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Performance of metakaolin concrete at elevated temperatures

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Abstract

An experimental investigation was conducted to evaluate the performance of metakaolin (MK) concrete at elevated temperatures up to 800 °C. Eight normal and high strength concrete (HSC) mixes incorporating 0%, 5%, 10% and 20% MK were prepared. The residual compressive strength, chloride-ion penetration, porosity and average pore sizes were measured and compared with silica fume (SF), fly ash (FA) and pure ordinary Portland cement (OPC) concretes. It was found that after an increase in compressive strength at 200 °C, the MK concrete suffered a more severe loss of compressive strength and permeability-related durability than the corresponding SF, FA and OPC concretes at higher temperatures. Explosive spalling was observed in both normal and high strength MK concretes and the frequency increased with higher MK contents.

Keywords: Fire resistance; Spalling; Metakaolin; High strength concrete; Durability; Permeability; Micro-structure; Silica fume; Fly ash

1. Introduction

Metakaolin (MK) is a recent addition in the list of pozzolanic materials. It is a thermally activated alumino-silicate produced from kaolinite clay through a calcining process. Unlike other pozzolans, MK is a primary product, not a secondary product or by-product. This allows the manufacturing process to be structured to produce the optimum characteristics for the MK, ensuring the production of a consistent product and a consistent supply. The white color of MK results in a concrete with lighter color, another advantage making it popular.

MK enhances the strength and durability of concrete through three primary actions which are the *filler effect*, the *acceleration of ordinary Portland cement (OPC) hydration* and the *pozzolanic reaction with calcium hydroxide* (CH). Wild et al. [1] found that the filler effect is immediate, the acceleration of OPC hydration has its major impact within the first 24 h and the maximum effect of pozzolanic reaction occurs between 7 and 14 days. It was concluded that the optimum replacement level of OPC by MK to give maximum long term strength is about 20% by weight.

Kostuch et al. [2] discovered that a 10% replacement of cement with MK reduced the CH content in concrete by 70%, and a 20% replacement reduced it to almost zero after 28 days. However, the amount of MK required for complete elimination of CH depends on a number of factors such as purity of MK, Portland cement composition, water/binder ratio and curing conditions [3]. The reduction in CH content results in superior strength and durability performance, even at elevated temperatures [4]. Poon et al. [5] prepared normal and high strength concrete (HSC) mixes incorporating 5%, 10% and 20% MK and compared their performance with the equivalent silica fume (SF) and fly ash (FA) mixes. They observed that the MK concrete possessed higher strength, lower permeability and less porosity as compared to the corresponding SF and FA concretes.

It was observed that the fire resistance of concrete is highly dependent on its constituent materials, particularly the pozzolans. A number of research studies [6] indicated that the addition of SF highly densifies the pore structure of concrete, which can result in explosive spalling due to the build-up of pore pressure by steam. Since the evaporation of physically absorbed water

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starts at 80 °C which induces thermal cracks, such concretes may show inferior performance as compared to pure OPC concretes at elevated temperatures.

On the other hand, the addition of FA or ground granulated blast furnace slag (GGBS) enhances the fire resistance of concrete [6]. Yigang et al. [7] indicated that the compressive strength of FA concrete at 250 °C was more than the original unfired strength. Moreover, the FA concrete retained higher strengths than the pure OPC concrete at higher temperatures up to 650 °C. Dias et al. [8] found that the addition of FA completely eliminated all visible surface crackings for specimens heated up to 600 °C.

Diederichs et al. [9] prepared 3 HSC mixes incorporating SF, FA and GGBS independently. The mixes were subjected to a maximum temperature of 900 °C and tested under loaded conditions. The GGBS concrete showed the best performance followed by FA and SF concretes.

Since human safety in case of fire is one of the major considerations in the design of buildings, it is extremely necessary to have a complete knowledge about the behavior of all construction materials before using them in the structural elements. So far no experimental data have been published on the fire resistance of MK concrete hence this research was conducted to investigate its performance at elevated temperatures as observed in fires or in oil, gas or power industries.

