy. Based on the evaluation of the behaviour of testing mixtures before and after a thermal load, it can be said:

(1) Density gradually decreases with increasing temperature. From this point of view, the use of dispersed reinforcement and basalt aggregate seemed to be the optimal solution for designing fire-resistant concrete. No major influence of cement was observed.

(2) Dispersed reinforcement, particularly with polypropylene fibers, has a positive influence on mechanical properties. Some of the mixtures showed partial increase of strength after exposition to 200 °C, which was caused by evaporation of free and partly physically bound water. Using a lightweight aggregate resulted in the degradation of mechanical properties and the highest changes of mechanical properties before and after the thermal load.

(3) Parameters from acoustic measurements decreased with increasing thermal load. However, the level of their changes varied and depended on the components used for the manufacturing of the concrete. The lowest decrease of parameters of tested concrete measured by ultrasonic pulse method was observed on mixtures with an addition of dispersed reinforcement.

(4) Microscopic examination of the concrete structure was indicated recipe CEM II-B-M/Bas as a suitable from the point of view of formation of micro-cracks; cracks were observed only after exposing the test specimen to the temperature of 800 °C. However, test specimens with dispersed reinforcement also showed very good properties.

The aim of future research is the optimization of composition of cement composites with basalt and dispersed reinforcement. Other possible properties of concrete will be studied so that the degradation of concrete exposed to a thermal load could be described more precisely, for example the thermal conductivity of the permeability of concrete.

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References

- Heikal, M., Ismail, N. M., Ibrahim, N. S. "Physico-mechanical, microstructure characteristics and fire resistance of cement pastes containing Al₂O₃ nano-particles." *Construction and Building Materials*. Vol. 91, pp. 232-242. 2015. DOI: 10.1016/j.conbuildmat.2015.05.036
- [2] Han, T., Wang, H., Jin, X., Yang, J., Lei, Y., Yang, F., Yang, X., Tao, Z., Guo, Q., Liu, L. "Multiscale carbon nanosphere-carbon fiber reinforcement for cement-based composites with enhanced hightemperature resistance." *Journal of Materials Science*. 50 (5), pp. 2038-2048. 2015. DOI: 10.1007/s10853-014-8655-8
- [3] Klingsch, W., Wittbecker, F. W. "Burning characteristics of building products in the light of European harmonization." *Fire and Materials*. 24(6), pp. 253-258, 2000. DOI: 10.1002/1099-1018(200011/12)24:6<253::AID-FAM 691>3.0.CO;2-0
- [4] Ring, T., Zeiml, M., Lackner, R., Eberhardsteiner, J. "Experimental investigation of strain behaviour of heated cement paste and concrete." *Strain.* 49 (3), pp. 249-256, 2013. DOI: 10.1111/str.12032
- [5] Teixeira, C. J. M., Rodrigues, J. P. C., Barata, P., Pires, T. A. "Experimental and numerical simulation of a natural fire in a compartment." In: Proceedings of ICEM. 15th International Conference on Experimental Mechanics, Porto, Portugal, Jul. 22-27, 2012. https://www.researchgate. net/publication/261510043_Experimental_and_Numerical_Simulation_ of_a_Natural_Fire_in_a_Compartment
- [6] Sabeur, H., Meftah, F. "Dehydration creep of concrete at high temperatures." *Materials and Structures*. 41 (1), pp. 17-30, 2008. DOI: 10.1617/s11527-006-9213-x
- [7] Zhang, H. L., Davie, C. T. "A numerical investigation of the influence of pore pressures and thermally induced stresses for spalling of concrete exposed to elevated temperatures." *Fire Safety Journal*. Vol. 59, pp. 102-110, 2013. DOI: 10.1016/j.firesaf.2013.03.019
- [8] Lunak, M., Kusak, I., Chobola, Z. "Dielectric Properties of Concrete Specimens after Heat Stress." *Applied Mechanics and Materials*. Vol. 446-447, pp. 1389-1394, 2014. DOI: 10.4028/www.scientific.net/ AMM.446-447.1389
- [9] FIB Federation of international concrete. "Fire design of concrete structures: materials, structures and modelling." Lausanne, Switzerland. 106 p. 2007. http://www.fib-international.org/fire-design-of-concretestructures-materials-structures-and-m
- [10] Bodnárová, L., Válek, J., Sitek, L., Foldyna, J. "Effect of high temperatures on cement composite materials in concrete structures." *Acta Geodynamica et Geomaterialia*. 10 (2), pp. 173-180, 2013. DOI: 10.13168/AGG.2013.0017
- [11] Martinec, P., Scucka, J., Vavro, M., Safrata, J. "Granodiorite aggregates from East Bohemia for high-performance and high-strength concretes." *Quarterly Journal of Engineering Geology and Hydrogeology.* 41 (4), pp. 451-458, 2008. DOI: 10.1144/1470-9236/07-035
- [12] Xing, Z., Beaucour, A. L., Hebert, R., Noumowe, A., Ledesert, B.
 "Influence of the nature of aggregates on the behaviour of concrete subjected to elevated temperature." *Cement and Concrete Research*. 41 (4), pp. 392-402, 2001. DOI: 10.1016/j.cemconres.2011.01.005
- [13] Vavro, L., Soucek, K. "Study of the effect of moisture content and bending rate on the fracture toughness of rocks." *Acta Geodynamica et Geomaterialia*. 10 (2), pp. 247-253, 2013. DOI: 10.13168/ AGG.2013.0025
- [14] Go, C. G., Tang, J. R., Chi, J. H., Chen, C. T., Huang, Y. L. "Fireresistance property of reinforced lightweight aggregate concrete wall." *Construction and Building Materials*. Vol. 30, pp. 725-733, 2012. DOI: 10.1016/j.conbuildmat.2011.12.081

