

Post Elevated Temperature Effect on the Strength and Microstructure of Thin High Performance Cementitious Composites (THPCC)

A. Q. Sobia, A. Shyzleen, M. S. Hamidah, I. Azmi, S. F. A. Rafeeqi, and S. Ahmad

Abstract—Reinforced Concrete (RC) structures strengthened with fiber reinforced polymer (FRP) lack in thermal resistance under elevated temperatures in the event of fire. This phenomenon led to the lining of strengthened concrete with thin high performance cementitious composites (THPCC) to protect the substrate against elevated temperature. Elevated temperature effects on THPCC, based on different cementitious materials have been studied in the past but high-alumina cement (HAC)-based THPCC have not been well characterized. This research study will focus on the THPCC based on HAC replaced by 60%, 70%, 80% and 85% of ground granulated blast furnace slag (GGBS). Samples were evaluated by the measurement of their mechanical strength (28 & 56 days of curing) after exposed to 400°C, 600°C and 28°C of room temperature for comparison and corroborated by their microstructure study. Results showed that among all mixtures, the mix containing only HAC showed the highest compressive strength after exposed to 600°C as compared to other mixtures. However, the tensile strength of THPCC made of HAC and 60% GGBS content was comparable to the THPCC with HAC only after exposed to 600°C. Field emission scanning electron microscopy (FESEM) images of THPCC accompanying Energy Dispersive X-ray (EDX) microanalysis revealed that the microstructure deteriorated considerably after exposure to elevated temperatures which led to the decrease in mechanical strength.

Keywords Ground granulated blast furnace slag, high alumina cement, microstructure at elevated temperature and residual strength.

I. INTRODUCTION

DEFICIENT existing building facilities need retrofitting/strengthening of structural members to protect them from natural hazards such as earthquakes, fires, etc.

A.Q. Sobia is the PhD. student at Universiti Teknologi MARA (UiTM), Shah Alam 40450 Malaysia (phone: +06-017-3931653; e-mail: s_Leo85@live.com).

A. Shyzleen is the MSc. student at Universiti Teknologi MARA (UiTM), Shah Alam 40450 Malaysia (e-mail: shyzleen@ gmail.com).

M.S. Hamidah is an Associate Professor at Faculty of Civil Engineering, Universiti Teknologi MARA (UiTM), Shah Alam 40450 Malaysia (e-mail: hamid929@salam.uitm.edu.my).

I. Azmi is a Professor and Dean at Faculty of Civil Engineering, Universiti Teknologi MARA (UiTM), Shah Alam 40450 Malaysia (e-mail: azmii716@salam.uitm.edu.my).

S.F.A. Rafeeqi is a Professor and Pro-vice chancellor at NED University of Engineering and Technology, Karachi, Pakistan (e-mail: pvc2@neduet.edu.pk).

S. Ahmad is a Professor at NED University of Engineering and Technology, Karachi, Pakistan (e-mail: sahmada@neduet.edu.pk).

Deficiency of facilities could be due to the number of reasons including; age related distress, inadequate design and or construction, exposure to natural hazards like earthquake or fire, or improvement in the design codes requirements to mitigate natural disasters like earthquake etc. One approach is to retrofit/re-strengthen the reinforced concrete (RC) members using fiber reinforced polymer (FRP) materials or steel plates. If FRP materials are used, one of the major concerns is the thermal resistance of FRP materials under elevated temperatures during fire.

When all other aspects fail to contain fire, then structural integrity is the last hope of defense [1]. Existing building structures may not have proper building elements and the only solution to enhance their fire safety, is to strengthen them against fire by providing a suitable passive fire protection (PFP) layer. Best PFP layer is the one that is fully compatible with reinforced concrete structural members; non-combustible, a barrier against fire spread towards adjacent buildings and a shield to sufficiently keep the temperature of FRP and structural elements low in case of fire [2].