For this study, eight normal and HSC mixes incorporating 0–20% MK were prepared and exposed to elevated temperatures up to 800 °C. The residual compressive strength, permeability, porosity and pore sizes were measured and compared with those of SF, FA and pure OPC concretes. A spalling frequency analysis was also carried out to consider the feasibility of using MK concrete in oil or power industries where hydrocarbon fires are not uncommon.

2. Experimental details

An experimental program was designed to investigate the residual properties of MK concrete after elevated temperatures. For this purpose, four normal strength and four HSC mixes were prepared incorporating 0%, 5%, 10% and 20% MK. In addition, three HSC containing 5%, 10% SF and 20% FA by weight, and one normal strength concrete (NSC) with 20% FA replacement were prepared for comparison.

2.1. Materials

The cementitious materials used were OPC complying with ASTM Type I, low calcium FA equivalent to ASTM class F, condensed SF, and MK. The chemical composition and physical properties of these materials, as supplied by the suppliers, are shown in Table 1.

The fine aggregate was natural river sand. Crushed granite, with maximum nominal sizes of 10 and 20 mm, was mixed in a ratio of 1:2 and used as coarse aggregate. A liquid sulfonated naphthalene–formaldehyde condensate containing 38.6% solids was used as superplasticizer.

2.2. Mix proportions

The mix proportions of seven high strength (HS) and five normal strength (NS) concrete mixes are shown in Table 2. The pozzolans were introduced as cement replacement materials and their proportions were decided on the basis of previous research to achieve the optimum strength and durability [1–6]. Two control mixes with pure OPC were also prepared for comparison purposes. All the mixtures were produced at a slump of 200 mm to make free flowing concrete. Due to the tropical climate of Hong Kong, no air entraining admixture was used.

Table 1
Chemical composition and physical properties of cementitious materials

	OPC	SF	FA	MK
Chemical composition (%)				
Silicon dioxide (SiO_2)	19.61	90.26	56.79	53.20
Aluminum oxide (Al_2O_3)	7.33	0.63	28.21	43.90
Ferric oxide (Fe_2O_3)	3.32	0.33	5.31	0.38
Calcium oxide (CaO)	63.15	3.18	< 3.00	0.02
Magnesium oxide (MgO)	2.54	0.33	5.21	0.05
Sodium oxide (Na_2O)	–	–	–	0.17
Potassium oxide (K_2O)	–	–	–	0.10
Sulfur trioxide (SO_3)	2.13	0.4	0.68	–
Loss on ignition	2.97	4.84	3.90	0.50
Physical properties				
Specific gravity	3.16	2.22	2.31	2.62
Specific surface (cm^2/kg)	3519	–	4120	12,680

As supplied by suppliers.

Table 2
Mix proportions of concrete mixtures

Mix	SF (%)	FA (%)	MK (%)	W/B	Batched quantities (kg/m^3)					28 days compressive strength (MPa)
					Water	Cement	Fine agg.	Coarse agg.	SP[a]	
High strength concrete (HSC)										
HS-CC[b]	–	–	–	0.30	150	500	758	927	0.5	85.9
HS-SF5	5	–	–	0.30	150	475	710	1066	0.6	96.5
HS-SF10	10	–	–	0.30	150	450	620	1151	0.8	108.3
HS-MK5	–	–	5	0.30	150	475	713	1065	0.6	107.3
HS-MK10	–	–	10	0.30	150	450	625	1149	0.8	123.1
HS-MK20	–	–	20	0.30	150	400	609	1142	1.1	131.3
HS-FA20	–	20	–	0.30	150	400	618	1147	0.8	82.7
Normal strength concrete (NSC)										
NS-CC[b]	–	–	–	0.50	195	390	768	917	–	35.8
NS-MK5	–	–	5	0.50	195	370	710	1067	–	40.7
NS-MK10	–	–	10	0.50	195	351	620	1154	0.2	47.6
NS-MK20	–	–	20	0.50	195	312	618	1149	0.5	58.4
NS-FA20	–	20	–	0.50	195	312	615	1143	–	39.3

[a] Superplasticizer content in percent by weight of binder. The amount of superplasticizer was decided during mixing of fresh concrete to achieve equal workability level (slump) for all concretes.
[b] Control concrete.

