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RESEARCH ARTICLE

Composites Based on Alternative Raw Materials at High Temperature Conditions

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Abstract

This paper presents newly developed polymer-cement composites. The primary binder (cement) was partially substituted by use of blast-furnace slag and high-temperature fly ash. A lightweight aggregate – agloporite (grain size in range 1-2 mm) was used among other components. This porous aggregate is produced from energy by-products (fly ash). Attention was focused on the behavior of the composites when exposed to elevated temperatures (400 °C–1,000°C). The influence of several different methods of temperature decrease was assessed – slow (in furnace 1°C/min) and rapid (laboratory ambient 22°C and water bath 18°C). Specific dimensional changes were determined, including strength characteristics and bulk density.

Structural deterioration and microstructural changes of selected specimens were investigated by analytical techniques (SEM and CT). Compressive and bending tensile strength changed variously depending on temperature changes, including several cooling conditions. Deterioration reactions (especially cracks) which were formed in investigated composite structures corresponded with results of physico-mechanical testing. That was confirmed by using the CT and SEM.

The fact that the agloporite has a positive effect on thermal resistance of developed polymer-cement composites was proved. Almost no cracks or other failures were identified (by using CT and SEM) in interfacial transition zones of agloporite after thermal stress. This indicates very good bond adhesion between the matrix and the porous aggregates during extreme temperature conditions (in case of different cooling methods).

Keywords

elevated temperature, rapid cooling, polymer-cement, mortar, fly ash, blast furnace slag, agloporite, strength, density, bond, residual properties

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1 Introduction

The research presented in this paper is focused on new repair mortars development. These mortars are based on alternative raw materials. In terms of simulating various exposition conditions, attention was paid to three methods of cooling. A non-traditional laboratory technique (computational tomography – CT) was used for assessing changes in mortar structure. Therefore, selected important information pertaining to this issue is described below (suitable alternative raw materials, cooling methods, use of CT, etc.).

Composites based on polymer-silicate matrices or binders are widely used in the construction industry. The applications include mortars for repair of reinforced concrete structures. Extreme temperature-resistant repair mortars should be used when a possibility of increased risk of accidental circumstances such as fire exists. Such mortars must fulfill certain demands concerning fire resistance of those elements repaired with the mortar. Simultaneously, good interaction between mortar and the repaired element must be achieved during thermal stress and subsequent cooling (extinguishing fire).

High temperatures (up to 1,300°C) during fires may affect building structures, so choosing suitable materials and specific compositions is important. Globally there are efforts to get potentially suitable raw materials from alternative sources. Development of repair mortars resistant to extreme temperatures using the raw materials from alternative sources is a very advantageous possibility.

Nevertheless these alternative raw materials are characterized by variability of their composition and parameters that depend on many factors. A thorough analysis of alternative raw material's effects on the parameters of the newly designed and developed material is essential for its proper functioning in the building structure.

Extreme temperature resistance of composites based on silicate matrices has been investigated by many researchers. The influence of various mixture components has been studied by modification of binder, aggregate and dispersed reinforcement or, eventually, by using additives and admixtures. Specific attention has been paid to the substitution of the primary binder (cement) by fly ash, blast furnace slag or addition nanoparticles of SiO₂, TiO₂, Al₂O₃, etc; [1] to [7].

It was found that blast furnace slag has a positive effect on the thermal resistance up to about 1,000°C. In the case of fly ash, improvement was evident only up to 800°C. The origin of aggregate used, which occupies most of the volume within the composite is as important as matrix binder type.

Research into using mainly lightweight aggregates from alternative sources is covered in various studies (see the references below). To this extent, the impact of pumice, slag, perlite, vermiculite, pyrolite, etc. was monitored and evaluated. Noteworthy results and findings are presented in [8] to [10]. The use of porous aggregates had a very good effect on the thermal resistance of composites based on silicate matrices. Results of research into assessing the partial replacement of aggregates by recycled glass in self-compacting mortars was presented by Guo et al. [12].

The cooling method during extinguishing of fire is another significant factor that contributes to final (residual) parameters of affected materials. In terms of basic research, slower cooling is usually preferred in laboratory testing of fire resistance (in furnaces) in order to simplify the process. However, real-life conditions are better characterized by rapid cooling by water. Situations where the centre of a fire moves or fire ceases to exist may occur. Thus there may occur a rapid cooling by the ambient air at a temperature of about $0-35^{\circ}$ C (depending on weather).