One approach for increasing the fire endurance of FRP strengthened RC members is encapsulating the strengthened RC members with cladding of Thin High Performance Cementitious Composites (THPCC) materials. Elevated temperature effects on THPCC, based on different cementitious materials have been studied in the past [3]-[14] but High-Alumina Cement (HAC)-based THPCC have not been well characterized. This research study will focus on the behavior of THPCC (mortar) containing HAC in conjunction with different dosages of GGBS exposed to elevated temperatures of 400°C and 600°C.

II. EXPERIMENTAL PROCEDURE

A. Materials

High-Alumina Cement (HAC), used in this study, is also known as calcium aluminate cement is obtained from CALUCEM with the brand name 'ISTRA 40'. Ground granulated blast furnace slag (GGBS) was obtained from YTL cement in dry form. The chemical analysis of HAC and GGBS shows the principal oxides, as shown in Table I. Silica sand containing 99% SiO₂ was used and the sieve analysis is shown in Fig.1. Glenium-389 was used as a high-range water reducing agent (HRWRA) supplied by BASF Malaysia.

B. Mix Proportion

Mixes with four different percentages (60%, 70%, 80% and 85%) of GGBS were cast next to the control mix containing pure HAC only. All mixes had the water-binder ratio of 0.26 and sand-cement ratio of 0.8. Details of the mix proportion are given in Table II. The materials were mixed according to ASTM C305-11 [15].

C. Casting and Curing

Cylinder specimens of dimension 50 mm Ø x 100 mm length and 100 mm Ø x 200 mm length were prepared for compression and splitting tensile strength tests respectively. Right after casting all specimens were cured into the conditioning room (20°C at 60% relative humidity) for 24hrs after which they were de-molded and cured in the conventional water tank for 28 and 56 days.

D. Elevated Temperature Test

All specimens were oven dried at 60°C for 24 hours before being exposed to the elevated temperature in order to avoid spalling due to the pore pressure development [16]. Specimens were exposed to 400°C and 600°C at a heating rate of 5°C/min and were soaked at these temperatures for one hour using the laboratory gas furnace. After heat treatment, specimens were left in the furnace to cool down at approximately the same rate and then were subjected to compression and splitting tensile strength tests. All samples exposed to elevated temperature were compared with the control samples whereas the control samples were referred to the one containing HAC only and tested at 28°C. Throughout the paper, 28°C refers to the room temperature.

E. Compressive Strength and Splitting Tensile Strength Test

For each mixture and curing regime, at least three specimens were surface dried and tested in compression according to ASTM C39/C39M-10 [17], whereas splitting tensile strength tests were performed using ASTM C496/C496M-11 [18]. For compressive strength test, dimension of the cylinder specimen was 50 mm Ø x 100 mm length whereas for splitting tensile strength test, specimen dimension was 100 mm Ø x 200 mm length.

F. Microstructural Examination

Microstructural examination was performed using Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray Microanalysis (EDX). FESEM images were taken at magnification up to 10KX to study the microstructure for the materials.

After 28-days of water curing, the specimens were subjected to elevated temperatures of 400°C and 600°C. Broken pieces from heated and unheated specimens were used for FESEM/EDX analysis.

III. RESULTS AND DISCUSSION

A. Compressive Strength

The relative compressive strength for heated and unheated specimens at different curing periods is given in Fig.2 and Fig.

TABLE I
CHEMICAL COMPOSITION OF HAC AND GGBS

Oxide Content	HAC (%)	GGBS (%)
SiO ₂	≤ 6	34.65
Al ₂ O ₃	38-42	13.76
Fe ₂ O ₃	13-17	1.43
CaO	37-40	41.42
MgO	-	7.51
SO ₃	-	0.55
K ₂ O	-	0.37
Na ₂ O	-	0.13

