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ÚSTAV TECHNOLOGIE STAVEBNÍCH HMOT A DÍLCŮ

BEHAVIOUR OF CEMENTITIOUS COMPOSITES EXPOSED TO HIGH TEMPERATURES

STUDIUM CHOVÁNÍ CEMENTOVÝCH KOMPOZITŮ PŘI PŮSOBENÍ VYSOKÝCH TEPLOT

DOCTORAL THESIS

DISERTAČNÍ PRÁCE

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ABSTRACT

Fire resistance is becoming increasingly important along with the development of new concrete types with high strength and dense structure with reduced porosity. Such concrete types are susceptible to fire spalling and extensive crack formation. At the moment, there are a limited number of methods for enhancement of fire resistance of existing structures, which could be applied in underground structures with restricted space and limited air exchange, such as tunnels, underground garages or nuclear powerplants.

This work is focused on the development of two methods, and both are dealing with porous structure modification. The first method is intentional heat treatment (IHT) method, suitable for the enhancement of fire resistance of existing structures. The second method emphasized the design of air-entrained concrete (AeA-FiResCrete) with the use of "new generation" air-entraining agents suitable for enhancement of fire resistance of newly designed concrete. Testing of compressive strength, porous structure modification was completed by the analysis of "moisture clog," which contributes to explosive spalling and extensive cracking. The efficiency of developing methods was verified during large-scale testing according to modified ISO834 (m-ISO) curve.

No extensive crack formation or explosive spalling was observed during the exposure period during the large-scale testing of slabs with the applied IHT method. The total thickness of the IHT method with configuration IHT200/2, composed of IHT zone and IHT transition zone, penetrated to the depth of 25,5 to 43,0 mm depending upon various concrete types. Moisture clog in AeA-FiResCrete was more significant than in the case of slabs with applied IHT method, and it could be concluded that the IHT method enhances fire resistance of concrete exposed to elevated temperatures without influencing its compressive strength and durability. Results from AeA-FiResCrete testing showed only a slight improvement of its fire resistance.

KEYWORDS

Fire resistance of concrete, intentional heat treatment (IHT) method, air-entrained concrete (AeA-FiResCrete), silica fume, polymer microspheres, porous structure, modified ISO 834 (m-ISO) curve.

ABSTRAKT

Trendem moderního stavitelství je časté používání vysokohodnotných a vysokopevnostních betonů. Spolu s vysokou pevností tyto betony obsahují malé množství vzduchových pórů, což může způsobit explosivní odpýskávání betonu a následný kolaps konstrukce. Dalším problémem jsou stávající konstrukce, které často nevyhovují současným zvýšeným požadavkům na požární bezpečnost. Využití existujících metod pro zvýšení požární odolnosti stávajících betonových konstrukcí, například nátěry a předsazené protipožární konstrukce, je limitované. Vývoj nových metod pro ochranu betonových konstrukcí je nezbytný, a to hlavně se zaměřením na aplikaci v tunelech, podzemních garážích a jaderných elektrárnách.

Tato práce se zabývá vývojem dvou metod pro zvýšení odolnosti betonů vůči působení vysokých teplot, se zaměřením na modifikaci pórové struktury. První metoda je záměrné předehřívání betonu (IHT method) pro zvýšení požární odolnosti stávajících betonových konstrukcí. Druhá metoda se zaměřuje na návrh provzdušněného betonu (AeA-FiResCrete) s vhodnými požárními vlastnostmi s použitím provzdušňujících přísad „nové generace“. Při experimentech byla kromě pevnosti a modifikaci pórové struktury sledována přítomnost tzv. vodní bariéry „moisture clog“. Účinnost vyvíjených metod byla ověřena testováním betonových desek dle modifikované teplotní křivky ISO 834 (m-ISO).

Desky ošetřené IHT metodou nevykazovaly při testování dle m-ISO křivky žádné nadměrné praskání ani explosivní odprýskávání. Celková tloušťka IHT metody skládající se z IHT zóny a IHT přechodné zóny byla v rozmezí 25,5 a 43,0 mm při konfiguraci IHT200/2. Při testování AeA-FiResCrete byla vodní bariéra výraznější než v případě desek s aplikovanou IHT metodou, a proto lze usuzovat, že IHT metoda přispívá ke zvýšení požární odolnosti, a to bez negativního vlivu na pevnost a trvanlivost betonu. Testované AeA-FiresCrete betony vykazovaly pouze nepatrné zlepšení odolnosti vůči působení vysokých teplot.

KLÍČOVÁ SLOVA

Požární odolnost betonu, metoda záměrného předehřívání betonu (IHT method), provzdušněný beton (AeA-FiResCrete), mikrosilika, polymerní mikrosféry, pórová struktura, modifikovaná ISO 834 (m-ISO) křivka.

CONTENT LIST

I. THE THEORY

1 FIRE EVENTS AND IMPORTANCE OF FIRE PROTECTION FEATURES

Throughout the years and decades, hundreds of smaller or larger scale fires have been recorded. Fire is one of the four elements and it is hard or sometimes even impossible to get it under control. Fire propagation depends on the materials present and local conditions. If the fire event takes place in open space, the type of flaming material, direction, and speed of wind play a major role. Access of firefighters is not restricted, and they can use various techniques such as water jets or sprinkle from the air with the use of cranes or planes. Other techniques for extinguishing a fire such as dry powder extinguishers and compressed air foam systems (CAFS) can be used in case of dealing with fire events which cannot be extinguished by water. When the fire occurs in an area where access is restricted, then the techniques and methods for extinguishing the fire are more complicated, and can lead to more rapid development of temperatures in the centre of fire. Fire spread in restricted areas such as closed underground car parks or road and rail tunnels depends on the quantity of inflammable materials in the area surrounding ignition of the fire and air velocity. Parameters of fire regarding temperature development and maximum temperature can cause significant damage to the structure. However, for evacuation and rescue of victims from the fire event the smoke is more severe, which is complicates not only evacuation but also visibility during the extinguishing of fire by firefighters, see Fig. 1 (1).

Fig. 1: (a) Structural damage caused by fire; (b) The smoke stratification in the initial moments of the fire (2, 3).

Another consequence of fire are damage of structure and its reparation or complete rebuild. Enhanced fire resistance of structure can contribute to lower reparation need. Fire safety in tunnels is improving and adjusting to the present situation concerning vehicles and their technological developments. In the past there were vehicles with either petrol or diesel engines, nowadays there are vehicles with gaseous fuels, electric and hybrid-electric engines, which are characterized by different fire scenario and requires different rescue operations.

2 METHODS AND ASSESSMENT OF FIRE RESISTANCE

Fire resistance of structures is an extremely important feature, and as most structures are constructed from concrete or steel, there are prescriptive Eurocodes for their design. There are three types of assessment of concrete structure fire resistance, namely fire testing, prescriptive methods and performance-based methods. The first method, fire testing, is the most critical and expensive but has high importance as the other two methods are based on results from this test. For fire testing are used standard fire curves presented in Fig. 2, and the most common is the standard time-temperature fire curve ISO 834.

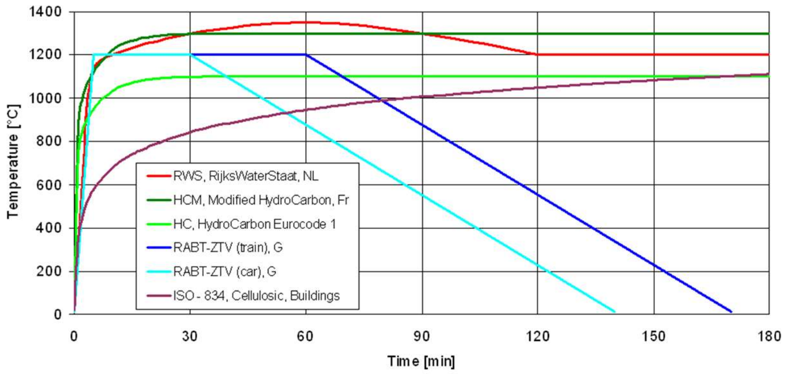


Fig. 2: Fire curves used for testing of building materials (4).

3 CONCRETE IN ELEVATED TEMPERATURES

Fire resistance of concrete can be rapidly improved by an appropriate selection of input materials and concrete design. Emphases are given to materials with higher thermal stability. Unfortunately, such materials are not available in all locations, and the quality of input materials is frequently compromised, and locally available materials are used. One concern is the selection of individual input materials, but the overall design and interaction of individual input materials under fire load is another issue. Additionally, load stresses, heating regimes, the shape of elements, and other parameters can cause the failure of structure build from well-designed concrete mix with suitable input materials.

As is well-known, concrete is composed of the cement matrix, aggregates, and air pores. Those three main components have various behaviour under thermal load, and their interaction defines the fire resistance of concrete. Apart from input materials and their behaviour, the role and influence of the porous system and the interfacial transition zone (ITZ), which is located between the cement matrix and individual grains of aggregates, plays important role. Nowadays, it is common to use various types of dispersed fibres or admixtures to modify the concrete mix and improve particular properties. A list of rules, which are essential for the design of concrete with improved fire resistance is given for demonstration of complexity of fore resistance of concrete.

There are many influencing factors which have to be taken into account during the evaluation and design of concrete exposed to elevated temperatures. Behaviour of individual components might not be severe during the fire, but their mismatch behaviour is causing degradation and complete disruption of concrete. To conclude, in this chapter several significant influencing factors will be listed and marked as a benefit (+) or disadvantage (-) for concrete behaviour in elevated temperatures.

- Cement paste expands up to 250°C, and shrinks when exposed to higher temperatures.
- Most aggregates are characterised by expansion, which introduce thermal stresses in ITZ.
- Most of the aggregates are stable up to 500°C, and then the scale of deterioration is dependent on their chemical and mineral composition.
- ITZ is the weakest area in the concrete, and pronounced as the most common reason for strength properties degradation and crack location (5).
- ITZ is the most exposed area and its lower tensile strength due to more porous structure composed predominantly by larger crystals of Ettringite and calcium hydroxide.

\+ Resulting from higher porosity and the presence of capillary pores in ITZ, it possibly retains less free or adsorbed water and can serve as a transportation channel for migrating water or vapor.

- Concrete with the greater maximum particle size aggregates is more susceptible to the formation of larger cracks. This finding could be connected to the coefficient of thermal expansion of aggregates (6, 7).
- Explosive spalling is more likely to occur in concrete types with reduced porosity, particularly the absence of capillary pores, such as hight-performance concrete (HPC), self-compacting concrete (SCC) or other new types.
- Reduced porosity and absence of capillary pores create an opportunity for high water vapour pressure build-up and more significant crack formation or spalling of concrete pieces.
- Densifying of microstructure by supplementary cementitious materials (SCM) lead to “discontinuity” of capillary pores, and additional formation of (calcium-silicate-hydrate) CSH gel in gel pores, which are predominantly fully saturated and release of water from gel pores and CSH interlayers is slower and can result in higher pressure development.

+ The addition of pozzolanic active SCM reduces calcium hydroxide content as it is consumed during the reaction. Such a reduction of calcium hydroxide results in degradation of cement paste at around 400°C when dehydroxylation of calcium hydroxide ($Ca(OH)_2$)takes place.

+ The addition of polypropylene fibres (PP-fibres) increases the amount of capillary pores (artificial capillary pores) or behaves like a network of evenly distributed cracks (8). First, the pressure-induced tangential space (PITS) serves for vapour migration, and after the melting of PPF at 130 or 150°C, larger space for moisture migration is provided (9, 10).

- Actual moisture content in concrete structure and relative humidity (RH) of surrounding ambient (tunnels approx. 75% RH) have the most influence on concrete behaviour, as the water evaporates first before all mineralogical and chemical reactions. If the relative humidity of ambient is below 50%, the free water is present predominantly in CSH interlayers, gel pores and adsorbed on walls of capillary pores. Water molecules can migrate towards the hot and cold regions without the formation of moisture clog and reducing risk of fire spalling.