The influence of the cooling method on the parameters of mortars based on blast furnace slag is presented by Shoaib et al. in [13]. Three methods of cooling were specifically investigated – slow cooling and shock cooling using water/air. Test specimens were exposed to temperatures up to 600°C. Shoaib et al. [13] came to the conclusion that water cooling did not result in cracking as much as shock cooling with air or gradual cooling in the furnace.

Use of non-traditional analytical techniques could be a very intriguing and suitable possibility for the assessment of building materials structural changes due to thermal exposition. Beneficial findings concerned with research into building materials are presented by Kim et al. [25], Henry et al. [26] and Ponikiewski et al. [27]. Kim et al. [25] presents an evaluation of pore structures and cracking in cement paste exposed to elevated temperatures by X-ray computed tomography. Ordinary Portland cement was used, prepared according to ASTM C 150. Mortar specimens were produced with a mixture of silica sand (maximum grain size 0.67mm) as an aggregate. Kim et al. [25] found that a massive network of fractures formed at temperatures greater than 900 °C, causing explosive spalling, which is considered the most damaging effect of fire on cement-based materials. Therefore CT appears to be a powerful technique with which to address the physical changes caused by elevated temperatures in composites based on cement matrix. Based on a survey of scientific literature it can be stated that there exists a wide area for research.

2 Materials and Experimental Procedures

Two mortars based on polymer-silicate matrix were designed. The first type of mortar contained blast furnace slag (Kotouč Štramberk; marked BFS). The second type of developed mortar contained high-temperature fly ash (power plant Počerady; marked FA). These alternative raw materials substituted for the binder (cement). Each substitute (FA, BFS) was applied in the amount of 35% (by weight). The aforementioned components come from alternative raw material sources currently produced in the Czech Republic.

Fly ash Počerady is characterized by specific surface $305m^2/$ kg, density 2,038kg/m³ and loss of ignition 1.23%. Blast furnace slag has following parameters: specific surface $364m^2/$ kg, density 2783kg/m³ and loss of ignition 1.8%. Cement CEM I 42.5 R (marked CEM) was used as the primary component of the binder – specific surface $457m^2/$ kg, density 3,079kg/m³ and loss of ignition 3.2%. Chemical and mineralogical composition of above mentioned components is stated in the tables below (see Table 1 and 2).

Table 1 Chemical composition of cement and alternative raw materials

Component	СЕМ	FA	BFS
SiO ₂	20.1	54.72	37.9
Al_2O_3	4.8	29.83	5.8
Fe ₂ O ₃	3.1	5.31	
SO_3	2.9	0.02	
CaO	63.7	1.82	40.8
MgO	1.4	0.87	12.1
K ₂ O	0.81	1.38	
Na ₂ O	0.07	0.36	
P ₂ O ₅		0.09	
MnO	1.1		0.7

Table 2 Mineralogical composition of cement and alternative raw materials

СЕМ	FA	BFS
C3S	Quartz	C2S
C2S	Mullite	C3A
C3A	Maghemite	Åkermanit
C4AF	Amorphous phase	Merwinite
MgO	Magnetite	Lime
		Quartz
		Portlandite
		Calcite
		Periklas
		Brucite
		Geglenite
		Hematite
		SiC
		Amorphous phase

An active support ingredient (microsilica) was also added (6%) along with a polymer additive based on a copolymer of vinyl acetate and ethylene (3%).

Černý et al. [14] and [15] discusses the possibility of producing porous aggregates based on sintered ash (agloporite). It is evident from the results that a body (base structure of the sintered aggregate) may be resistant to temperature up to 1,500°C. Lack of knowledge in this area is evident. There are no publications or studies discussing in detail the possibility of composites based on polymer-silicate matrix and aglpoporite resistant to high temperatures.