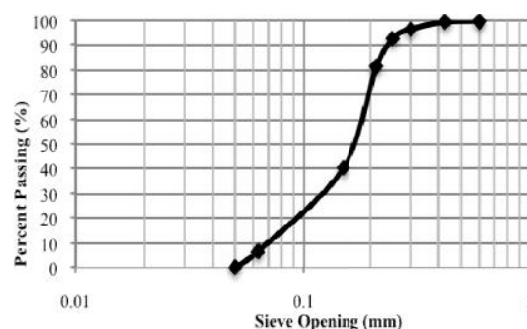


Fig. 1 Sieve Analysis of Silica Sand

TABLE II
MIX PROPORTION FOR THE MORTAR

Mix	Total Binder		HRWRA (%)	W/B	S/HAC
	HAC (%)	GGBFS (%)			
G0	100	0	0.57		
G60	40	60	0.25		
G70	30	70	0.4	0.26	0.8
G80	20	80	0.45		
G85	15	85	0.45		

W/B=Water-Binder Ratio, S/HAC= Sand to HAC Ratio

3. The strength of the control samples cured at 28 days and 56 days was 61.49 MPa and 65.85 MPa respectively. These values were used to normalize (taken as 100%) the strengths of the other samples at respective age.

At 600°C, major drop in compressive strength is found in the mixture with 70% GGBS content after 28 days of curing i.e. approximately 42% of the control specimen compressive strength. However, the least drop is found in the specimen without GGBS after 56 days of curing i.e. 81% of the control specimen compressive strength.

Samples containing 60% GGBS content showed the strength comparable to the control specimen after 28 days of curing at 28°C. For the same samples, the strength was even better after 56 days of curing which might be because of the slow reactivity of GGBS present in the mixture as reported elsewhere [19], which was not much pronounced at 28 days of curing. On the other hand for the samples with 80% and 85% GGBS content, strength is too low because of less HAC content available for hydration and GGBS has an average reactivity to develop strength at early age [20]. However, there

was a marked increase in strength when exposed to 400°C,

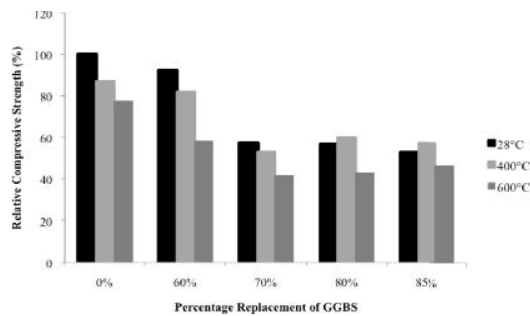


Fig. 2 Relative compressive strength for 28 days cured specimens exposed to different temperatures.

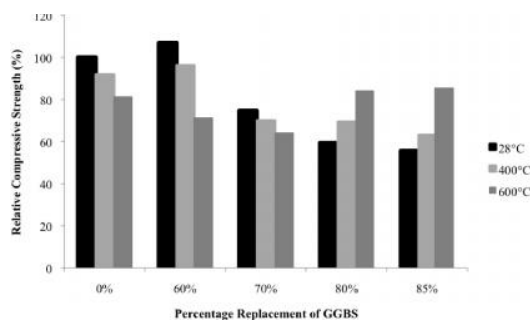


Fig. 3 Relative compressive strength for 56 days cured specimens exposed to different temperatures

which was due to the increase in reactivity of GGBS due to the activation by calcium ions and production of aluminum rich hydrates [21]. Whereas, at 600°C there was a drastic drop in strength due to the release of crystalline water from the matrix, increasing the apparent porosity of the material and hence decreased its strength [22].

Samples cured for 56 days underwent continuous increase in compressive strength after exposure to 400°C and 600°C unlike the samples cured for 28 days. This is corroborated with the average reactivity of GGBS, which developed stable hydration products after 56 days [19] leading to the high strength even at 600°C. This finding is in agreement with the research study performed on ordinary Portland cement replaced by 50%-80% of GGBS [9].

B. Splitting Tensile Strength

The relative splitting tensile strength for heated and unheated specimens at different curing periods is given in Fig.4 and Fig. 5. The strength of the control samples cured at 28 days and 56 days was 5.97 MPa and 6.7 MPa respectively. These values were used to normalize (taken as 100%) the strengths of the other samples at respective age.