2.3. Curing and heating regimes

The specimens were demolded 24 h after the casting and placed in a water tank at 20 °C. After 28 days of water curing, they were transferred to an environmental chamber maintained at 20 °C and 75% relative humidity, the average climate in Hong Kong.

At an age of 60 days, the specimens were heated in an electric furnace up to 200, 400, 600 and 800 °C. Each temperature was maintained for 1 h to achieve the thermal steady state [10]. The heating rate was set at 2.5 °C per minute to simulate both natural fire and oil/gas industry temperatures. The specimens were allowed to cool naturally to room temperature.

2.4. Specimen dimensions and testing details

1. Unstressed residual compressive strength test (same as the standard cube test) was performed on 100 mm concrete cubes according to BS 1881: Part 120:1983. Three specimens were tested for each temperature and average values are reported.
2. To determine permeability and resistance to chloride-ion penetration, the Rapid Chloride Diffusion test was conducted according to ASTM C1202-94. The specimens were 50 mm thick slices of 100 mm nominal diameter which were cut from the standard concrete cylinders (100 mm × 200 mm) after 28 days.
3. Porosity and average pore sizes were measured by mercury intrusion porosimetry (MIP). Pellets of about 5 mm in the size of hardened cement paste (HCP) were collected from the crushed concrete cubes and immediately soaked in acetone to stop the further hydration. The samples were then dried in a vacuum oven at 60 °C for 48 h before testing to achieve constant weight.
4. Spalling frequency was determined as a percentage of cubic specimens exploded in the furnace up to 800 °C, i.e., spalling frequency = (specimens exploded up to 800 °C)/(total number of specimens) × 100.

3. Test results and discussion

3.1. Residual compressive strength

The residual compressive strength after cooling was determined by an *unstressed compression test* [6]. This method gives lower values of compressive strength as compared to *stressed tests* and hence is thought to be suitable for getting the limiting results [6]. The test results shown in Figs. 1–4. Figs. 1 and 2 indicate the residual compressive strength of each specimen at different elevated temperatures while Figs. 3 and 4 depict the relative increase or decrease in the compressive strength of each specimen as compared to its original compressive strength before heating.

From the perspective of residual compressive strength of MK concrete, the heating regime can be divided into two regions as 0–400 °C and 400–800 °C. A distinct pattern of strength gain and then loss was observed in each region. Initially MK concretes showed an increase in compressive strength at 200 °C. This increase may probably be due to the hydration of unhydrated MK particles which were activated as a result of temperature rise. Since the hydration in MK concrete is slowed down

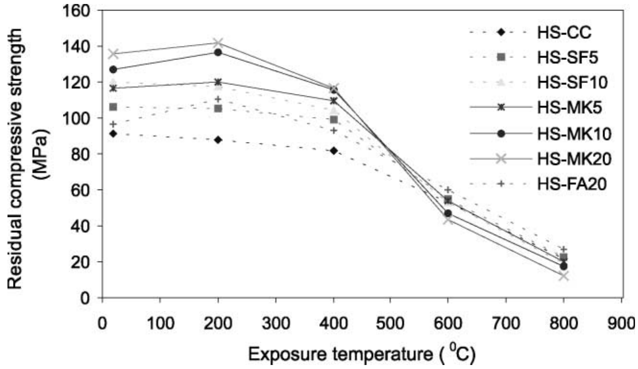

Fig. 1. Residual compressive strength of HSCs.