- [15] Bogas, J. A., Gomes, A. "Compressive behavior and failure modes of structural lightweight aggregate concrete - Characterization and strength prediction." *Materials and Design*. Vol. 46, pp. 832-841, 2013. DOI: 10.1016/j.matdes.2012.11.004
- [16] Hela, R., Hubertova, M., Bodnarova, L., Klecka, K. "Lightweight Self Compacting Concrete (LWSCC) in the ready mix." In: Proceedings of the 6th International Symposium on Cement and Concrete; CANMET/ ACI International Symposium on Concrete Technology for Sustainable Development, XI AN, China. Foreign Languages Press, Beijing. pp. 1381-1390, 2006. https://www.vutbr.cz/en/research-and-development/ publications?action=detail&pub_id=62152&str=953
- [17] Jogl, M., Reiterman, P., Holcapek, O., Kotatkova, J. "Proposal of Fire Resistant Composites with Application of Lightweight Aggregate Liaver." *Advanced Materials Research*. Vol. 1054, pp. 43-47. 2014. DOI: 10.4028/www.scientific.net/AMR.1054.43
- [18] Nasser, K. W., Marzouk, H. M. "Properties of mass concrete containing fly ash at high temperatures." *ACI Journal Proceedings*. 76 (4) pp. 537-551, 1979. https://www.concrete.org/publications/ internationalconcreteabstractsportal/m/details/id/6958
- [19] Collepardi, M. "The new concrete." Grafiche Tintoretto, Castrette di Villorba. 2006. http://www.worldcat.org/title/new-concrete/ oclc/173972942
- [20] Fridrichova, M., Kulisek, K., Hoffmann, O., Dvorak, K., Magrla, R. "Utilisation of Fluidised Fly Ash for Reduction of CO₂ Emissions at Portland Cement Production." *Advanced Materials Research*. Vol. 1054, pp. 168-172. 2014. DOI: 10.4028/www.scientific.net/AMR.1054.168
- [21] Hela, R., Bodnarova, L., Marsalova, J. "Fly ashes thermal modification and their utilization in concrete." In: *System-Based Vision For Strategic And Creative Design*. Vols 1-3, Bontempi, F. (Ed), Balkem, A.A., pp. 1649-1653, 2003. https://www.vutbr.cz/en/research-and-development/ publications?action=detail&pub_id=39642&str=984&aid_redir=1
- [22] Hela, R., Orsakova, D. "The Mechanical Activation of Fly Ash." *Procedia Engineering*. Vol. 65, pp. 87-93, 2013. DOI: 10.1016/j. proeng.2013.09.016
- [23] Sobhan, C. B., Peterson, G. P. "Microscale and nanoscale heat transfer: fundamentals and engineering applications." 440 p. CRC Press, Boca Raton. 2008. https://www.crcpress.com/Microscale-and-Nanoscale-Heat-Transfer-Fundamentals-and-Engineering-Applications/Sobhan-Peterson/p/book/9780849373077
- [24] Hertz, K. D. "Danish Investigations on Silica Fume Concretes at Elevated Temperatures." *Materials Journal*. Vol. 89, pp. 345–347, 1992. https:// www.concrete.org/publications/internationalconcreteabstractsportal. aspx?m=details&ID=9750
- [25] Hela, R., Zach, J., Sedlmajer, M. "Possibilities of regulation of temperature in concrete during hydration by means of selection of suitable input materials." *Applied Mechanics and Materials.* Vol. 507, pp. 199-203, 2014. DOI: 10.4028/www.scientific.net/AMM.507.199
- [26] Zach, J., Kminova, H., Horky, O., Brozovsky, J. "Determination of intensity of hydration heat development of silicate binders." In: Proceedings of the 8th International Conference of the Slovenian Society for Non-Destructive Testing, Conference Proceedings: Application of Contemporary Non-Destructive Testing in Engineering, Portoroz, Slovenia, Sep. 1-3, 2005. pp. 375-382, 2005. http://www.ndt.net/article/ ndt-slovenia2005/PAPERS/45-NDTP05-66.pdf
- [27] Ciganek, J., Materna, A., Brozovsky, J. "Optimalization of the Fibre-Concrete Mixture." In: Proceedings of the 5th International Conference Fibre Concerete, Prague, Czech Republic, Sep. 17-18, 2009. pp. 71-77, 2009.