In order to inhibit conversion reaction [23] in HAC, lower water-binder ratio of 0.26 was selected, which led to the high strength in control specimens. Due to the high strength and dense microstructure all the specimens underwent explosive spalling at 600°C owing to the vapor pressure built up in the pores resulting in the development of stresses greater than the

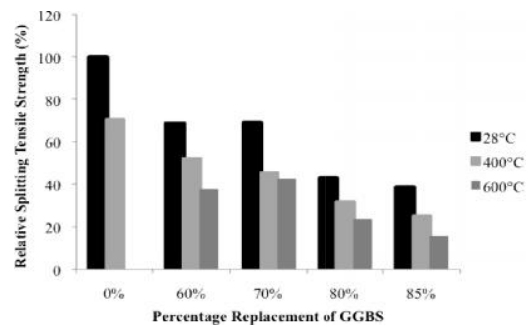


Fig. 4 Relative splitting tensile strength for 28 days cured specimens exposed to different temperatures.

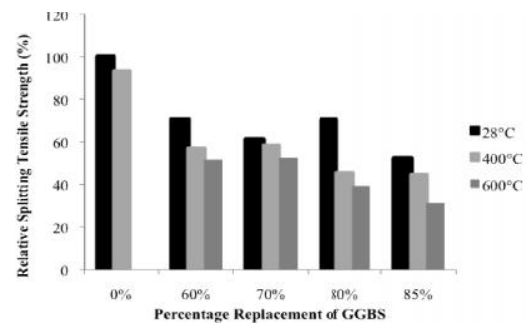


Fig. 5 Relative splitting tensile strength for 56 days cured specimens exposed to different temperatures

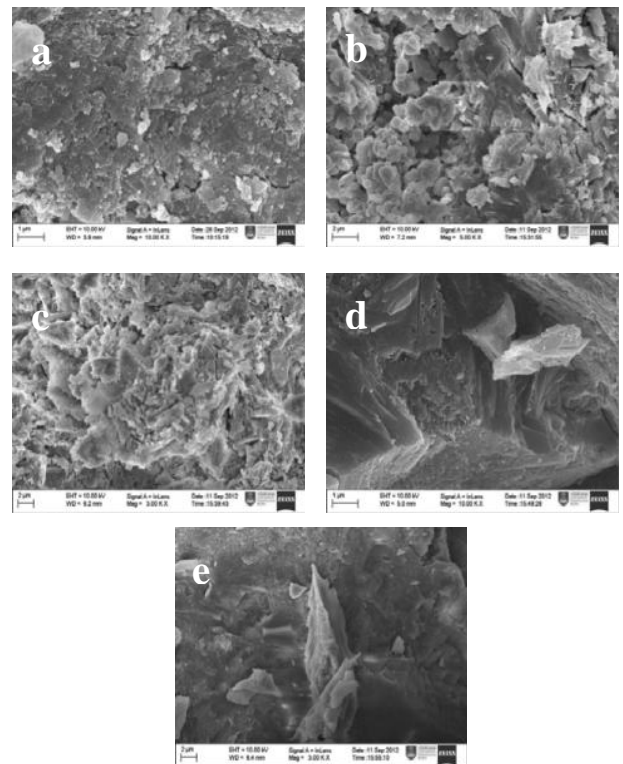


Fig. 6 FESEM Images of samples at room temperature (28°C): a) G0 b) G60 c) G70 d) G80 e) G85

tensile strength of the material [16]. Therefore, tensile strength test could not be performed on the control specimen unlike compressive strength test specimens, where no such phenomenon was observed. This also explains the relation between the size effect of samples and explosive spalling at elevated temperature. Splitting tensile strength of the samples decreased significantly with the increase in percentage of GGBS as compared to the control specimen. With the increase in temperature from 28°C to 400°C and 600°C, drop in tensile strength was observed which may be due to the thermal cracking at elevated temperature.