+/- The water/cement or water/binder ratio has a significant influence on porous structure and properties in general. If the w/c ratio is high, more capillary pores are formed, and concrete has a better ability to release the water, and prevent the development of high vapour pressure. Unfortunately, the strength and durability of concrete are highly reduced by high w/c ratio. If the w/c ratio is low, the denser structure is formed, and mobility of water or vapour restricted, which leads to crack formation and spalling.

- The rough surface of aggregates forms stronger ITZ, which can resist higher vapour pressure. The probability of explosive spalling could be reduced or delayed (11).
- The thermal conductivity of concrete is closely related to aggregate type and porosity. If the thermal conductivity is high, stresses are evenly distributed over the whole bulk of concrete element, and might contribute to better fire resistance. On the contrary, the low value of thermal conductivity can create areas with different actual properties and give rise to uneven thermal strengths in the concrete mass. Different rules apply if the concrete element is reinforced, then slow thermal conductivity is favourable.

Behaviour of concrete is also highly depended on the properties of elevated temperature development such as (i) length of exposure, speed of temperature gain, maximum temperature, speed of cooling, (ii) one side heating (tunnel lining), one point heating (centre of fire), full exposure (columns), (iii) rate, level and direction of loading, no loading. As can be seen, there are too many factors involved, and therefore, fire resistance evaluation, prediction and design are highly complex and sometimes unpredictable.

4 CONCRETE SPALLING IN ELEVATED TEMPERATURES

Spalling of concrete was for the first time observed and recorded in 1854 (12), and probably the first description of the spalling behaviour of concrete exposed to elevated temperatures was published by Barret (13). During those 167 years, which have passed since the first description of spalling phenomena, several theories that explain the cause and mechanism of concrete spalling were introduced. Explosive spalling was not frequently observed in the past, thanks to the higher porosity of concrete. With the introduction and use of plasticizers, the water/cement ratio was reduced and led to a reduction of the porous system. Another aspect that contributes to the reduction and modification of porous structure is the usage of supplementary cementitious materials with a high surface area.

One of theories referring to water/vapour migration in concrete is termed "moisture clog" theory (14). Water migration in a concrete structure is caused by temperature and pressure differences when the concrete structure is exposed to elevated temperatures from one side. Water evaporates on the heated side and also migrates towards the colder non-heated surface to even out the temperature and pressure. Migration of water toward the colder surface leads to full saturation and condensation, which results in the formation of "moisture clog", schematically explained in Fig. 3. When this moisture clog is formed, further moisture transport inwards to the colder region is slowed down or completely stops, which leads to a rapid build-up of pore pressure. When this pressure exceeds the tensile strength of concrete, spalling and explosive spalling occurs (15, 16).

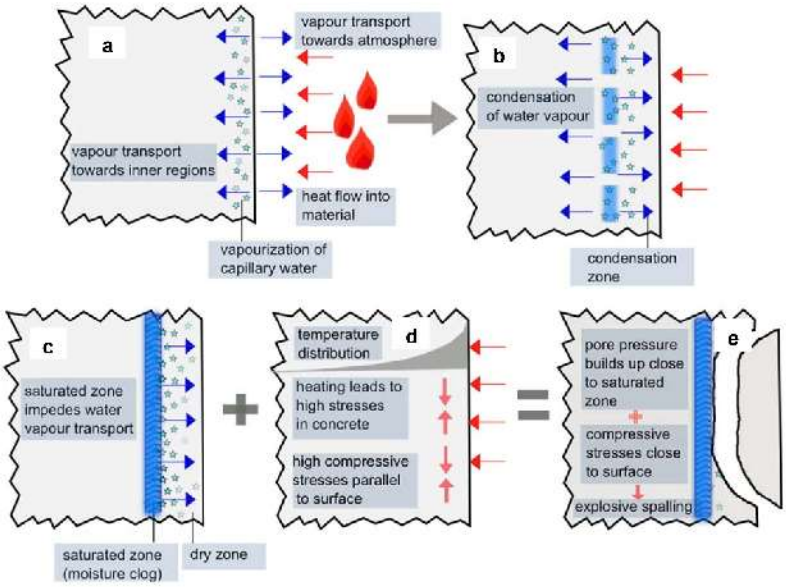


Fig. 3: Fire curves used for testing of building materials (17).

AIM OF DISSERTATION THESIS

Fire resistance is one of the less-discussed properties of concrete, and yet crucial when it comes to closed underground structures or structures with increased fire risk level. Fire can cause extensive damages to structures, and concrete degradation and disintegration can result in a complete collapse of the structure. Problematic areas concerning behaviour of individual components in concrete exposed to elevated temperatures, such as cement paste, porous structure and aggregates, was explained in detail in the theory part of the thesis. Gained knowledge is going to be applied during the development of a new and innovative method for fire resistance of existing concrete structures based on a modification of the porous structure of the surface layer by application of heat. This method, entitled intentional heat treatment (IHT) method will serve predominantly for fire protection of existing structures with restricted space or ventilation possibilities. The main target is to describe in detail the principle of the IHT method using chemical and porous structure analysis and verification of its efficiency during large-scale laboratory testing. In parallel, the contribution of traditional and "new generation" air-entraining agents, and their potential to be used for the improvement of fire resistance of newly designed concrete (AeA-FiResCrete) will be developed and tested. Finally, both methods will be compared, and their efficiency evaluated.

II. EXPERIMENTAL PART

Experimental work conducted within this research was performed in different locations, the Czech Republic at the laboratory of Brno University of Technology and AdMaS center, in Germany, laboratory located at Hochschule Wismar, University of Applied Science, Technology, Business and Design, and the last location was Iceland, with the help of Reykjavik University and Innovation Center Iceland. Various test facilities were available for performing the testing, and I am very grateful for all the support I got from academic stuff and laboratory assistants at individual institutes. The experimental part is divided into 4 chapters.

5 CHAPTER: DESCRIPTION OF DEVELOPING METHODS

In chapter 5 will be described two methods for fire resistance of concrete structures, which are the subject of this PhD thesis. The first method is focused on the modification of pores structure of the surface layer. The second method is the usage of standard and "new generation" AeA to intentionally form an extensive pore structure, which can serve as a transporting system for migrating water and vapour. Research focused on the application of basalt micro and macro fibres as a potential solution for fire resistance enhancement was performed, and published as scholar articles within conferences during the PhD studies.

5.1 Description of the IHT method

The main trigger for the development of this new method for fire protection of existing structures is extensive tunnel lining testing in Germany (18). Standards have imposed a new requirement in terms of the level of fire resistance and imposed new limits, which should be met even by existing tunnel structures. Extensive tunnel lining testing took place and results from the testing were not always satisfactory, and therefore we focused on the development of a new method that could be applied on tunnel lining without space occupation.

The new fire protection method which is going to be described and verified within this PhD thesis is based on intentional preheating of the surface layer of the concrete structure which we expect to lead to the creation of a suitable porous structure that will allow transport of water and vapour in the event of fire, and delay or entirely omit explosive spalling. Two main benefits of the IHT method are (i) no requirements for space in front of the existing structure except during the application procedure, and (ii) IHT method does not emit harmful and poisoning gasses.

The main area of application of the IHT method are locations with limited ventilation possibilities and flue gas exhaust such as tunnels and underground garages. Newly built underground structures are designed and equipped with a high-tech fire alarm and protection systems to meet the regulations, but those regulations should also be met by existing tunnels, which is more challenging and technically demanding. Fire in a closed place can have devastating consequences not only at the centre of the fire but also on surrounding structures. Most dangerous are tunnels in big cities where there are structures above the tunnels, and if the tunnel collapses, not only people in the tunnel are endangered but also in the area above the tunnel.

There is a direct dependency between rising exposure temperature and porous pressure. When the pressure in the pores exceeds the tensile strength of pore walls, cracks are created. Formed cracks connect the pores in cement paste and generate an interconnected porous structure. The problem is that these cracks have a direct effect on the strength of the whole concrete element. If the cracks which are caused by extensive vapour pressure are formed slowly, and in a small scale may not necessarily have an extensive influence on the strength and integrity of the whole element. Also, if the cracks are created only in the surface layer, the strength can remain unchanged. The creation of cracks and consequently connected porous structure could allow the transfer of a greater amount of vapour and thus prevent explosive spalling. Unfortunately, there is one more phenomenon that should be taken into account, and that is the penetration of chemical agents into the concrete structure which could accelerate degradation of concrete. Therefore, the ratio of interconnection and permeability should be carefully balanced.

One solution to create a suitable interconnected porous structure in the surface layer of the concrete element is to heat it up until the desirable porous structure is created. This layer permits vapour transfer from the concrete mass but restricts penetration of chemical agents from surrounding ambient into concrete mass. The speed of heat transfer, and respective, coefficient of thermal heat conductivity, is the decisive parameter for IHT method configuration. This raises the first question for developing of the IHT method: At which **maximum temperature** is the best pores structure created? The second question considers the thickness of the surface layer with a modified porous structure. **How long** should the thermal exposure be held to create the best performing layer in a suitable thickness? The third and last question, which defines the parameters of IHT method is: **How fast** can the heating be applied to enlarge the porous structure without causing damage, and still remain time efficient during the application? The main parameters of IHT method are speed of heating, maximum exposure temperature and duration of exposure to a maximum selected temperature.

There are many influencing factors, that need to be taken into consideration while designing the ITH method. Those factors may lead towards the variation of IHT method configuration based on individual concrete types, structures and conditions.

5.2 AeA-FiResCrete: Concrete mix design modified by air-entraining agents (AeA)

AeA are primarily added to enhance freeze-thaw resistance by modification of porous structure, such as spacing factor, the specific surface area of air voids and void frequency. The pore size provided by AeA should be approximately in a range from 10 to 300 μm, as each different source states slightly different values. This pore diameter is also partly desirable for fire resistance, and can contribute to easier water and vapour migration through the cement paste in case of a fire event. The air content produced by AeA can strongly influence the compressive strength of concrete if ‘overdosed’, and therefore the number of pores contributing for better freeze-thaw resistance should be carefully balanced with other properties. Results from analysis of air voids characteristics in hardened concrete can provide useful data and help to increase understanding of concrete porous structure. Three different types of AeA were used for the development of AeA-FiResCrete.

6 CHAPTER: DESIGN OF MIXES AND LABORATORY WORK DESCRIPTION

In this chapter selection of the input materials, their individual testing and validation of their suitability will be described as a first, and followed by a description of mix design, used tests, special test setup for large scale testing and heating regimes. Special emphasis will be given to samples with incorporated thermocouples for temperature development in concrete specimens during the fire testing.

The selection of materials used during the laboratory work was quite challenging as the work was carried out at three different locations as mentioned earlier. Basalt aggregates from Iceland and Granite aggregates from Norway were used for concrete mixes, and for mortar mixes was used CEN Standard sand EN 196-1. Other components such as cement (CEM I 42,5N-SR5 from Aalborg, DK and CEM II/B-M 42,5 R from Norcem, NO and CEM II/A-LL 42,5R), supplementary cementitious materials (silica fume-SF) and admixtures (air-entraining agent, superplasticizer) were matched without any significant complications.

Properties of input materials such as water absorption, density, particle size distribution, resistance to fragmentation of coarse aggregates (LA test) and properties of cement mortar were performed. Fresh concrete was examined to workability, air content, and density. Prepared samples were cured in a climate chamber with 20 ± 1°C and 100% RH, and afterward tested for compressive and flexural strength, density, air void characteristic, freeze-thaw resistance. A mercury intrusion porosimetry (MIP), thermal analysis (TGA-DTA) and X-ray diffraction analyses (XRD) were used for cement paste chemical composition and porous structure evaluation.