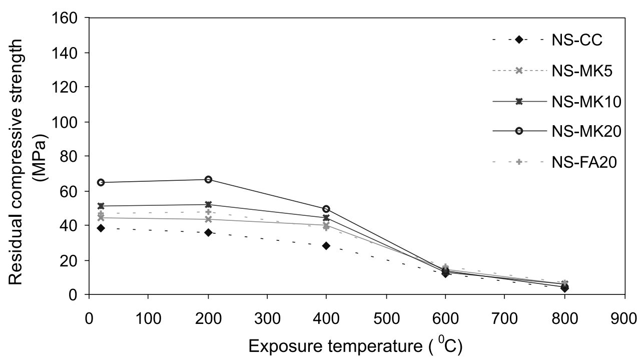

Fig. 2. Residual compressive strength of NSCs.

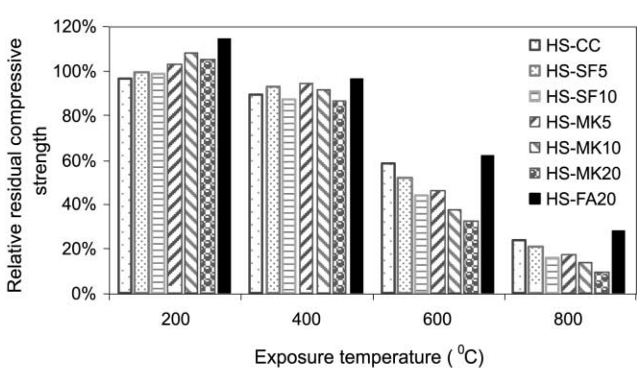

Fig. 3. Relative residual compressive strength of HSCs.

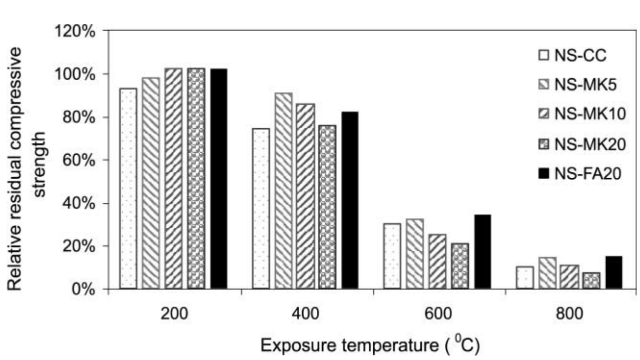

Fig. 4. Relative residual compressive strength of NSCs.

after 14 days due to the blocking of capillaries [1], such an increase in strength at elevated temperatures can be anticipated. A similar increase in strength was observed in FA concrete due to the formation of tobermorite [11]. The pure OPC and SF concretes showed a slight loss in strength. Up to 400 °C, most of the HSCs maintained their original compressive strength with higher residual strength found in MK5, SF5 and FA20. No spalling occurred in this temperature range. However multiple cracking was observed in some MK and SF concrete specimens. A 10–20% compressive strength loss was observed in NSCs.

After 400 °C, the MK concretes showed a sharp reduction in compressive strength followed by explosive spalling and severe cracking. In this temperature range, the compressive strength of MK concrete was lower than those of other concretes. The results showed a close correlation between the MK content and the degree of damage as specimens with higher MK contents suffered a bigger loss. As indicated later by MIP analysis, this severe strength loss was due to the very dense pore structure of MK concrete which enhanced the build-up of vapor pressure upon heating and resulted in spalling and cracking. The best performance at all temperatures was given by FA concretes followed by pure OPC, SF, and MK concretes with the exception of NS-MK5 which showed better results than the corresponding concretes.

3.2. Resistance to chloride-ion penetration

This test determines the electrical conductance of concrete to provide a rapid indication of its resistance to the penetration of chloride ions. The chloride-ion resistance of concrete gives an indirect measure of its permeability and internal pore structure, as more current passes through a more permeable concrete.

The test method consists of monitoring the amount of electrical current which passed through 50 mm thick slices of 100 mm nominal diameter during a 6 h period. A potential difference of 60 V dc is maintained across the ends of the specimen, one of which is immersed in a sodium chloride solution, the other in sodium hydroxide solution. The total charge passed in coulombs, has been found to be related to the resistance of the specimen to chloride-ion penetration. The details of this test can be found in ASTM C1202-94 and its results can be used to assess the durability of concrete.