Splitting tensile strength for the samples containing 60% and 70% of GGBS are almost the same after 28 days of curing at 28°C. Specimens containing 85% of GGBS are found to have the greatest drop in strength at 600°C i.e. 15% of the control sample strength. However, two samples; one without GGBS and the other with 60% GGBS content showed the lowest drop in strength i.e. approximately 52% of the control specimen tensile strength after 56 days of curing.

C. Microstructural analysis

Field Emission Scanning Electron Microscope (FESEM) images were taken at magnifications up to 10 KX to study the microstructure of the materials. The FESEM images for the specimens before and after exposure to high temperatures are shown in Fig. 6a–e, Fig. 7a-e and Fig. 8a-e. Microstructure of control specimen was more compact as compared to other samples contributing towards high strength development of the sample. EDX analysis, shown in Fig. 9, revealed the presence of hydration product of monocalcium aluminates; CA (major phase in HAC), which consists of hexagonal calcium-aluminum hydrates, presumably CAH_{10} and C_2AH_8 [24], [25]. After the control samples exposed to 400°C, the microstructure became porous may be due to the dehydration of hydration phases including CAH_{10} , C_2AH_8 , C_3AH_6 and AH_3 [26]. Further heating to 600°C caused the removal of crystalline water from the phases, which appeared in the microstructure as a platy formation (Fig. 8a).

Mixture with 60% replacement by GGBS after 28 days of curing showed gel structure, presumably gehlenite hydrate (C_2ASH_8) as also shown from FESEM image (Fig. 6b) and the EDX analysis of the image (Fig.10). This is in good agreement with the study performed by Majumdar et al. [27]. The above-mentioned study also explained that gehlenite hydrate is a product of C_3AH_6 (conversion product of HAC) and silica (SiO_2) in GGBS. As this reaction proceeds, C_3AH_6 disappears and certainly inhibits the strength reduction in HAC based THPCC. On the other hand, Ca^{+2} ions in HAC were also consumed to activate GGBS by removing silica, leaving behind the aluminum ions in the paste, which favored the production of other aluminum-silicate rich hydrates [21]. This process was accelerated even at higher temperatures, resulting in the microstructure as shown in Fig. 7b.

Microstructure of the samples having 80% and 85% (Fig. 8d and Fig.8e) of GGBS showed un-reacted particles of GGBS due to the insufficient amount of HAC present in the mixture, which embodied lowest strength to the matrix as

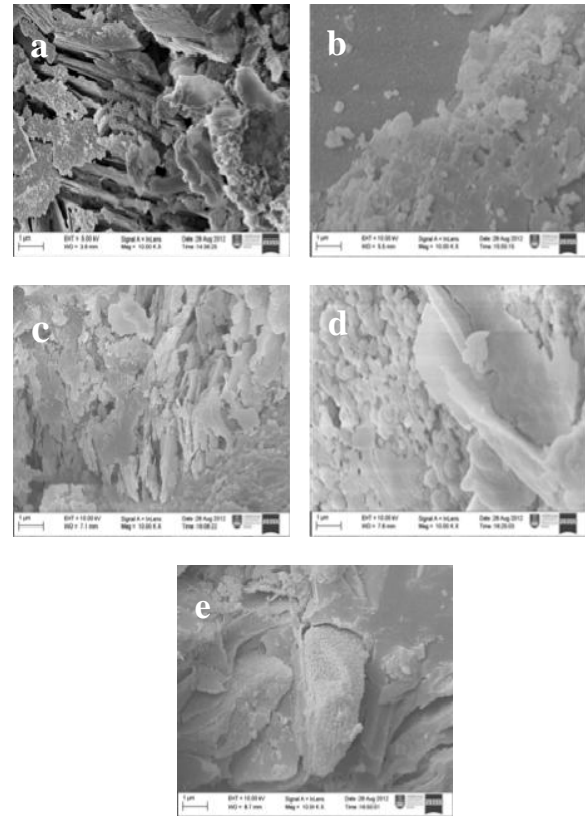


Fig. 7 FESEM Images of samples exposed at 400°C: a) G0 b) G60 c) G70 d) G80 e) G85