Large-scale testing of one-side heated uniaxially loaded slabs was performed with the use of an electric oven with an opening from the top was used. The support frame was built with height-adjustable feet to eliminate the overloading of the oven by slabs which weighed approx. 55 kg. The loading frame for the sample was made out of two steel profile HEB 100 fixed together on both ends by two threaded rods (Fig. 4a), and lean on four points of the support frame, as displayed in Fig. 4b.

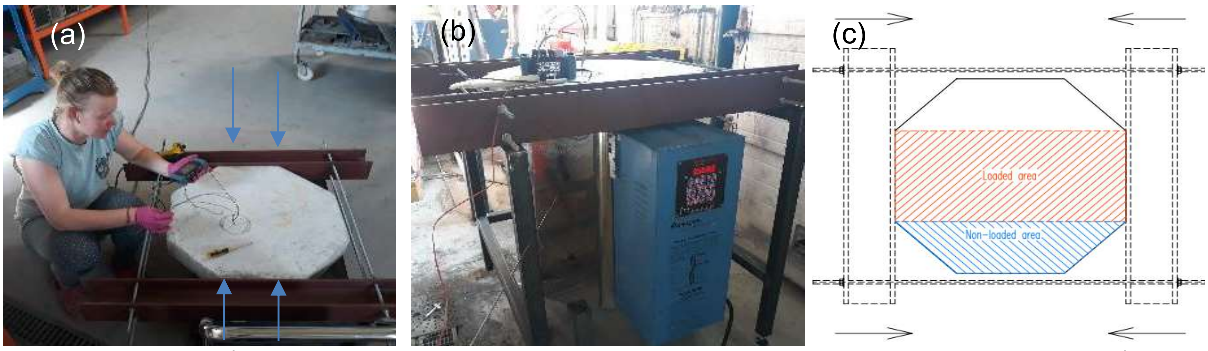


Fig. 4: Test setup for large scale one-side heated uniaxially loaded slabs: a) Uniaxial loading steel profile HEB 100 with prestressed treaded rods, assembling thermocouples and plugging in thermometer log; b) Testing slab placed on load-bearing frame and connected to thermal log; (c) Schematic drowing of the test slab: The uni-axial loading is applied in central area due to the shape of tested slabs.

Samples were tested either according to the heating regime with defined temperature ramp, maximum temperature, length of exposure and cooling, or according to modified time-temperature ISO 834 curve. All fire tests were performed in an electric oven, which is highly suitable for defined heating regimes, but has certain limitations when it comes to rapid heat development required by ISO 834, approx. 570°C in the first 5 minutes. Nine types of heating regimes were used for the IHT method development. Standard time-temperature curve ISO 834 was not possible to obtain with available equipment, and therefore samples for fire testing were loaded in the fastest possible rate. Therefore, the term ‘modified ISO 834’ (m-ISO) curve will be used throughout this work when referring to the heating regime.

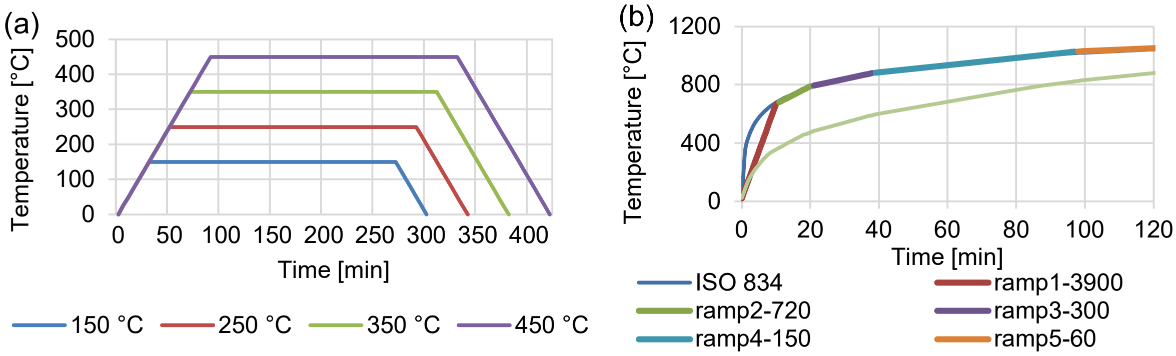


Fig. 5: (a) Heating regimes for development of IHT method 1st series; (b) Modelling of ISO 834 for an electric oven set up, and modified ISO 834 for testing of slabs instead of lid.

Several different series of concrete types have been prepared and tested within this research. The main approach of mix design is to keep constant all properties besides those that are investigated. For the processing of the mix design, the program ComPose5, developed at the Innovation Center Iceland, was used, and it is able to take into account water absorption and moisture content of aggregates. Icelandic aggregates have high water absorption, and it is necessary to consider this parameter during the calculation of mix proportions.

Mix design for IHT method

For the development of the IHT method, 4 various series of concrete were prepared with an emphasis on various types of cement paste, aggregate types and compressive strength to cover most common concrete types used currently and in the past 50 to 100 years. Also, fire spalling is a phenomenon more common for new types of concrete, for instance, HPC or UHPC, and therefore those types were involved during the development of the IHT method.

- IHT method 1st series: One type of high strength concrete class C65/80 with unknown mix design. Input materials were CEM II/A-LL 42,5 R, superplasticizer, and natural sand fraction 0-4 mm, coarse granite aggregates fraction 8-16 mm and 11-22 mm.
- IHT method 2nd series: One type of concrete in strength class C45/55 with the same mix design as used in the 4th series for IHT method development marked as 7NO. Concrete tested in this series is composed of CEM I-42,5 N-SR5, superplasticizer and Granite_NO aggregates.
- IHT method 3rd series consists of 3 different mortars with various cement types and SCM. Samples were prepared in the same way as for standard mortar testing according to EN196-1, and the only variable is binder type: CEM I-42,5 N-SR5, CEM II/B-M 42,5 R and CEM I 42,5 N + 25% SF. The content of sand and water was kept constant.
- IHT method 4th series consists of 4 concrete types, 3 cement types, namely CEM I-42,5 N-SR5, CEM II/B-M 42,5 R and CEM I 42,5 N + 6% SF, superplasticizer, Basalt_IS and Granite_NO aggregates fraction 0-8 mm, 8-16 mm and 8-22 mm.

Mix design for the development of AeA-FiResCrete

Three types of AeA, Icelandic basalt aggregates, CEM I-42,5 N-SR5 and plasticiser were used for the development of AeA-FiResCrete; standard AeA based on surface tension from BASF, and two “new generation AeA” based on the chemical reaction from Sika, and micro hollow spheres polymer-based AeA from MC-Bauchemie.

Fig. 6: Photo documentation of air-entraining agents: (a) MasterAir 11; (b) Centrament Airpolymer; (c) SikaControl AER-200 P.

7 CHAPTER: PRACTICAL DEVELOPMENT OF THE IHT METHOD

The development of the IHT method started in Germany under the supervision of Ulrich Diederichs. The main parameters to be defined throughout the test program are the speed of heating, maximum exposure temperature, and length of exposure to selected maximum temperature. The intentional heat treatment of the surface layer is supposed to modify the porous structure of the surface layer, which is predominantly composed of cement paste and fine aggregates. Concrete is non-homogenous material, and thermal exposure of various sample sizes and concrete types can result in different performance, it is highly challenging to define one configuration for the IHT method applicable to all element shapes and concrete types. This work attempts to verify if our innovative method will improve fire resistance of concrete. The database of configurations for individual concrete types could be subject to the further development of the IHT method. Development of the IHT method was divided into 4 individual parts, The marking system used during the development of the IHT method will be "IHT exposure temperature/duration of exposure to selected maximum temperature", an example would be IHT200/2. If it will be referred in general to application of the IHT method, the abbreviation IHT_T/t will be used.

7.1 The 1st series: Various temperature analysis on high strength concrete

Activities in this chapter were the first attempt to verify the assumption that the intentional heating of the concrete element would modify the porous structure of the surface layer beneficially for fire resistance. Test specimens for thermal treatment were cubes with an edge of approximately 73 mm. Reference samples were tested without any thermal exposure, and the rest of the samples were exposed to heating regimes with maximum temperature 150, 250, 350 and 450°C. The temperature ramp 5°C/min (300°C/hour) was selected based on the reviewed literature. The length of exposure to maximum temperature was 4 hours with the aim to penetration of the maximum temperature in the whole mass of tested samples to gain representative results for compressive strength change. Natural cooling back to ambient temperature follows exposure to the selected temperature. The total length of the IHT method will play a significant role for industrial use, and the speed of reaching selected exposure temperature and its duration is critical for the thickness of surface layer modified by IHT method.

Half of the thermally loaded samples were directly tested, and the second half was submerged in water for 28 days. Water storage (WS) after IHT_T/t is meant to represent relative humidity to which the concrete structure with IHT_T/t would be exposed in reality. The same tests were performed before thermal treatment, after IHT_T/t, and after IHT_T/t&WS. Monitored properties were weight, compressive strength, porous structure analysed by MIP, and identification and quantification of chemical composition by TGA-DTA. Samples for MIP and TGA-DTA were taken from the surface layer and vacuum dried before testing.

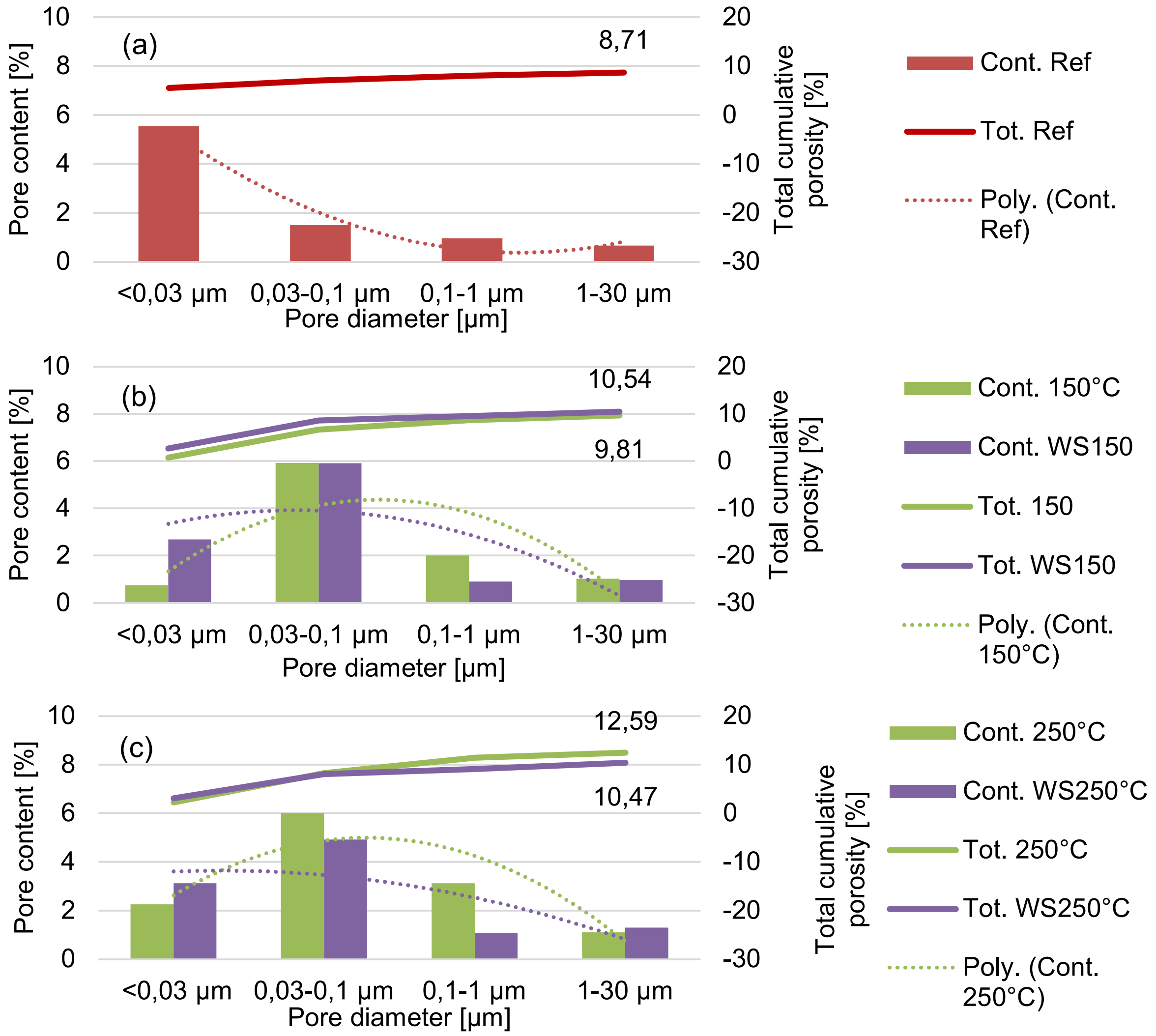
Compressive strength was reduced by thermal exposure, which is most likely caused by the formation of micro-cracks as a result of free water evaporation and chemical changes of Ettringite, CSH gels, and eventually $Ca(OH)_2$ in the case of IHT450/4. WS caused further compressive strength reduction of all samples, besides the IHT450/4, where a gain of strength by 22,3% was recorded. Regain of strength due to water storage might be caused by re-formation of Ettringite from the reaction of water with Bassanite and/or γ-anhydrite (19, 20), and additional formation of CSH gel (21, 22).