- [28] Rehacek, S., Hunka, P., Citek, D., Kolisko, J., Simunek, I. "Impact Resistance of Fibre-reinforced Concrete." *Advanced Materials Research*. Vol. 1054, pp. 48-53, 2014. DOI: 10.4028/www.scientific.net/ AMR.1054.48
- [29] Bodnarova, L., Fialova, M., Kopkane, D., Moravek, T., Stahel, P., Cernak, M. "Importance of polypropylene fibers as a reinforcement in concrete." *Chemicke Listy.* Vol. 106, pp. 1427-1430, 2012.
- [30] Mindeguia, J. Ch., Pimienta, P., Carre, H., La Borderie, Ch. "Experimental analysis of concrete spalling due to fire exposure." *European Journal of Environmental and Civil Engineering*. 17 (6), pp. 453-466, 2013. DOI: 10.1080/19648189.2013.786245
- [31] Huismann, S., Weise, F., Meng, B., Schneider, U. "Transient strain of high strength concrete at elevated temperatures and the impact of polypropylene fibers." *Materials and Structures*. 45 (5), pp. 793-801, 2012. DOI: 10.1617/s11527-011-9798-6
- [32] Knack, I. "The use of PP fibers in tunnel construction to avoid explosive concrete spalling in case of fire. New test results for the clarification of the mode of action." In: 2nd International RILEM Workshop on Concrete Spalling due to Fire Exposure. Koenders, E. A. B., Dehn, F. (Eds), RILEM Publications SARL, pp. 297-307, 2011. http://www.rilem.org/ gene/main.php?base=500218&id_publication=408&id_papier=7694
- [33] Fiedlerova, M. "Behavior of concrete at high temperatures." Master thesis, Supervisor: L. Bodnarova, Brno University of Technology, Brno. 98 p. 2014.
- [34] Brozovsky, J., Martinec, P., Matejka, O. "Using of ultrasonic pulse method for prediction of strength of blended cements." In: Proceedings of the 8th International Conference of the Slovenian Society for Non-Destructive Testing, Conference Proceedings: Application of Contemporary Non-Destructive Testing in Engineering, Portoroz, Slovenia, Sep. 1-3, 2005. pp. 117-122, 2005. http://www.ndt.net/article/ndt-slovenia2005/ PAPERS/13-NDTP05-06.pdf
- [35] Brozovsky, J., Zach, J. "Ultrasonic Pulse Method the Cement Strength Evaluation Tool." In: Proceedings of the 2nd International Conference on Waste Engineering Management, ICWEM. Xiao, J. Z., Zhang, Y., Cheung, M. S., Chu, R. P. K. (Eds), Rilem Proceedings. Vol. 73, pp. 314-323, 2010. http://www.rilem.org/gene/main.php?base=500218&id_ publication=77&id_papier=3559
- [36] British Standards Institution. "Testing concrete Part 4: Determination of ultrasonic pulse velocity." EN 12504–4. CEN. 2004.
- [37] Technical and Test Institute for Construction Prague. "Nedestruktivní zkoušení betonu – Ultrazvuková impulzová metoda zkoušení betonu." (Non-destructive testing of concrete - Method of ultrasonic pulse testing of concrete.) CSN 731371. 2011. (In Czech) http://www.technickenormy-csn.cz/731371-csn-73-1371_4_88876.html
- [38] Sitek, L., Bodnarova, L., Souček, K., Staš, L., Gurkova, L. "Analysis of inner structure changes of concretes exposed to high temperatures using micro X-Ray Computed Tomography." *Acta Geodynamica et Geomaterialia*. 12 (1), pp. 79-89, 2015. DOI: 10.13168/AGG.2015.0009
- [39] Hager, I. "Behaviour of cement concrete at high temperature." *Bulletin of the Polish Academy of Sciences-Technical Sciences*. 61 (1), pp. 145–154, 2013. DOI: 10.2478/bpasts-2013-0013
- [40] Zhang, B., Bicanic, N., Pearce, J. Ch., Phillips, D. V. "Relationship between brittleness and moisture loss of concrete exposed to high temperatures." *Cement and Concrete Research.* 32 (3), pp. 363-371, 2002. DOI: 10.1016/S0008-8846(01)00684-6
- [41] Wittmann, F. H. "The structure of hardened cement paste-a basin for a better understanding of the material properties." In: Hydraulic cement pastes: Their Structure and Properties. University of Sheffield, Wegsham Springs, Apr. 6-9, 1976. pp. 96-117, 1976. https://trid.trb.org/view. aspx?id=44012