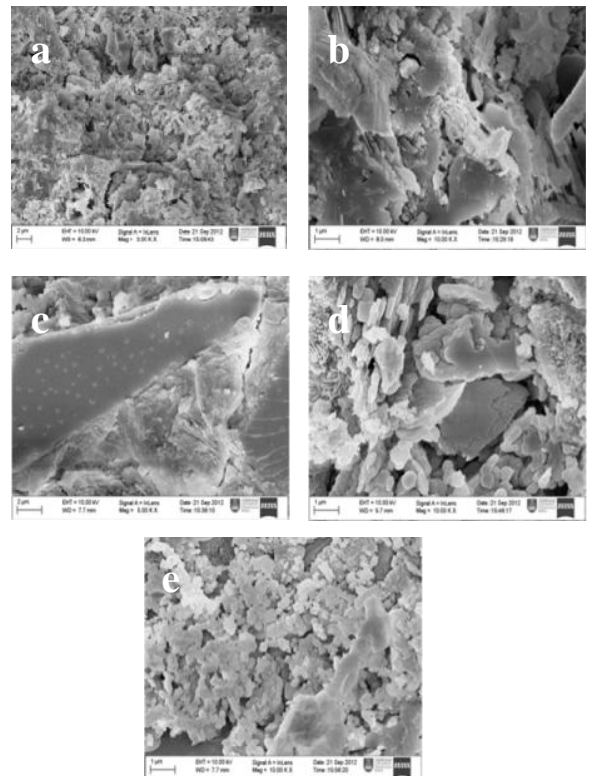


Fig. 8 FESEM Images of samples exposed at 600°C: a) G0 b) G60 c) G70 d) G80 e) G85

compared to other mixtures. Reactivity of GGBS particles increased a bit after exposure to 400°C but was reduced at 600°C due to dehydration of products.

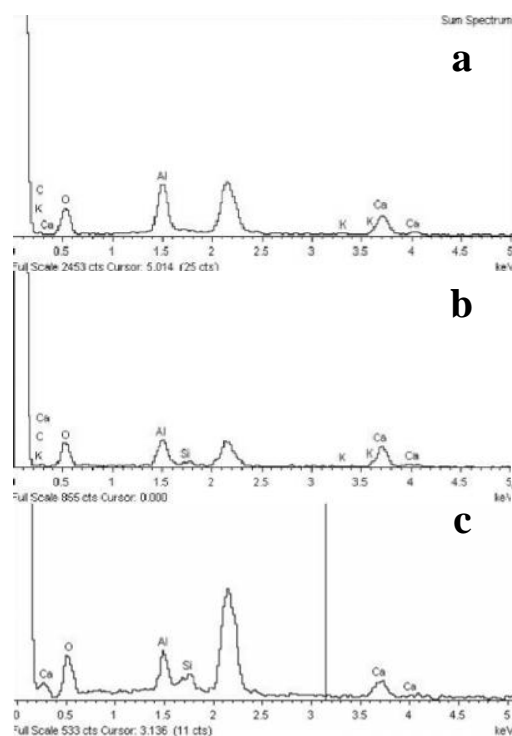


Fig. 9 EDX test results of control sample exposed at: a) 28°C b) 400°C c) 600°C

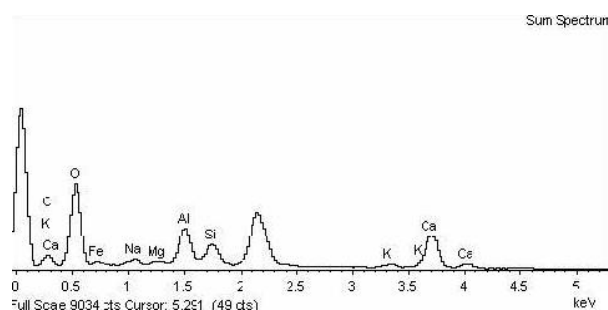


Fig. 10 EDX test results of sample containing 60% GGBS cured for 28 days at 28°C

IV. CONCLUSION

In this paper, the effect of elevated temperature on the mechanical properties and the microstructure of THPCC containing HAC and GGBS were studied. The following conclusions may be interpreted from the experimental results.