Results from MIP were processed for individual exposure temperatures, water storage, and pore size. Pores were divided into five groups; gel pore <0,03 µm; capillary pores in three segments, 0,03 to 0,1 µm; 0,1 to 1 µm and 1 µm to 30 µm, and the bigger macropores belong to the last group with a diameter greater than 30 µm. Calculation of individual pore size share in total porosity is processed in connection to the total porosity of individual samples,

The porosity of Ref sample without exposure to elevated temperature is characterised by a high content of gel pores, 62,3% of total porosity, with a diameter below 0,03 µm. Tested concrete was HSC with a dense porous structure. The measured content of capillary pores is very low, which contributes to higher compressive strength and durability, but increases the risk of fire spalling in case of fire exposure.

Detail analyses of data obtained from MIP showed increasing content of pores with diameter from 0,03 µm to 0,1 µm and 0,1 µm to 1 µm due to rising exposure temperature. This fact also correlates with total measured porosity and decrease the presence of Ettirngite and CSH gel detected by TGA-DTA. Total porosity, and especially capillary pores in the same range, from 0,03 µm to 0,1 µm and 0,1 µm to 1 µm, were reduced by WS in almost all cases. The change of total porosity in the sample IHT150/4 increased by 7,5%, which is a negligible change and is something that can be discussed. Other samples IHT250/4, IHT350/4, IHT450/4 recorded a reduction in porosity by 16,8%, 41,9% and 66,1%. The reduction of porosity resulting from the WS might not be suitable for the IHT_T/t and negatively influence its performance ability.

Another proof of the formation of new hydration products such as Ettringite and CSH gel can be an increase of gel pores, which are an indispensable part of CSH gel formation. Increase of gel pores with a diameter under 0,03 µm was not recorded in the case of the sample IHT450/4, which might be caused by the formation of denser CSH gel or other products thanks to additional free lime (CaO) from the decomposition of $Ca(OH)_2$ initiated at 390°C.



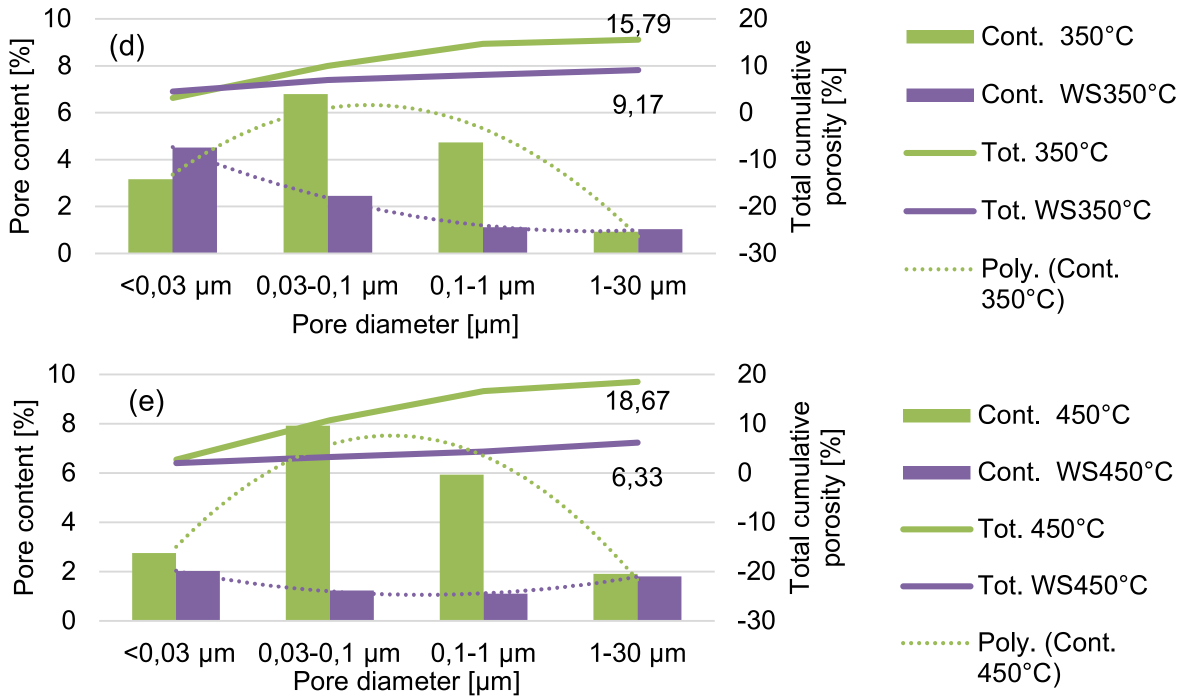


Fig. 7: Results from MIP of individual pore size share correlated to total measured porosity. Figure combines pore content and total cumulative pore content of individual exposure temperatures (a) Ref with no heating; (b) 150°C; (c) 250°C; (d) 350°C; (e) 450°C and samples after IHT_T/t&WS.

Thermal analyses are entirely appropriate for the study of chemical compound changes due to thermal exposure. Reactions between 20 and 450°C are (i) evaporation of remaining free or physically bonded water between 20 and 70°C; (ii) dehydration of Ettringite with hydration products water, Bassanite and γ-anhydrite between 70 and 120°C; (iii) dehydration of CSH gel with hydration products water and β-C_2S (Larnite) between 120 and 200°C; (iv) continuous hydration of CSH gel and C_2AS with hydration products water, β-C2S (Larnite) and Gehlenite (22) between 200 and 390°C; (v) dehydroxylation of $Ca(OH)_2$ with hydration products water and CaO (lime) between 390 and 475°C. It must be taken into consideration that processes are overlapping, and it is not fully possible to totally decouple them. Measured weight loss during the TGA-DTA analysis was higher in the case of Ref sample and samples after TE & WS, while weight loss of TE samples was decreasing with increasing exposure temperature. Results from TGA-DTA confirm the presumption that all earlier described processes are based on the release of bonded water and its evaporation. Increasing weight loss of IHT_T/t&WS samples follows the theory that exposure to high RH initiates rehydration and formation of new or recovered hydration products.

7.1.1 Conclusion of the 1st series

The decrease of compressive strength due to water storage in the case of samples IHT150/4, IHT250/4 and IHT350/4 is the extensive formation of new, large crystals, which cause severe damage to cement paste microstructure.

The Ref sample, which was not exposed to elevated temperature, contained a large share of gel pores under 0,03 µm, and a low number of capillary pores, which was inversed by exposure to all test temperatures. The content of capillary pores increased, and number of gel pores reduced. Selected exposure temperatures changed porosity of concrete and, in all cases, increased the number of capillary pores. However, porosity was further modified in the case of IHT350/4&WS and IHT450/4&WS where exposure to high humidity was simulated by water storage of samples for 28 days. Exposure to high humidity caused the formation of hydration products in shaped capillary pores with a diameter from 0,03 to 1 µm. The porosity of samples IHT150/4 and IHT250/4 was positively extended by IHT_T/t, and only slightly modified by consequent water storage, which is substantial for the IHT method. Further development of the IHT method will be focused on exposure temperatures between 150°C and 250°C.

7.2 The 2nd series: Various temperature analysis on normal strength concrete

The 2nd series will be focused on modification of porous structure and chemical composition of concrete with compressive strength 66,8 MPa and porosity 2,3% in the hardened state, due to exposure to 150°C, 200°C and 250°C with 2 hours exposure to a selected temperature. The speed of heating 5°C/min and natural cooling remains the same. The total length of IHT150/2 is 3,7 hours, IHT200/2 is 4,2 hours and IHT250/2 is 5,7 hours.

The total porosity of samples with applied IHT_T/t slightly increased. Evaporation of residual water from CSH gel interlayers, and also their partial recrystallisation caused a reduction of pores by 2,2% in average, and extend the content of capillary pores. The trend of higher formation of capillary pores with a diameter between 0,03 and 0,1 μm in sample IHT200/2, while exposure to 250°C contributed to the greater formation of larger capillary pores with diameter 0,1 to 1 μm, is the same as in the 1st series IHT development.

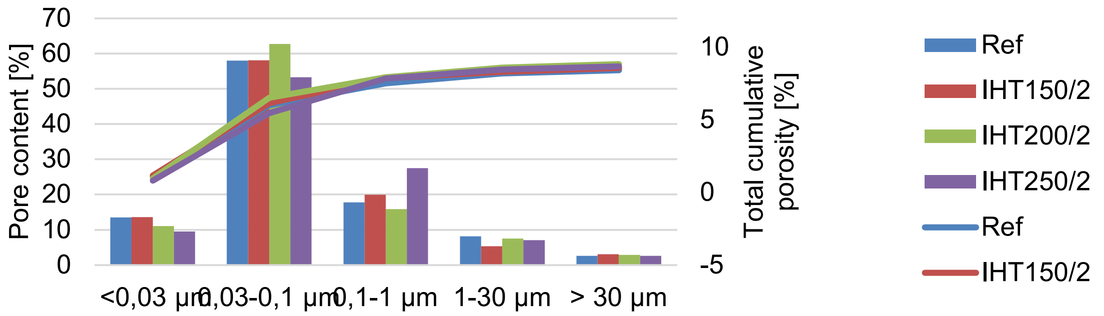


Fig. 8: Graphical presentation of results processed as the total porosity is equal to 100%, presented in ***Error! Reference source not found.****.*

Configuration of March! software used for XRD analysis contained minerals before and after their crystalline structure change due to thermal exposure, (i) dehydration of **Ettringite** with hydration product **Bassanite** between 70 and 120°C; (ii) crystalline phase of CSH gel – **Tobermorite** with hydration product **β-C_2S (Larnite)** between 120 and 200°C; (iii) partial dehydration of **Brownmillerite** and formation of **Gehlenite**. Formation and recrystallisation of other phases are not excluded but the selected 6 minerals are considered as the most significant ones. The content of those 6 minerals was considered as 100%.

The Ref sample had a higher content of Ettringite, Tobermorite and Brownmillerite as expected, and the quantity was reduced due to the application of the IHT method. Crystallisation of amorphous CSH gel is signified by a rapid increase of Larnite, from 2,3% to 29,9-33,1-33,1%, respectively. Formed crystals of Larnite probably creates more porous microstructure than CSH gel, which correlates with the results from MIP. Furthermore, formed crystals might be stronger, which would partly explain the compressive strength gain. The selected technique for evaluation of diffractograms is not representative for the samples as a unit, as the main purpose was a comparison of the Ref sample and samples with applied IHT150/2, IHT 200/2 and IHT250/2.