This type of porous aggregate is therefore considered as one of the developed composite components. Thus the aggregates were formed by a mixture of amphibolite and agloporite. The aggregate mixture consisted of agloporite with particle size in range of 1-2mm, in combination with amphibolite of grain size 0-1mm. Agloporite is characterized by loose bulk density around 740kg/m³, porosity approximately 32.4% and water absorbing capacity near 19.5%. Phase and chemical composition is close to primary raw material, i.e. high temperature fly ash (mullite, anorthite, silica). The essential factor is the behavior at high temperatures. Resistance to temperatures up to around 1,200°C was proved in the furnace with observation possibility. Amphibolite has loose bulk density up to 1,530kg/m³, porosity 4.4% and water absorbing capacity 1.2%. The amphibolite could be characterized as metamorphosed rock based on minerals - mainly amphibole, hematite, pyroxene and spar. Initiation of shape change of grains of amphibolite was identified in the furnace with observation possibility at the temperature of 1,178°C. Results of sieve analysis are given in the graph below (see Fig. 1; amphibolite – M, agloporite – G and mixture – MG).



Fig. 1 Sieve analysis of agloporite and amphibolite

Dispersed reinforcement was also applied based on cellulose fibres in amount of 0.53 kg/m^3 .

All in all, 120 (60 of each mortar formulation) specimens of dimensions $40 \times 40 \times 160$ mm for determination of compressive and flexural strength including bulk density were produced. The average was always determined from testing of four specimens. Mortars were also applied on 6 concrete blocks of dimensions $600 \times 300 \times 50$ mm in layer of 20mm for bond strength determination. The test specimens, including concrete blocks with mortars, were covered with a foil for three days after

forming, i.e. maintained in such an environment to prevent rapid evaporation of water. Afterwards, the prism specimens were left free under laboratory conditions, i.e. at about 22°C (reference specimens) and a relative humidity of approximately 45%. This was followed by incremental thermal exposure at 400, 600, 800 and 1,000°C.

Table 3 Formulations of developed mortars				
Commonant	11 : 4	Mixture		
Component	Unit	BFS/MG	FA/MG	
Cement I 42.5	kg.m ⁻³	423	423	
Blast furnace slag – Štramberk	kg.m ⁻³	227	_	
High temperature fly ash – Počerady	kg.m ⁻³	_	227	
Admixture vinyl acetate copolymer	kg.m ⁻³	19.5	19.5	
Microsillica	kg.m ⁻³	39	39	
Amphibolite 0-1mm	kg.m ⁻³	842	842	
Agloporite 1-2mm	kg.m ⁻³	641	641	
Cellulose fibres	kg.m ⁻³	0.53	0.53	
Water	kg.m ⁻³	173	181	

Gradual cooling was carried out and took place in furnaces with a rate of about 1°C/min. The shock cooling of the specimens was carried out by taking the specimens out from furnaces at given temperatures and stored in laboratory conditions to be rapidly cooled by air at about 22°C (see Fig. 2). The second, more intense method presented immediate immersion of the specimens into a water bath at about 18°C (see Fig. 3).



Fig. 2 Rapid cooling by air (from 1,000°C)



Fig. 3 Rapid cooling by water (from 1,000°C)

Then the test specimens (dimensions 40×40×160mm) were used for determining basic material characteristics. Advanced physico-chemical and microstructural analysis (CT and SEM) were also done. Mortars were assessed visually as well. Additionally, the mortar was applied to a substrate (concrete blocks $600 \times 300 \times 50$ mm) representing a real-life structure. A layer was created of 20mm. After 28 days, a fire test by a real flame was carried out to achieve a temperature of about 1,000°C at the substrate/mortar interfacial zone (see Fig. 4). After the desired temperature was reached, it was maintained for about 20 minutes. Then occurred shock water cooling – spraying to simulate a fire extinguishing action as in case of real construction. Bond strength was determined on the reference and thermally stressed mortar in accordance to technical standard [20].



Fig. 4 Thermal stress of mortar before bond strength testing – FA/MG

Use of CT for non-destructive assessment of structural changes of materials exposed to extreme temperatures is very advantageous. This technique could be very effective, particularly when combined with microstructure methods – SEM for examination of degradation of structure.

3 Results

Subsequent figures (see Fig. 8 and 9) give comparison of dimensional changes (length - 1, width -b and height - h of prism specimens $40 \times 40 \times 160$ mm) of tested mix-designs from the view point of various conditions of cooling down.



Fig. 5 Dimensional changes - slow and rapid (by air) cooling

For better clarity, shock cooling in air and in water is compared separately (indicated as A - shock air cooling, W - shock water cooling; gradual cooling is not marked).