- 1- Each of the samples cured at 56 days bear highest strength as compared to the samples cured at 28 days. At 600°C, major drop in compressive strength is found in the mixture with 70% GGBS content after 28 days of curing. However, the least drop is found in the specimen containing only HAC after 56 days of curing
- 2- Splitting tensile strength for the samples containing 60% and 70% of GGBS is almost the same after 28 days of

curing at 28°C. At elevated temperature (400°C and 600°C) there is a continuous drop in tensile strength for all the samples. Specimens containing 85% of GGBS are found to have the greatest drop in strength at 600°C. Interestingly, two samples; one without GGBS and the other with 60% GGBS content showed the lowest drop in strength after 56 days of curing.

- 3- THPCC have a stable microstructure after exposure to 400°C whereas exposure temperatures greater than 400°C causes hydration products to decompose considerably, causing the significant decrease in the mechanical strengths.

ACKNOWLEDGMENT

The authors would like to acknowledge Universiti Teknologi MARA (UiTM) and NED University of Engineering & Technology (NEDUET) for providing all the research facilities required for conducting this research study. YTL cement and BASF Malaysia, who provided GGBS and Glenium 389 respectively, for the experimental work, are also acknowledged. Above all, the authors are thankful to faculty for the future program that provided financial support to conduct this research.

REFERENCES

- [1] V. Kodur, F. Cheng, and T. Wang, "Effect of strength and fiber reinforcement on fire resistance of high-strength concrete columns," *Journal of Structural Engineering*, vol. 129, no. 2, pp. 253-259, 2003.
- [2] S. Qazi, M. S. Hamidah, A. Ibrahim, S.F.A. Rafeeqi and S. Ahmad, "State-of-the-art review- behaviour of thin high performance cementitious composites (THPCC) at elevated temperatures," in 11th International Conference on Concrete Engineering and Technology 2012 (CONCET-2012), 2012, pp. 83-89.
- [3] L. Sarvaranta and E. Mikkola, "Fibre mortar composites under fire conditions: effects of ageing and moisture content of specimens," *Materials and Structures*, pp. 532-538, 1994.
- [4] M. M. Shoaib, S. A. Ahmed, and M. M. Balaha, "Effect of fire and cooling mode on the properties of slag mortars," vol. 31, pp. 1533-1538, 2001.
- [5] M. S. Culfik and T. Özturan, "Effect of elevated temperatures on the residual mechanical properties of high-performance mortar," *Cement and Concrete Research*, vol. 32, no. 5, pp. 809-816, 2002.
- [6] C. Leiva, L. Vilches, J. Vale, and C. Fernandezpereira, "Influence of the type of ash on the fire resistance characteristics of ash-enriched mortars," *Fuel*, vol. 84, no. 11, pp. 1433-1439, 2005.
- [7] S. Aydin and B. Baradan, "Effect of pumice and fly ash incorporation on high temperature resistance of cement based mortars," *Cement and Concrete Research*, vol. 37, no. 6, pp. 988-995, 2007.
- [8] S. Aydin, "Development of a high-temperature-resistant mortar by using slag and pumice," *Fire Safety Journal*, vol. 43, no. 8, pp. 610-617, 2008.
- [9] H. Wang, "The effects of elevated temperature on cement paste containing GGBFS," *Cement and Concrete Composites*, vol. 30, no. 10, pp. 992-999, 2008.
- [10] D. Bentz, M. Peltz, A. Duran-Herrera, P. Valdez, and C. Juarez, "Thermal properties of high-volume fly ash mortars and concretes," *Journal of Building Physics*, vol. 34, no. 3, pp. 263-275, 2010.
- [11] J. Formosa, J. M. Chimenos, A. M. Lacasta, L. Haurie, and J. R. Rosell, "Novel fire-protecting mortars formulated with magnesium by-products," *Cement and Concrete Research*, vol. 41, no. 2, pp. 191-196, 2011.
- [12] J. P. Won, H.-B. Kang, S.-J. Lee, and J.-W. Kang, "Eco-friendly fireproof high-strength polymer cementitious composites," *Construction and Building Materials*, vol. 30, pp. 406-412, May 2012.
- [13] S. Djaknoun, E. Ouedraogo, and A. Ahmed Benyahia, "Characterisation of the behaviour of high performance mortar subjected to high