The results of rapid chloride diffusion test are shown in Figs. 5 and 6. It is important to note that this test was performed only on specimens subjected to 600 and 800 °C as more damage occurred at these temperatures. Long testing time was another reason to limit the extent of this analysis.

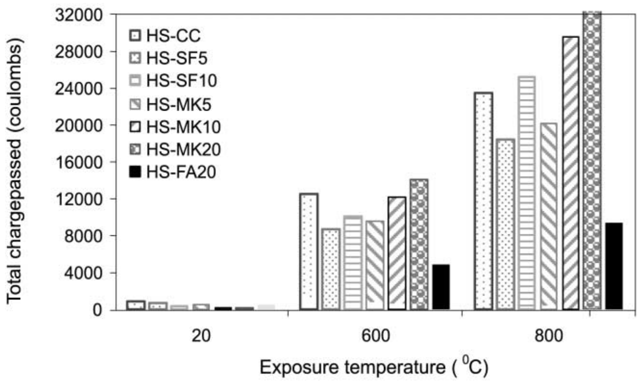

Fig. 5. Resistance of HSC against chloride-ions penetration.

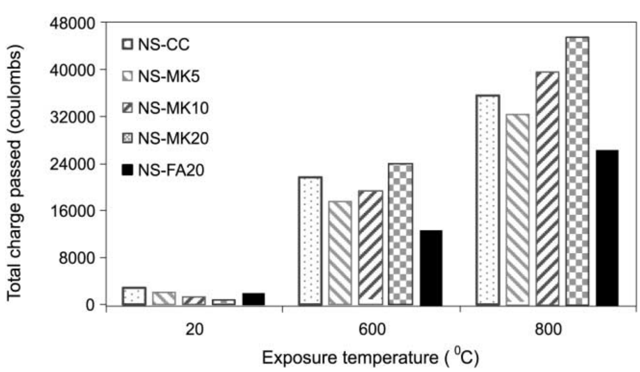

Fig. 6. Resistance of NSC against chloride-ions penetration.

The test results indicate a loss of impermeability with the rise in temperature, as indicated by the high amount of charge passed through the specimens. This loss of impermeability may be due to the internal cracking and pore structure coarsening of the concrete at high temperatures [12]. The highest loss was suffered by MK concretes followed by SF, pure OPC and FA concretes. However, the MK and SF concretes with 5% cement replacement showed better results than the pure OPC concrete due to the reduced CH content which initiates internal cracking during heating and disintegration at cooling [4].

It is important to note that the MK concrete showed a high increase in the loss of impermeability and durability after heating as compared to other tested concretes. Consequently, the use of such a concrete should be considered carefully in oil/gas structures in marine environment, which are often subjected to heating and cooling cycles.

3.3. Porosity and average pore size measurements

The porosity and average pore sizes were measured using a mercury intrusion porosimeter (MIP), which has a measuring pressure range from 0.01 to 207 MPa. The contact angle selected was 140°, so the measurable pore size range was from 0.007 to 144 μm. The test was conducted on specimens subjected to temperatures of 600 and 800 °C due to the reasons mentioned earlier. The results are plotted in Figs. 7–10.

The MIP test results highlight pore structure coarsening and increase in porosity at elevated temperatures which are the major reasons for the strength and durability losses [12]. In MK concretes, this effect was more pronounced at 800 °C as compared to 600 °C, which reveals that the initial strength and durability loss occurred due to the internal cracking. The dissociation of CH crystals, increased vapor pressure of steam and crystal transformation of quartz may be the three main reasons for such internal cracking. An interesting fact found was that despite the high strength loss, the MK concretes had lower porosities as compared to those of the corresponding SF and pure OPC concretes. However, the average pore diameters of these MK concretes were larger than those of the other concretes, which in combination with internal cracking caused more strength and durability loss at all temperatures above 200 °C.