Fig. 6 Dimensional changes - slow and rapid (by water) cooling

Below are the graphs (see Fig. 10–12) that show a comparison of the bulk density (marked ρ) and strength characteristics (marked fc – compressive strength; fb – flexural strength) results. Each graph shows a comparison of all the formulations incl. all types of cooling.



Fig. 7 Bulk density - slow and sudden cooling (air, water)



Fig. 8 Compressive strength - slow and sudden cooling (air, water)

The left vertical axis (see Fig. 10–12) represents monitored parameters. Percentage changes of the monitored parameters after thermal stress are given on the right vertical axis. Percentage change is marked by symbol " Δ " in the legend of graphs.



Fig. 9 Flexural strength – slow and sudden cooling (air, water)

The next parameter analysed was bond strength (determined by pull-off). This type of stress represented real-life conditions of repair mortar applications in the construction. Therefore bond strength is quite an important characteristic for repair mortars. Evaluated bond strength results (before and after thermal stress) are given in the table below (see Table 3).

Table 4	Evaluation	of bond	strength	by pull-off method
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Mixture – Exposition temperature	Bond strength [N.mm ⁻²]	Change of bond strength [%]	Place of failure
BFS/MG – 22°C	2.34		Mortar
BFS/MG - 1000°C	0.78	66.7	Concrete base/ Mortar
$FA/MG - 22^{\circ}C$	1.92		Mortar
FA/MG - 1000°C	0.69	64.1	Concrete base/ Mortar

During the thermal stressing of composites based on polymer-silicate matrices a gradual decomposition or modification change of some phases or minerals occurs. The cracks could be formed in matrices or aggregates as well. Failures, particularly in the interfacial zone of matrix and aggregates, are also no exception. In the overwhelming majority of cases each component in the composite system has a different coefficient of thermal expansion. Computer tomography (CT) was used as one of the key analysis methods. Reference specimens were investigated, as well as those exposed to 1,000°C and subsequently cooled in various ways. Selected outputs of CT analysis are shown in the following pictures (see Fig. 13–17).

CT analysis was also supplemented by the SEM method, where the degradation of the matrix was the focus, namely the matrix hydration products and microscopic cracks found generally in the matrix-to-filler interface (see Fig. 18, 19).



Fig. 10 CT picture - slice of BFS/MG, exposure 1,000°C, slow cooling



Fig. 11 CT picture – slice of BFS/MG, exposure 1,000°C, cooling by air shock (detail of crack – on the right)



Fig. 12 CT picture – slice of BFS/MG, exposure 1,000°C, cooling by water shock



Fig. 13 CT picture – slice of BFS/MG, exposure 1,000°C, cooling by water shock (detail of cracks)



Fig. 14 CT picture – slice of FA/MG, exposure 1,000°C, cooling by water shock (detail of cracks)



Fig. 15 Microstructure of BFS/MG, exposure 1,000°C, sudden cooling (water; bond of matrix and porous aggregate), mag. 500×



Fig. 16 Microstructure of BFS/MG, exposure 1,000°C, sudden cooling (water; crack on the surface of the matrix pore), mag. $1,000\times$

4 Discussion

4.1 Physico-mechanical parameters

Agloporite of particle size in range 0-1mm has far fewer pores than other larger grains of this aggregate. For this reason, agloporite of particle size in range 1-2mm was used with respect to possible volumetric changes. It is interesting that most marked volumetric changes occur at temperature 800°C (mix-design FA/MG). Different volumetric changes in dependence on every single method of cooling (slow and rapid) are also intriguing. Contractions prevail in both cases, i.e. cooling by water and air. However, noticeable expansion was observed in the case of rapid cooling by air (see Fig. 9, temperature 800°C). Based on comparison of influence of rapid cooling down with air it can be stated that relatively fewer changes occur at temperatures up to 600°C. Considerable difference was observed for curves Δb or Δh (change of specimens crosssection) for mix-design FA/MG, where the development was diametrically opposed. Cooling by water caused significant reduction of dimensions, respective to volume after exposition to temperature 800°C (see Fig.9). On the contrary, cooling by air caused expansion by about 1.8% (see Fig 8). For this reason, microstructure was examined. It is important that more marked variations of dimensional changes were observed in the transversal direction of test specimens.