7.2.1 Conclusion of the 2nd series

Application of three different configurations of the IHT method, namely IHT150/2, IHT200/2 and IHT250/2 on normal strength concrete caused a slight increase of compressive strength, and a less significant change of porous microstructure than in the case of HSC tested in the 1st series. The content of capillary pores slightly increases in the case of samples IHT200/2 and IHT250/2, while IHT150/2 remains almost unchanged. The IHT method modifies the pores up to diameter 30 μm, and does not influence freeze-thaw resistance as suitable pore diameter for freeze-thaw resistance is between 10 to 100 μm. It seems that a negligible overlap of pore diameter suitable for fire resistance and freeze-thaw resistance, 10-30 μ, does not play a significant role.

The duration of thermal exposure to individual maximum temperatures seems appropriate as the porous structure of the surface layer was modified, but the strength of the sample has not decreased. The length of the IHT method is relatively long, thanks to natural cooling. It would be suitable to investigate this area further if faster cooling can be performed and not cause an increase of microcracks formation or other defects of surface layer microstructure. Exposure temperature 200°C and 250°C showed promising results, and 2 hours of thermal exposure also seems favourable.

7.3 The 3rd series: Various temperature and heating length analysis on various binders

The 3rd series will be focused on two main parameters. The first is testing of the IHT method configurations IHT200/1, IHT200/2, IHT250/1 and IHT250/2 with heating ramp 5°C/min and natural cooling. The second approach is the evaluation of various types of binders, namely CEM I-42,5 N-SR5, CEM II/B-M 42,5R (sold as Standard FA-sement in Iceland) and CEM I-42,5 N-SR5 with 25% replacement of cement by silica fume (SF), with and without applied IHT method thermally loaded according to m-ISO curve. The influence of the IHT method on various binders will be controlled by testing of weight change, flexural and compressive strength, and visual evaluation of cracks and discolouration using an optical microscope.

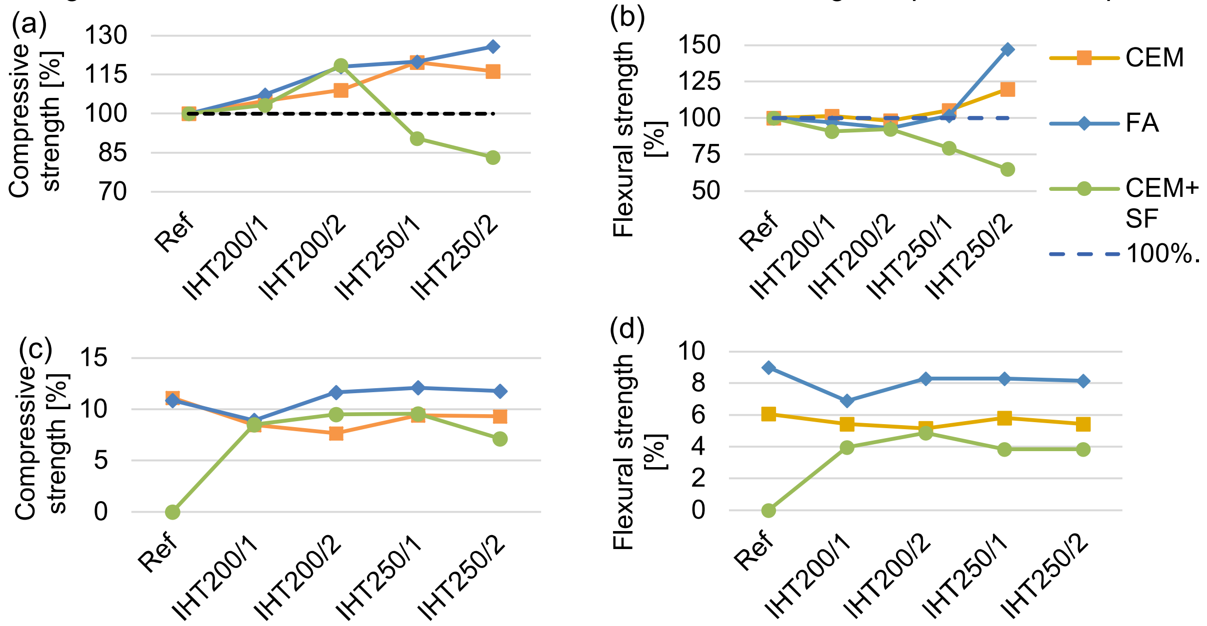


Fig. 9: Graphical presentation of the compressive and flexural strength change due to (a, c) application of IHT_T/t expressed as a percentage change in relation to Ref sample; (b,d) application of IHT_T/t and thermal exposure according to m-ISO heating regime expressed as a percentage change in relation to Ref sample without thermal exposure according to m-ISO curve.

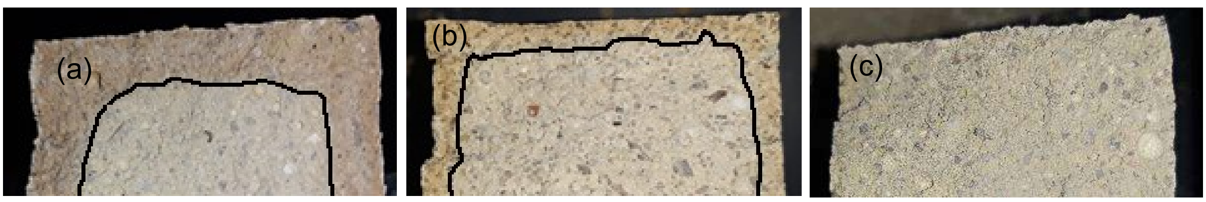


Fig. 10: Photo documentation of samples after thermal exposure according to m-ISO: (a) Ref sample; (b) IHT200/1; (c) IHT200/2.

The samples without IHT_T/t exposed according to m-ISO curve were discoloured on the surface area, Fig. 10a while the discolouration was weakened with an increase of heating duration length and maximum exposure temperature of IHT method., Fig. 10b and 10c.

7.3.1 Conclusion of the 3rd series

Application of IHT_T/t increased compressive strength up to 25,8% and flexural strength up to 47,3% in the case of all binders and IHT method configurations. The only exception were samples CEM+SF with applied IHT250/1 and IHT250/2, where compressive strength decreased by 16,7% and flexural strength by 35%. The strength loss was caused by microcracks and extensive evaporation of chemically bonded water, which had difficulties in migration through the dense mass of CEM+SF when exposed to 250°C. Generally, the loss of flexural and compressive strength due to exposure to m-ISO thermal regime was extremely high, and besides the binder type, the influence of used quartz sand must be taken into consideration.

The IHT200/2 and IHT250/1 had the same weight loss, which could signify the same penetration depth of IHT method with different speed. As was mentioned in the description of the IHT method, the variation could be needed for different types of binders or aggregates. Results from the 3rd series showed that the influence of the IHT method on CEM is not sufficiently contributing to preservation of the strength, but FA and CEM+SF binders registered an increase of strength, and, indeed, complete fragmentation of CEM+SF samples was eliminated.

Based on the results from this chapter, it was decided that the final configuration of IHT for testing of various concrete types and large-scale testing of slabs will be performed with IHT200/2.

7.4 The 4th series: Application of IHT200/2 on various types of concrete

The 4th and the final series of the IHT method development is going to verify the selected IHT method configuration, heating ramp 5°C/min (300°C/hour), exposure temperature 200°C held for 2 hours and finished by natural cooling – IHT200/2. Compressive strength change and weight loss will be tested on two different sizes of samples, cubes with an edge of 50 or 100 mm and 4 types of concrete. Three with Basalt_IS aggregates and various binders: 4CEM with CEM I-42,5 N-SR5 from Aalborg cement Denmark; 5FA with CEM II/B-M 42,5R (sold as a Standard FA-sement in Iceland) from Norcem Norway; 6SF with CEM I-42,5 N-SR5 with 6% replacement of cement by silica fume (SF). The fourth concrete, 7NO, contains CEM I-42,5 N-SR5 from Aalborg cement Denmark and Granite_NO aggregates.

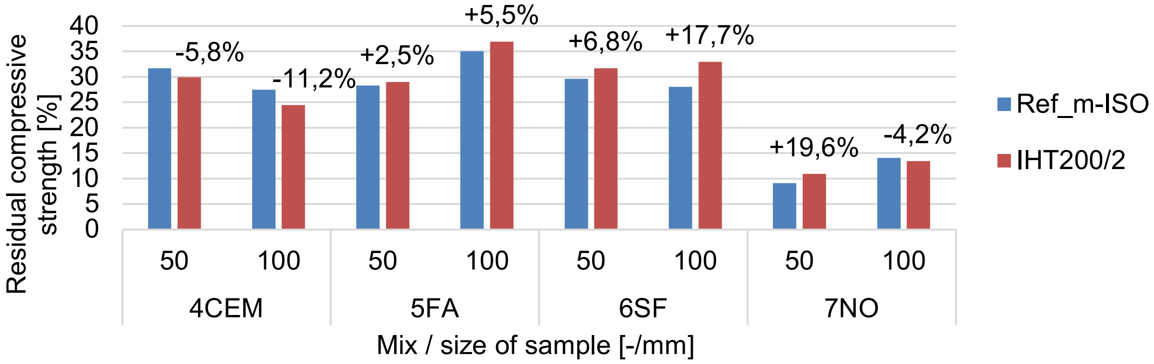


Fig. 11: The compressive strength reduction due to thermal exposure according to m-ISO curve expressed as a percentage of Ref samples without thermal exposure, and the difference between samples with and without application of IHT200/2.

The application of IHT200/2 improved compressive strength in the cases of all samples 5FA and 6SF regardless of the shape, but lower compressive strength was measured in the case of 4CEM. Samples 7NO preserved more strength when a smaller sample, 50, was tested.

7.4.1 Conclusion of the 4th series

Size of the samples and its origin (cut or individually cast) is more significant in the case of mixes 4CEM and 5FA and may be a result of lower heat transfer in concrete, reduced depth of heat penetration, and lower water release from a structure. There was no significant influence of shape and origin on weight loss in the case of the other two tested mixes, 6SF and 7NO, which might be positive when applied in industrial scale on whole structural elements.

Application of IHT200/2 increased residual compressive strength up to 17,7% in case of mix 6SF sample 100, and the difference between residual strength of mix 7NO sample 50 with and without application of IHT200/2 was 19,6%. However, the sample 100 of mix 7NO recorded impairment due to application of IHT200/2 as the influence of Granite_NO aggregates expansion was greater than modification of porous structure in cement paste. Slide improvement was also recorded in the case of mix 5FA by 2,5 and 5,5%. The only mix where the residual strength was higher without application of IHT200/2 was 4CEM, and results from the 3rd series were confirmed.

Visual evaluation of samples proved a less dense network of wider microcracks on the surface of all samples, which might be positive in case of reparation of structures affected by the fire.

The strength improvement in case of mix with silica fume is crucial as it is known that concrete with denser microstructure formed by additional hydration is more susceptible to spalling than concrete with traditional Portland cement and higher w/c ratio. The reduction of the total weight of samples with applied IHT200/2 can be closely connected to the extent of microcrack formation in the whole mass of samples confirmed by lower strength loss of non-treated samples. The S/C ratio is lower for blended types of cement and concrete with SCM, which is supposed to contribute to a lower quantity of released water, but the dense microstructure seems to play a more significant role when it comes to the fire resistance of concrete.