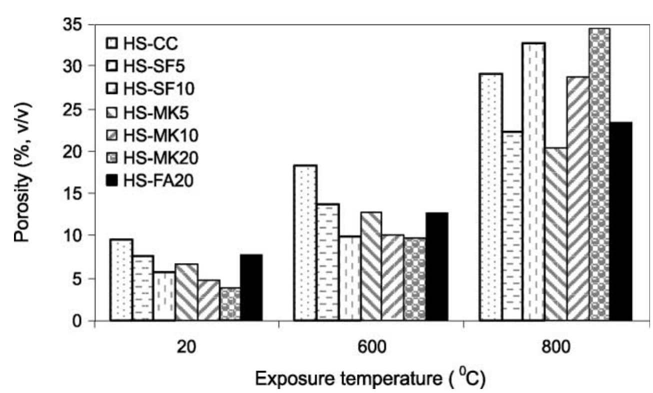

Fig. 7. Residual porosities of HSCs.

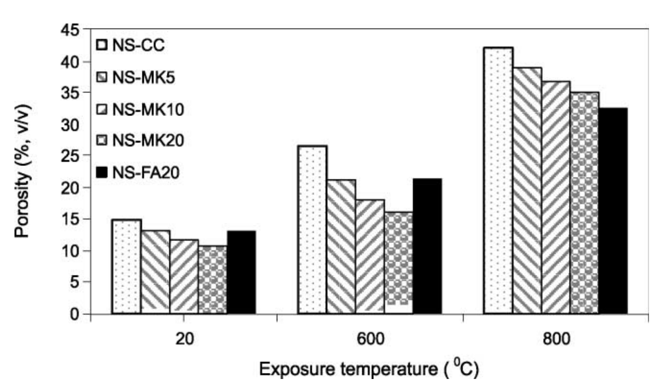

Fig. 8. Residual porosities of NSCs.

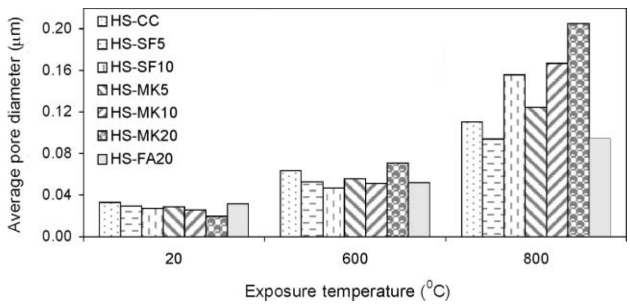

Fig. 9. Average pore sizes in HSCs.

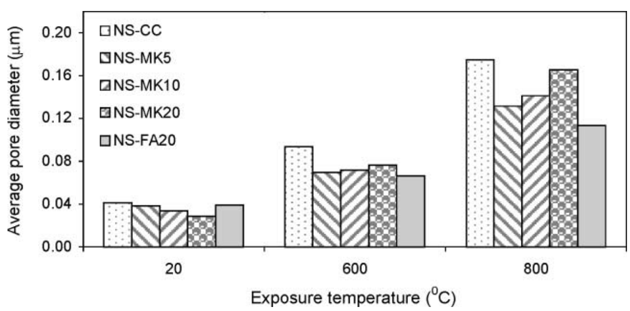

Fig. 10. Average pore sizes in NSCs.

The effect of internal cracking was more pronounced in NS-MK concretes in which in spite of lower porosities, more damage occurred with the increase in MK content. The increased internal cracking is due to the high CH content present in NSC. This fact is supported by the results of rapid chloride diffusion test and indicates that internal cracking is another parameter which results in strength and durability loss at elevated temperatures along with pore structure coarsening.

3.4. Spalling frequency analysis

The ability of concrete to withstand high temperatures can be hampered considerably by spalling which may cause loss of the concrete cover over reinforcing steel bars. Direct exposure of reinforcing bars to high temperatures severely reduces the structural integrity and bearing capacity of the reinforced concrete structure.