Bulk density of the mortars without thermal stress (see Fig. 10) was around 1.600kg/m³. Due to thermal exposure, a decrease of about 15% is noticeable. In the case of gradual cooling, no significant differences are recorded among formulations containing slag and fly ash.

More noticeable differences may be found in the case of shock cooling as mostly seen for the immersion into a water bath. For the sudden cooling with water there is no significant decline as that observed for gradual cooling in furnaces. This is most likely due to the ongoing decomposition of matrix components as a result of increased temperature in the furnace. The course of the decline in density due to heating and subsequent cooling with water suggests a better temperature resistance when using fly ash as a modification component of the matrix. The FA/MG mortar features a decrease in density of up to 10% if heated to 1,000°C and shock cooled.

The decrease of compressive strength (see Fig. 11) is much more significant compared to bulk density. Under reference temperature conditions, mortars containing slag / fly ash reach $36.2N/mm^2 / 33.5N/mm^2$. In the case of rapid cooling by water, significant differences were evident between BFS/MG and FA/ MG mortars. However, this occurred in temperature range of 400 to 800°C.

After exposing mortars to 1,000°C, substitution of the binder using the mentioned materials showed almost identically for all ways of cooling. Differences of the residual compressive strengths after exposition to 1,000°C were negligible. Water cooling had less negative effect on the final strengths

than the slow cooling. Average values of residual compressive strength were observed in range from 28% (9.7N/mm²) to 32% (11.8N/mm²).

The mixed binder based on cement and slag appears to be much more resistant than that based on fly ash in the case of shock cooling in water from temperatures of 400 to 800°C. Nevertheless, slow and rapid cooling by air is characterized by very similar effects on compressive strength decrease for both the tested formulations.

By using blast furnace slag (BFS/MG) about 17% higher flexural strength (see Fig. 12) is evident in the case of reference specimens (those without thermal load). After exposure to 800°C, flexural strengths became comparable for both BFS/MG and FA/MG. The mixture containing fly ash can be assessed as better in case of thermal stress at 1,000 °C. There is no significant decrease of flexural strength after 600°C exposure with subsequent slow and rapid cooling by air (except values Δ ff-BFS/MG; see Fig. 12) which is quite an interesting finding. On the contrary, rapid cooling in water had a very significant impact on the flexural strength decrease after exposure up to 400°C. Composite BFS/MG showed a decrease of analysed characteristic slightly over 50% after exposure at 400°C (at 1,000°C was evident decrease of about 87%). So it can be stated that investigated mortars are more sensitive to rapid cooling by water in terms of flexural strength compared with compressive strength.

The fact that this effect mostly occurs up to 400°C is important, which is an interesting finding. At lower temperatures there is not such a considerable decomposition of matrix hydration products (portlandite, etc.). Subsequently, the residual compounds could be rehydrated and thereby enlarge their volume. Such an increase of volume would then logically cause expansion. This expansion may be related (if pore and cell volume is insufficient) to the pressure that could lead to destruction of the material structure.

Different behaviour of analysed mortars BFS/MG and FA/ MG in terms of rapid cooling in water is a very interesting finding. Using blast furnace slag better effected extreme temperature impacts on compressive strength. On the contrary, substitution of binder by fly ash can be assessed more positively in case of flexural strength.

Results of bond strength (see Table 1) show that substitution of the cement by slag can be evaluated as a better option than fly ash. However, the difference is not striking. After thermal stress 1,000 °C and subsequent shock cooling by water, bond strength declined to 0.78N/mm² (33% residual strength, BFS/ MG) and 0.69N/mm² (36% residual strength, FA/MG). The results of both formulations are very similar considering the residual strengths (thermal resistance). When compared with the above characteristics under consideration, the course of bond strength values can be stated to rather approximate the compressive strength. This can be demonstrated through similar residual strength values (bond and compression).

4.2 Micro/structure

CT analysis showed formation of cracks/micro-cracks particularly in the matrix and in the interfacial transition zone (contact of matrix and aggregate). It was primarily found that cracks often occur in water shock cooled mortars. In this case the cracks were characterized by greater length and width. Mortars cooled by air (slowly or even rapidly) contain virtually no defects in structure. An overwhelming majority of cracks occur in the interfacial transition zones – around the grains of amphibolite. Cracks were recorded sporadically in amphibolite as well. Agloporite grains were also affected by cracks in the structure, which however did not occur in their interfacial transition zone with the matrix. Surface structure of these porous aggregates ensures very good bond with silicate matrices, which is investigated by Ke et al. [21].