7.5 Large scale testing of one-side heated uniaxially loaded slabs with applied IHT200/2

Testing of various concrete types with applied IHT200/2 under thermal load according to m-ISO curve was performed on one-side heated uniaxially loaded slabs. The same concrete types, as in the 4th series (4CEM, 5FA, 6SF and 7NO) were prepared and tested. Slabs thickness was 80±5 mm with exposure area 0,148 m^2, and tested when the surface moisture content was around 3% (recommended value from Eurocode 2). Thermocouples were monitoring temperature development at depths of 0, 20, 40 and 60 mm from the heated surface. Set up was inspired by experimental work presented during the 5th International RILEM Workshop on Concrete Spalling due to Fire Exposure, Borås, Sweden.

Test slabs were weighed prior to the application of IHT200/2 and subsequently loaded and placed on the test frame. After the application of IHT200/2 cracks, discolouration, and weight loss of slabs were inspected. Thermal exposure, according to m-ISO curve, was performed within 72 hours after the IHT200/2 application, the load level was unknown. After reaching the requested maximum temperature 1050°C, the oven was switched off, and the test slab remained placed on the oven with applied uniaxial loading during the whole cooling period. Temperature and moisture content on non-heated surfaces were recorded before, during and after application of IHT method and exposure according to m-ISO curve. Finally, overall monitoring of phenomena such as explosive spalling, moisture release from cold surface, and discoloration or shape changes of the slab were carefully observed during the whole exposure period.

7.5.1 Application of IHT200/2

The weight loss due to application of IHT200/2 was low, from 0% in the case of 5FA up to 0,17% in the case of 7NO. During the application, cracks on the side of slab 7NO appeared. The Tg in the first 20 mm from the heated surface was increasing rapidly during the whole heating ramp period up to 200°C and reached values from 7,63 to 8,72*10^3 °C/m. During the temperature dwell, Tg was gradually decreasing until the end of the temperature dwell, and subsequently Tg was dropping rapidly. The same trend, but with the maximum Tg between 0,8 to 1,0 *10^3 °C/m was observed in depths between 20 to 40 mm, and even lower between 40 to 60 mm which is favourable for IHT method as it is meant to be applied only up to a depth of 50 mm (standard thickness of a cover layer of steel rebars). The gradual decrease of Tg between 0 and 20 mm during the temperature dwell is resolute for efficiency (length and depth of application).

The thickness of IHT zone and IHT transition zone was calculated for tested concrete types, and is based on all processes which arise during the thermal exposure of concrete to temperatures between 200 and 95°C, schematically presented in Fig. 12.

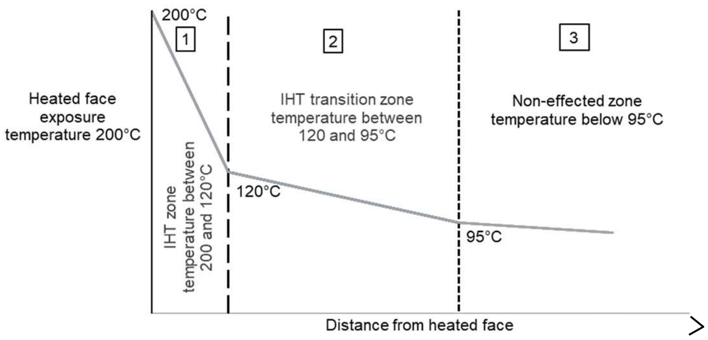


Fig. 12: Graphical presentation of the IHT method zones. Zone 1: Evaporation of free and mechanically bonded water from pores and CSH interlayers, and release of chemically bonded water from Ettringite and CSH gel; Zone 2: Evaporation of free and mechanically bonded water from pores and CSH interlayers; Zone 3: Evaporation of free water formed pores in limited amount and low speed.

Processes which are taking place between 200°C and 120°C are (i) modification of CSH gel and predominant formation of Larnite, with the highest intensity between 120°C and 200°C; (ii) recrystallisation of Ettringite between 70°C and 120°C, and (iii) evaporation of free, surface adsorbed (physically bonded by van der Waals forces), and chemically bonded water. The highest ratio of free water evaporation is between 95 and 105°C (23), which is extending the thickness of the affected area and is called a transition zone between non-treated concrete and IHT method. In this transition IHT zone, defined by minimum exposure temperature 95°C, is a partly reduced amount of free water, adsorbed water and chemically bonded water from Ettringite.

Tab. 1: Parameters for IHT zone and IHT transition zone for tested concrete types.

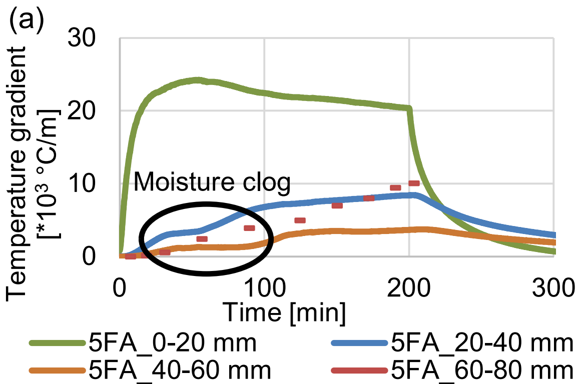
	IHT zone: Target temp. 120°C				IHT transition zone: Target temp. 95°C				Total IHT
	Surface temp. [°C]	Width [mm]	Time [min]	Tg [*10^3 °C/m]	Surface temp. [°C]	Width [mm]	Time [min]	Tg [*10^3 °C/m]	[mm]
4CEM	193,1	14,5	158	5,04	147,5	11,0	206	2,06	25,5
5FA	196,5	15,5	154	4,94	140,6	23,0	184	1,18	38,5
6SF	199,3	16,0	148	4,96	142,8	27,0	168	1,11	43,0
7NO	197,1	15,5	154	4,97	146,0	13,5	178	1,76	29,0

Application of IHT200/2 did not cause any damage to all mixes with Basalt_IS aggregates – 4CEM, 5FA and 6SF. Cracks with a width of 0,4 mm formed on the non-loaded sides of slab 7NO with Granite_NO aggregates, during the application of IHT200/2. Cracks were forming during the heating period, and therefore it is assumed that temperature gradient (8,28*10^3 °C/m) is too high and by a selection of slower heating regime, or reduction of maximum temperature, crack formation could be prevented. The temperature gradient of all slabs was up to 8,72*10^3 °C/m, and the depth of IHT200/2 penetration from 14,5 to 16 mm (6SF). The width of the IHT transition zone was between 11,0 and 27,0 mm, and is dependent on the speed of cooling and related to penetration depth of temperatures between 120 and 95°C, below 95°C is concrete considered non-affected by the IHT method. The total thickness of IHT method is between 25,5 and 43,0 mm.

7.5.2 Thermal exposure of slabs with applied IHT200/2 according to m-ISO cure

Exposure of one-side heated, uniaxially loaded slabs was performed approx. 48 hours after the application of IHT200/2. The behaviour of individual slabs, 4CEM, 5FA, 6SF and 7NO was slightly different, but overall results from testing were comparable and followed the same trend, no explosive spalling and formation of one larger crack during the cooling period. The only expectation was slab 6S with silica fume, which contributed to formation of a network of cracks with width up to 0,6 mm. Temperature development in individual test slabs is presented in Annex 3, and it could be concluded that 6SF has the highest thermal conductivity, which is also confirmed by Tg.

Fourier´s law explains temperature gradient relation to thermal conductivity, which is essential for research connected to fire resistance of concrete. Measurements of temperature in depths 20, 40 and 60 mm from the heated surface provided valuable data for evaluation of moisture migration through the test slab. As the cold surface of the slab was not isolated, moisture could freely migrate in all directions and evaporate from all surfaces (heated and non-heated). From Fig. 13a, areas are visible where Tg is constant, which signifies moisture migration towards a non-heated surface. This phenomenon was observed in the case of all samples and varies based on the coefficient of thermal expansion, moisture content and porous structure of given concrete. Moisture migration and its evaporation from the non-heated surface was also verified by moisture measurements performed during the thermal exposure according to m-ISO curve on top and sides of individual slabs. As an example, moisture development of sample 5FA, Fig. 13 is presented where the release of moisture was recorded around temperature 888°C at 120 min from test initiation.



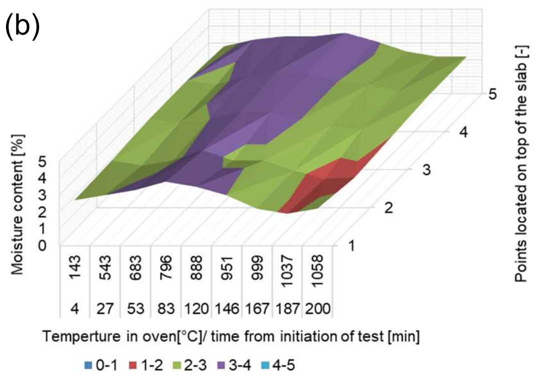


Fig. 13: (a) Temperature gradient development of sample 5FA during the thermal exposure according to m-ISO curve; (b) Moisture development on the top surface of slab 5FA with applied IHT200/2 during thermal exposure according to m-ISO curve.

The most significant damages were recorded in the case of 7NO and 6SF. The cracks which were formed during the thermal exposure on slab 6SF have not closed as in the case of other tested samples and remain open. The release of vapour from slab 7NO was significant, and the width of a single crack during cooling was the biggest, see Fig. 14.

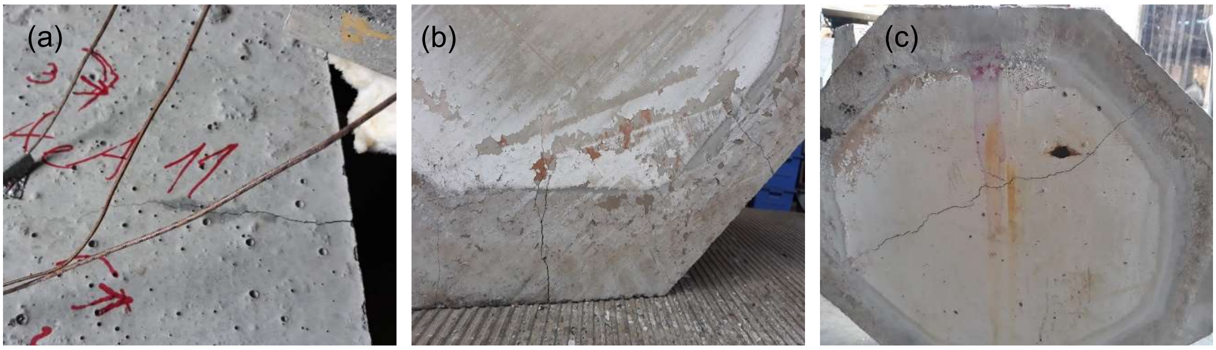


Fig. 14: Photo documentation of sample 7NO and 6SF during and after thermal exposure. (a) Moisture evaporation during heating period; (b) Cracks on the edges after cooling; (c) one crack formed during cooling across the whole slab in the direction of loading.

7.6 The final conclusion of IHT method parameters

The efficiency of the IHT method is based on three main parameters, speed of heating, maximum exposure temperature and length of thermal exposure.

The maximum rate of the heating should not cause cracking of surface and extensive microcrack formation. The speed of heating is closely connected to a temperature gradient, which can serve as a monitoring parameter for the selection of the heating speed. The IHT200/2 with the maximum temperature gradient $8{,}72 \cdot 10^{3}$ °C/m showed satisfactory results besides the concrete mix with Granite_NO aggregates where the slower heating rate with a lowered temperature gradient would be recommended.

It is beneficial to keep the maximum exposure temperature under 200°C due to thermal volume changes of cement paste and degradation due to recrystallisation of chemical compounds in the cement paste. The IHT200/2 achieved a depth of IHT penetration between 14,5 to 16,0 mm in the case of all tested concrete types, and the cracks formed during the heating of 7NO stabilised and have not expanded further. The thickness of the IHT zone and IHT transition zone is predominantly depended on the coefficient of thermal conductivity and moisture content of concrete. However, as those parameters are changing based on exposure temperature, it is not convenient to set them as reference parameters for the configuration of individual IHT method parameters for various concrete types.