- temperatures,” *Construction and Building Materials*, vol. 28, no. 1, pp. 176–186, 2012.
- [14] R. K. Ibrahim, R. Hamid, and M. R. Taha, “Fire resistance of high-volume fly ash mortars with nanosilica addition,” *Construction and Building Materials*, vol. 36, pp. 779–786, Nov. 2012.
- [15] ASTM. Standard practice for mechanical mixing of hydraulic cement pastes and mortars of plastic consistency ASTM C305-11. West Conshocken (PA): ASTM International; 2011
- [16] A. Ahmed, “Behavior of FRP-strengthened reinforced concrete beams under fire conditions,” Michigan State University, Michigan, USA, 2010.
- [17] ASTM. Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens ASTM C39/C39M-10. West Conshocken (PA): ASTM International; 2010
- [18] ASTM. Standard Test Method for splitting tensile strength of cylindrical concrete specimens ASTM C496/C496M-11. West Conshocken (PA): ASTM International; 2011
- [19] Y. Fu, F. Ding, and J. Beaudoin, “Temperature dependence of compressive strength of conversion-inhibited high alumina cement concrete,” *ACI materials journal*, vol. 94, no. 6, pp. 540–544, 1997.
- [20] S. Diamond, “Reply to the discussion by S. Chatterji of the paper “Hydration of C12A7 and Granulated Blast furnace Slag,” *Cement and Concrete Research*, vol. 31, no. 11, p. 1655, Nov. 2001.
- [21] A. Majumdar, B. Singh, and R. Edmonds, “Hydration of mixtures of C12A7 and granulated blastfurnace slag,” *Cement and Concrete Research*, vol. 19, pp. 848–856, 1989.
- [22] S. Martinović, M. Vlahović, J. Majstorović, and B. Matović, “Thermal and mechanical properties of high alumina low cement castable,” *Metall. Mater. Eng.*, vol. 18, no. 1, pp. 53–65, 2011.
- [23] H. G. Midgley and A. Midgley, “Conversion of high alumina cement,” *Magazine of Concrete Research*, vol. 27, no. 91, pp. 59–77, 1975.
- [24] N. Ukrainczyk, J. Šipušić, P. Dabić, T. Matusinović, , “Microcalorimetric study on calcium aluminate cement hydration,” in *International conference on Materials, processes, friction and wear-MATRIB’08*, 2008, pp. 382–388.
- [25] D. Madej, J. Szczerba, W. Nocuń-Wezelik, and R. Gajerski, “Hydration of Ca₇ZrAl₆O₁₈ phase,” *Ceramics International*, vol. 38, no. 5, pp. 3821–3827, Jul. 2012.
- [26] P. Myers, “Calcium Aluminate Hydrates - Phases and Structure of Calcium Aluminate Hydrates,” 2002.
- [27] A. J. Majumdar, B. Singh, and R. N. Edmonds, “Hydration of mixtures of ‘ciment fondu’ aluminous cement and granulated blast furnace slag,” *Cement and Concrete Research*, vol. 20, no. 2, pp. 197–208, 1990.