Also significant is the fact that cracks of BFS/MG and FA/ MG were identified in slightly higher amounts identified with mortars containing blast furnace slag. The area where cracks are formed is important. Shock cooling by air caused development of cracks mainly in surface areas of the specimens. On the other hand, shock cooling in water caused formation of cracks evenly throughout the entire structure of the specimens.

This corresponds with the method of cooling. When the material is put into water it is cooled much more rapidly and due to its capillary-porous system, more evenly. Further it is also evident that identified cracks significantly influence changes of flexural strength (apparent connection with rapid cooling by water). Differences of flexural strength depending on various cooling ways are most noticeable in the range from 400°C to 600°C.

At higher temperatures, the structure is more degraded, so the less compact material is not so sensitive to thermal shock. It is important that CT revealed almost no cracks in the structure of mortars exposed to temperatures 400 to 800°C. These cracks were observable only by SEM. Dimensions of identified cracks were in μ m. As regards matrix composition, more cracks (identified by both CT and SEM) were observed in materials with blast furnace slag. However, the differences between BFS/MG and FA/MG are minimal.

Cracks determined by SEM were observed in the matrix and particularly at the interfacial transition zone around the amphibolite. Matrix structure degradation was increasingly observed in specimens cooled slowly. Conversely, cracks were identified in a greater amount within the structure of mortars rapidly cooled by water. The interfacial transition zone between agloporite and matrix (see Fig. 18) was well retained even at exposition to 1,000°C. The figure shows matrix penetrating the surface area of the porous aggregates grains. There is in the picture (see Fig. 18) also a clearly shown degradation of matrix structure. Subsequent figure (see Fig. 19) shows cracks of μ m width on the surface of the pore in matrix also damaged by extreme temperature.

4.3 Recommendation for consequential research

On the basis of the aforementioned methods and outcomes, only certain consequences and intriguing findings concerned with the various behaviour of investigated mortars thermal resistance were exactly described. Continued testing could be recommended. Determination of the modulus of elasticity in compression seems to be a key factor, particularly in cases of various mortar compositions (fly ash, blast furnace slag etc.). Some materials (especially containing fly ash) are characterized by a lower modulus of elasticity through which it may be more resistant for example in terms of flexural strength. Conversely, a higher modulus of elasticity for mortars containing slag would be reflected in higher compressive strengths. Modulus of elasticity is important in terms of thermal resistance as well.

Although complex sets of techniques and procedures were used, consequential research would be suitable. Very interesting results and findings regarding the problem of the interfacial transition zone between aggregates and matrix are stated in studies [22] to [24]. These studies present both current knowledge and new progressive methods, however porous aggregate or particularly agloporite are not investigated. Interfacial transition zone of agloporite and silicate matrices (incl. polymer-cement) are not thoroughly examined at either normal or extreme temperatures.

Research of developed materials after longer periods of ageing (90, 180 and 360 days) including exposition to chemically aggressive environment (sulphates, chlorides) is a very interesting topic extending beyond the presented and discussed results and findings. Then, after various types of cooling a synergic effect of real adverse influences could be simulated and investigated more closely.

5 Conclusions

The results of the investigations presented in this paper show that with alternative raw materials (35% of cement substituted by high temperature fly ash / blast furnace slag, high proportion of porous sintered fly ash based aggregates) it is possible make a composite material with high resistance to extreme temperatures. Considerable attention was paid to evaluation of properties and structure respectively microstructure after various cooling conditions. These conditions involve shock cooling by air and water, which should simulate real conditions. CT was a very important examination method of the extreme temperatures influencing investigations. Non-destructive examination of three-dimensional structure is possible by using this technique. CT analysis showed that porous aggregate - agloporite with particle size in range of 1 - 2mm, contributes positively on thermal resistance of analyzed mortars. Only very few cracks were identified in the interfacial transition zone of aforementioned porous aggregates and matrix. This is also proved by identification of a flawless transition zone between matrix and agloporite with SEM analysis. No defects connected with extreme thermal load were identified in these aggregates. Hence, behavior of newly developed composite system (with alternative raw materials currently produced in the Czech Republic) was examined and described at various methods of extreme thermal stress (high temperature and shock cooling).

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