The length of the application of the IHT method is decisive for its efficiency and thickness of the surface layer modified by the IHT method. The penetration of heat is decreasing over the exposure time, and with longer exposure time, the method is becoming less efficient. The temperature gradient showed a decreasing tendency, and therefore the exposure length 2 hours seems to be the most effective. The thickness of the IHT zone, and the IHT transition zone cannot be wider than the cover layer due to the overheating of steel rebars, which was achieved. Furthermore, the IHT method does not have any impact on bearing capacity and durability of the existing structure.

The results of all four series are consistent and would serve for the development of a mathematical model for configuration of the IHT method and its design for various concrete types. Furthermore, all four series adjoin each other and confirm the final configuration and mechanism of the IHT method. During the development of the IHT method more influencing factors than expected was identified and the extension of the development was enormous. Due to this fact, the detailed investigation of the other two methods was reduced and served only for comparison to the IHT method and its verification.

Moisture content and porous content influencing the heat transfer in concrete, and penetration of heat is lower with denser porous structure. Higher moisture content contributes to higher conductivity, but when more pores are present, conductivity is reduced despite higher moisture content in the pores. Also, higher porosity is commonly characterised by higher moisture content and when the moisture evaporates, the heat conductivity is changed. Thanks to the higher conductivity of dense structure with silica fume, heat transfer during the application of the IHT method is faster, and thickness of the IHT transition zone is bigger, but still within the limits of the cover layer, which is commonly 50 mm. This phenomenon is highly beneficial for extension of drying zone, and a significant reduction of pressure development and formation of moisture clog. Graphical explanation of the IHT method principle by temperature (T), vapour pressure (p_v) and moisture content (w) changes in relation to time during the thermal exposure as described in Fig. 15**Error! Reference source not found.**.

The Dry zone (DZ) and Vapour zone (VZ) are wider in the case of normal strength concrete while high strength concrete, such as high-performance or ultra-high performance concrete has thinner VZ closer to the heated surface. As moisture clog and rise of vapour pressure is concentrated in thin area, explosive spalling could occur. The application of IHT method extends the VZ and remains closer to the heated surface as in the case of high-performance concrete, which provides easier migration of moisture with lower vapour pressure as the release takes place in a larger area (wider vapour zone).

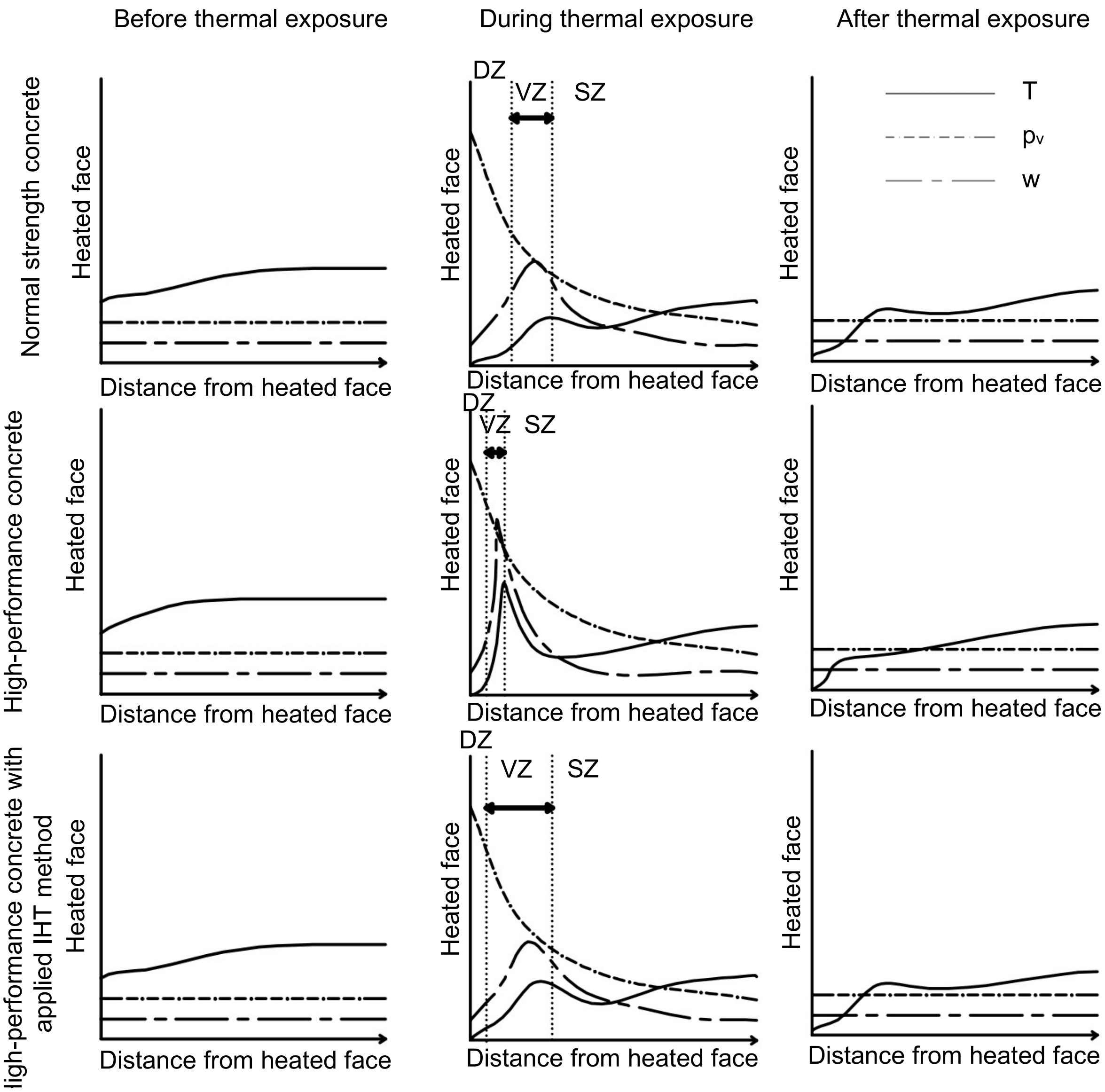


Fig. 15: Graphical explanation of IHT method principle. The thickness change of dry zone and extension of vapour zone thanks to application of the IHT method. Abbreviations: Dry zone (DZ); Vapour zone/Drying zone (VZ); Saturated zone/Moist concrete (SZ); Temperature (T); Vapour pressure (p_v); Moisture content (w).

In comparison to polypropylene fibres (PPF) IHT method is active since the first moment of a fire event, while PP fibres contribute in a bigger share to fire resistance after their melting, which could be from 150 to 170°C depending on their individual characteristics. Prior to the melt of PPF, the vapour escape channels are only in porous ITZ around the fibres - PITS principle (10).

The technique for application of the IHT method on existing structures would be executed by truck with hydraulic arms finished with radiator boards in the shape of the surface prepared for treatment. A truck would move according to the configuration of IHT_T/t. The speed of the truck will define the heat ramp (indirect radiation from sides of main heat panel), maximum exposure temperature (adjustable on the radiator), and length of exposure to requested maximum exposure temperature will be again defined by the speed of the truck. The cooling phase will depend on overall conditions in the tunnel, and its duration can be eventually regulated by some shielding located next to the radiator for extension of the cooling phase. An example of the truck for the application of the IHT method is shown in Fig. 16.

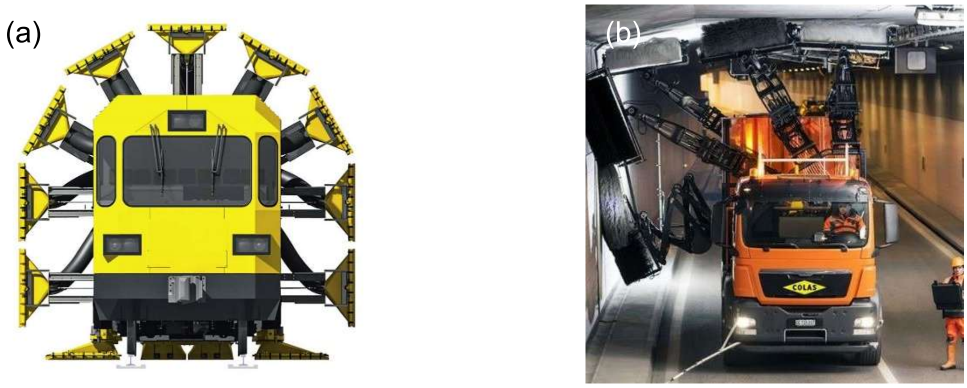


Fig. 16: Example of truck for application of IHT method: (a) for train tunnels; (b) for road tunnels.

Newly developed IHT method for enhancement of fire resistance might be innovatively used also for the treatment of industrial floors in fabric with high temperature production processes.

8 CHAPTER: AEA-FIRESCRETE UNDER THERMAL LOAD

Experimental work focused on the behaviour of concrete with the addition of traditional and "new generation" AeA, AeA-FiResCrete, exposed to elevated temperatures consists of 4 mixes. The assumption that higher air content in concrete contributes to fire resistance will be verified. Data from fire testing consists of small-scale testing on cubes with an edge 100 mm and large-scale test of one-side heated, uniaxially loaded slabs.

Results from air void characteristics correlate with freeze-thaw resistance in the case of all tested samples, 1AeA-N and 3AeA-Si had sufficient air content, which contributed to low scaling during the freeze-thaw testing, while the other two samples had low air content in hardened concrete and failed freeze-thaw resistance testing. Freeze-thaw resistance of 4CEM was insufficient and explained by poor air void distribution and total air content in hardened concrete.

Tab. 2: Properties of four AeA-FiResCrete mixes in the fresh state, hardened state and after thermal exposure according to m-ISO curve.

Name	Fresh concrete	Hardened concrete						
	Air content	Compressive strength [MPa]			Air void characteristics			F-T res*
	[%]	28 days	After m-ISO	Remaining f_c after m-ISO [%]	α [mm^{-1}]	A [vol.%]	L [mm]	56 cyc. [kg/m^2]
1AeA-N	9,6	49,9	13,5	21,6	22	8,4	0,11	0,05
2AeA-MC	4	48,3	13,2	23,5	21	2,9	0,3	0,76
3AeA-Si	5,1	55,3	16,1	27,2	23	4,8	0,21	0,23
4CEM	3,0	55,7	16,9	27,5	10	3,0	0,68	4,65

*The amount of scaled material after exposure to 56 freeze-thaw cycles.

The cause of insufficient freeze-thaw resistance in the case of 2AeA-MC might also be due to incomplete dispersion of polymer microspheres during the mixing process. The high content of air (8,4%) in mix 1AeA-N ensured good freeze-thaw resistance, but the reduction of compressive strength by 16 MPa in comparison to 4CEM (same mix without AeA) is inappropriate.

Fire resistance of AeA-FiResCrete mixes was at first tested on cubes with an edge of 100 mm and loss of compressive strength along with weight loss recorded. Concrete mix 1AeA-N, which performed the best in freeze-thaw resistance test, had the highest strength loss due to thermal exposure according to m-ISO curve. Both “new generation” AeA performed slightly better than traditional AeA (1AeA-N), but remaining strength was lower or equal to samples treated with IHT method. The dependency of the high content of the air in hardened concrete on remaining compressive strength after thermal exposure was unclear. Weight loss of 2AeA-MC was the highest due to melting and subsequent evaporation of polymer microspheres, which was also proven by the discolouration of the test sample.

The behaviour of AeA-FiResCrete thermally loaded according to m-ISO was also verified on one-side heated, uniaxially loaded slabs. Development of heat in slab 1AeA-N and 3AeA-Si was similar, but temperature development in slab 2AeA-MC was significantly influenced by the melting of polymer microspheres. Melting of polymer microspheres also influenced the temperature gradient and “moisture clog” is extremely visible from the results of Tg Fig. 17 and might be misleading.