Two mechanisms are thought to be responsible for spalling: the vapor pressure build-up mechanism [13] and the thermal stress mechanism [14]. The former mechanism occurs because the dense HCP prevents moisture from escaping under high temperatures, thus causing a considerable pressure build-up resulting in spalling. The latter mechanism occurs because the exposure to fire produces a thermal gradient within the concrete, which increases internal stresses and results in spalling. A combination of these two mechanisms is also possible. In a recent study, Chan et al. [15] found that moisture content and strength are the two main factors governing thermally induced explosive spalling of concrete. No spalling would occur if either strength or moisture content is below a certain threshold value which was found to be 60 MPa (strength) or 62% (moisture content) for their concretes.

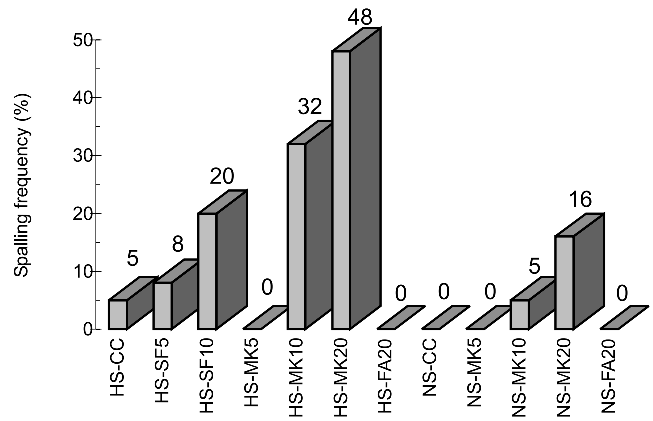

Fig. 11. Spalling frequency analysis of MK concrete at elevated temperatures.

In this research study, explosive spalling and splitting were observed particularly in MK concrete specimens. The results are plotted in Fig. 11 in which spalling frequency indicates the percent of total cubic specimens exploded up to 800 °C. It is important to note that spalling occurred between 400 and 600 °C with more frequency between 450 and 500 °C.

The results indicate high proneness of MK concrete to spalling as compared to SF, FA and pure OPC concretes. Based upon this evaluation, the obvious reason may be the dense pore structure of MK concretes which held the vapor pressure of steam and resulted in explosive spalling. This was verified from the results of MIP analysis which indicated a low porosity of MK concretes as compared to other concretes even at elevated temperatures. No spalling was observed in thin cylindrical slices for chloride diffusion test which reveals that the path of moisture escape has a significant influence on spalling as indicated by Sanjayan [16]. This urges the need to also conduct fire tests on full-scale members to obtain realistic and consistent results. The reason why most of the spalling occurred between the narrow range of 450–500 °C needs further investigations.

4. Conclusions

1. The MK concrete showed a distinct pattern of strength gain and loss at elevated temperatures. After gaining an increase in compressive strength at 200 °C, it maintained higher strengths as compared to the

corresponding SF, FA and pure OPC concretes up to 400 °C. A sharp reduction in compressive strength was observed for all HSC after 400 °C followed by severe cracking and explosive spalling. Within the range 400–800 °C, MK concretes suffered more loss and possessed lower residual strengths than the other concretes.
2. Dense micro-structure and lowest porosity are the main reasons for the poor performance of MK concrete at elevated temperatures. These concretes showed a higher loss of impermeability as compared to the mechanical strength. This indicates that even after a fire, if the concrete retains a high proportion of its original compressive strength, an examination of the durability should be made. The impermeability loss occurred initially due to the internal cracking and then increased by pore-structure coarsening.
3. Explosive spalling was observed in both normal and high strength MK concrete specimens particularly between 450 and 500 °C. The spalling frequency increased with the higher MK content. The vapor pressure build-up by dense pore-structure seems to be the obvious reason for such spalling.
4. The MK concrete with 5% cement replacement showed better performance than the corresponding pure OPC and SF concretes at all tested temperatures. No spalling was observed in this concrete.

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