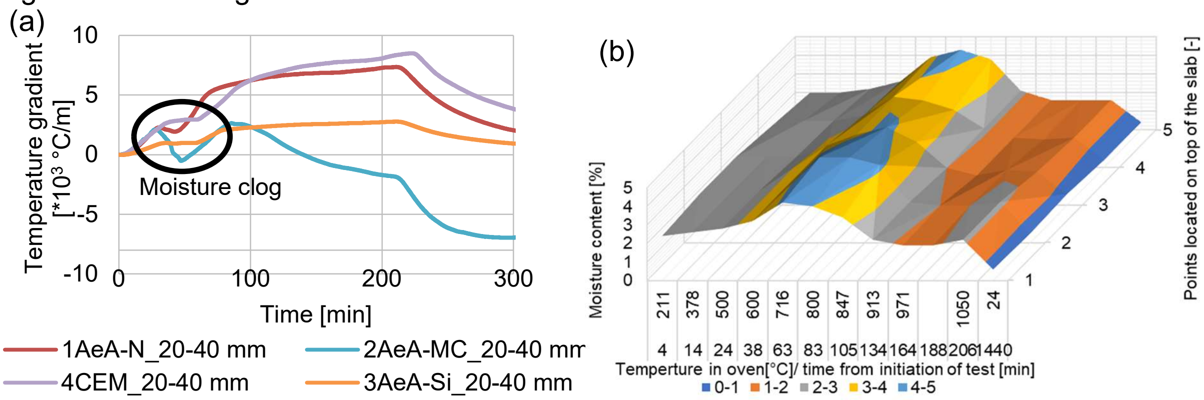


Fig. 17: (a) Temperature gradients of individual slabs measured between 20 to 40 mm from a heated surface according to m-ISO curve; (b) Moisture development on the top surface of slab 2AeA-MC during thermal exposure according to m-ISO curve.

Moisture clog was significant in the case of 1AeA-N and 2AeA-MC, while 3AeA-Si had a less dense and wider condensation zone demonstrated by temperature dwell and subsequent gain recorded by inbuilt thermocouples. Such a temperature dwell is expressed in Tg as drop and subsequently rapid increase of Tg. From Fig. 17 migration of moisture clog (vapour zone) towards the cold surface is visible, and from moisture content measured on the top surface (Fig. 17) it can be concluded, that the moisture zone migrated through the whole thickness of the test slab. Moisture content was the highest at 600°C (38 min from initiation of the test) and consequently decreased below the initial measured value. Explosive spalling has not occurred as the moisture was not restricted and could evaporate through a non-heated surface. In real situations would be further migration of moisture clog would be restricted, and extensive development of vapour pressure in the surface layer might cause crack formation and explosive spalling.

Fig. 18: Photo documentation of tested slabs. (a)1AeA-N crack formed over the whole slab during the cooling period; (b) 2AeA-MC heated surface where the polymer microspheres coloured the non-heated surface; (c) 3AeA-Si cracked formed on the non-heated surface during the cooling period.

During the thermal exposure, according to m-ISO curve, cracks with a width of 0,2±0,1 mm were formed on the unloaded sides of the slab. Those cracks closed during the cooling phase, and one larger crack over the whole length in loading direction was formed. The width of the crack formed during cooling differs for individual slabs and was caused by uneven cooling signified by rapid reduction of thermal gradient in the zone 0-20 mm from the heated surface. Furthermore, volume changes of concrete at the time of cooling, expansion of cement paste, also contributed to single crack formation, and, as a consequence of uniaxial loading, expansion of slab was permitted only perpendicularly to the loading direction.

8.1 Conclusion of AeA-FiResCrete

The assumed theory of the relation between freeze-thaw resistance and fire resistance of concrete was not fully confirmed, and only a small overlap of pore diameter suitable for both is registered and confirmed by pores structure evaluation in IHT method development 1st series, chapter 7.1. Concrete mixes prepared in this chapter serve for comparison to the IHT method, especially behaviour of slabs. The detailed evaluation of porous structure is subject to further study. For air content in fresh concrete Airvoid analyzer (AVA) might be suitable, and use of intrusion mercury porosimetry or 1H NMR analyser for analysis of the porous structure of hardened concrete might verify present conclusions.

Higher porosity suitable for freeze-thaw resistance of concrete is not necessarily improving fire resistance of concrete. The higher porosity is no assurance of the easier migration of water through the microstructure. If the porous structure is formed by larger pores characterised by a poor spacing factor, migrating water and vapour have difficulties in passing between individual pores through narrow “necks” explained by Bažant (24). Further investigation of the “new generation” AeA – Centrament Airpolymer (polymer microspheres) from MC-Bauchemie is adventurous as the number of detected air pores was insufficient.

Moisture clog was more significant in the case of higher air content (1AeA-N), while mix 4CEM with denser structure with applied IHT200/2 recorded a wider vapour zone with lower moisture cumulation. Also, AeA SikaControl AER-200 P from Sika in mix 3AeA-Si contributed to the extension of the vapour zone, which is favourable for mitigation of crack formation and fire spalling.

CONCLUSION

Fire resistance is a complex problem, which involves many variables and influencing parameters. Therefore, extensive literature review and theoretical explanation of the necessity of fire resistance, legislation connected to fire protection and presently used fire protection methods are described. Finally, a detailed description of individual components and subsequently concrete as a cementitious composite was described, to gain basic knowledge about mechanical, physical and chemical changes due to thermal exposure.

The subject of this PhD thesis was the development of two different approaches for enhancement of fire resistance of concrete structures, especially structures from the higher fire risk group such as tunnel lining, underground garages, and possibly nuclear power plants. The greatest attention was given to the development of a new method for enhancement of fire resistance of existing structures based on porous structure modification by intentional heat treatment. There is no completely suitable method for enhancement of fire resistance for existing tunnel lining and other structures with the higher fire risk level and restricted space and reduced ventilation possibilities. The second method AeA-FiResCrete is suitable for new constructions where modification of concrete design will lead to the improved fire resistance of the concrete structure.

For the development of both methods, smaller samples exposed entirely to elevated temperatures were used, and followed by large-scale testing. A final assessment of efficiency of developing method was performed on one-side heated uniaxially loaded slabs with inbuilt thermocouples for monitoring of temperature development in concrete mass. All samples, either for development or verification, were thermally exposed according to the modified ISO 834 curve. Equipment for the development of all three methods was not primarily adjusted for concrete with eventual explosive spalling. The electric oven performs excellently for pre-set regimes with defined ramp-dwell-cooling phase, but not for fire testing curve ISO 834, which has extremely steep temperature gain. In connection to the test oven, the one-side heated uniaxially loaded slabs were also designed to fit the oven, and due to this, shape and loading were unconventional, but still, inspired by test setups presented at the 5th International RILEM Workshop on Concrete Spalling due to Fire Exposure, Borås, Sweden. Equipment introduced for industrial application of the IHT method has not been tested, and therefore increase of ambient temperature in tunnels during application must be closely monitored.

IHT method development

The detailed description of the porous structure, chemical changes and mechanical changes were described in 4 series during the development of the IHT method. Based on the results from the 1st series, it was concluded that suitable pores are capillary pores with a diameter between 0,03 and 30 μm analysed by MIP and TGA-DTA, and if the exposure temperature exceeds 350°C, rehydration takes place, and porosity is reduced. Testing of normal strength concrete in the 2nd series showed the same trend of porous structure modification and also chemical changes of individual compounds due to thermal load analysed by MIP and XRD. In the 3rd series, various configurations of the IHT method on 3 different binder types were verified as the surface layer is predominantly formed by cement paste. Analyses showed, that the IHT method has a positive influence on compressive and flexural strength, and the most suitable configurations are IHT200/2 and IHT250/1. Described in detail, the temperature ramp of 5°C/min until the requested temperature 200°C or 250°C held for 1 or 2 hours and naturally cooled to ambient temperature. Further testing was performed only with IHT200/2 as temperature 250°C might cause greater microcracking, especially in the case of concrete with the addition of silica fume and larger samples. Binder testing was followed by the application of IHT200/2 on 4 different concrete types. Based on results from remaining compressive strength after thermal exposure of samples with and without applied IHT method (IHT200/2) according to m-ISO curve, beneficial impact on concrete with blended cement (FA cement - CEM II/B-M) and concrete with the content of silica fume can be concluded. Contribution to CEM I is negligible even slightly negative, and in the case of Granite_NO aggregates, the effect of the IHT method was lost to the degradation of Granite aggregates.

Verification of IHT method efficiency was tested on one-side heated, uniaxially loaded slabs with integrated thermocouples for heat spread monitoring. From the results, it could be concluded that the vapour zone/drying zone is extended, and the risk of moisture clog formation reduced, as is also the case of explosive spalling and extensive crack formation. Furthermore, individual parameters and their connection to a thickness of the IHT zone and IHT transition zone were explained.

The total thickness of IHT method is between 25,5 and 43,0 mm, and consists of the IHT zone ranged from 14,5 till 16,0 mm, and thickness of the IHT transition zone was between 11,0 to 27,0 mm, which is in conformity to a thickness of the cover layer, and no impact on steel rebars is ensured. Finally, the method for industrial application is suggested, trucks with radiators on hydraulic arms.

The benefits of the new IHT method are (i) after treatment of the IHT method, fires can be extinguished by water and do not emit harmful and poisoning gasses; (ii) locations of installations such as tunnels do not need to be necessarily fully closed during the application period which is relatively short in comparison to other fire resistance methods; (iii) application of IHT method has a low environmental impact; (iv) its application does not have a negative impact on strength and durability of concrete and whole concrete structure; (v) calculation of cost for realisation of the IHT method is transparent as heat (energy) is the only requirement; (vi) IHT method is adjustable to various concrete types and different structure shapes.

Further development of IHT method would include a detailed investigation of (i) stability of IHT_T/t in natural ambient with relative humidity around 75% and exposure to exhaust gases; (ii) further investigation of the cooling phase; (iii) the development of the IHT method was highly comprehensive, and all results are interconnected, the set of data might serve for the development of a mathematical model for IHT method design, and also for prediction of crack formation in heated concrete.

AeA-FiResCrete

The second method investigated air-entraining agents (AeA) and their contribution to the fire resistance of newly produced concrete. Three types of AeA were tested, including two "new generation" AeA and conclusions are that pores formed by AeA are not entirely suitable for fire resistance. Moisture clog was visible from temperature gradient measured between individual thermocouples, and migration of moisture through the whole thickness of slab was recorded. Further investigation of porous structure and moisture migration is needed for a distinct conclusion of AeA's contribution to the fire resistance of concrete. Despite that, results showed, that more moisture is captured with higher porosity, which could cause explosive spalling and extensive cracking if the non-heated surface is impermeable.

In general, the use of polymer fibres is not entirely environmentally friendly, and thus air-entraining agents might be a beneficial option for a sustainable method for enhancement of fire resistance of newly built concrete structures.

Contribution to science and practice

The following points were selected as a significant contribution to science, (i) a detailed description of chemical changes of cement paste exposed to elevated temperatures demonstrated by results from TGA-DTA, XRD, MIP and temperature measurements in concrete during the thermal exposure; (ii) detail description of porous structure modification of various concrete types. Definition of pores suitable for fire resistance; (iii) evaluation of 4 concrete types with 2 different aggregates, 3 binder types and 3 air-entraining agents; (iv) system for moisture clog monitoring and heat penetration based on temperature measurements and temperature gradient calculation.

The following points were selected as significant contribution to practice, (i) new method for enhancement of fire resistance of existing concrete structures with no need for additional materials and low environmental impact; (ii) innovative solution for the design of new concrete with improved fire resistance properties by "new generation" air-entraining agents, which is more ecological than the use of polypropylene fibres.

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