



# Experimentally evaluation of high-performance concrete mixes used for tunnels and containing silica fume and polypropylene fiber after exposed to high temperatures

Seleem S. E. Ahmad

Faculty of Engineering, Zagazig University, Egypt seleemahmad62@yahoo.com, http://orcid.org/0000-0001-9894-0209

# Mohamed M. Elmesiri, Mohamed Fawzy Ahmed

The Petroleum Projects and Technical Consultations Company (PETROJET), New Cairo, Cairo, Egypt M.Elmesiri93@gmail.com, http://orcid.org/0000-0002-2085-8931 Moh.Fawzy.Ahmed@gmail.com, http://orcid.org/0000-0002-5810-9338

# Mohamed Bneni

Faculty of Engineering, University of Zawia, Libya Mohamedbneni@yahoo.com, https://orcid.org/0000-0002-4352-5654

# A.A. ELShami

Housing & Building National Research Centre, Dokki, Giza, Egypt materialhnbrc@yahoo.com, http://orcid.org/0000-0002-9075-7669

ABSTRACT. This work introduces an experimental study to evaluate the effect of elevated temperatures on the mechanical properties of highperformance concrete (HPC) mix with changes in Water-Cementitious ratios, W/(C+SF), Silica Fume percent, SF, and Polypropylene, PP, fiber contents. This mix was typically designed to satisfy the requirements of tunnel concrete. The compressive and indirect tensile strengths were measured at room temperature, RT, and after exposure to 400°C and 800°C. Moreover, SEM micrograph and EDS spot analysis tests were done to evaluate the effect of elevated temperatures. Fifteen mixes of HPC with different ratios of W/(C+SF), SF, and PP fiber were tested. According to the test results, the compressive strength values of design mixes increased significantly after exposure to 400°C. Moreover, using SF = 10%, the results indicated remarkable improvements in the compressive strength at 400°C and 800°C, in the case of the W/(C+SF) ratio of 0.31. On the other hand, the highest effect of the presence of PP fibers was 0.211, depending on variable ratios of the W/(C+SF) ratio and the SF content. In the case of PP=0.106 and SF=10%, the mass loss was higher at exposure to temperatures of 800°C.



**Citation:** Ahmad, S.S.E., Elmesiri, M. M., Ahmed, M. F., Bneni, M., ELSharmi, A. A., Experimentally evaluation of highperformance concrete mixes used for tunnels and containing silica fume and polypropylene fiber after exposed to high temperatures, Frattura ed Integrità Strutturale, 62 (2022) 408-425.

Received: 02.07.2022 Accepted: 27.08.2022 Online first: 03.09.2022 Published: 01.10.2022

**Copyright:** © 2022 This is an open access article under the terms of the CC-BY 4.0, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.

**KEYWORDS.** HPC; Compressive strength; Silica fume; Polypropylene fibers;



Elevated temperatures; Mass loss.

# INTRODUCTION

If igh-performance concrete, HPC, has many advantages to the construction high rise buildings, tunnels, bridges with long spans of reinforced concrete beams, and concrete structures depending on high durability comparing traditional concrete. HPC has low permeability concrete using low water - cementations, also produced with partial replacement of cement by additives like silica fume, SF, fly ash, Nano-silica, and superplasticizers used to produce these kinds of concrete [1,2].

Explosions and fires are some of the most dangerous factors affecting structures and tunnels. K. S. Kulkarni et al. [3] noticed in their study that the fire resistance of HPC does not appear to be as good as that of normal concrete. This research presents the fieldwork to discover the physical characteristics and mechanical properties of partial cement-based HPC subjected to elevated temperatures. Mechanical properties of concrete are changing due to elevated temperatures [4-8], leading to Izabela Hager et al. [9] present an experimental investigation of the effect of the cement type on the mechanical and transport properties of heated concretes. The results indicated that damage increases with permeability, and it follows an exponential type formula for both types of cement. Another study presented an experimental program undertaken to quantify the effect of elevated temperatures of 60, 75, 100, 200, 400, and 600°C on high-strength concrete cylinders for comparing their performance and residual tensile and compressive strengths of concrete, as well as mass loss values after high temperature were determined [8, 10].

Another study on the effect of high temperature on the performance of concrete was implemented by Nesrine Khodja and Hadda Hadjab [11]. They were carried out using two mixtures: normal concrete and HPC with 10% silica fume (SF) replaced by cement weight. The results showed that the relative strength of the concrete samples decreased as the exposure temperature increased and reached about a quarter of its initial strength at 900°C. While Chi-Sun Poon et al. [12] discussed the influence of high temperatures on the strength and durability performance of normal and high-strength pozzolanic concrete containing fly ash, silica, and slag. It was found that the high-strength pozzolanic concrete has a significant loss in permeability-related durability than compressive strength. An extensive experiment was carried out to develop a better ratio of water–cementitious and SF for the compressive and tensile strength of HPC at high temperatures. It has been found that the compressive and tensile strengths increased using SF; this improvement depends on the water–cementitious material ratio of the mix [13]. On the other hand, many studies focused on the addition of fibers to concrete that it's known to have a noticeable positive effect on the mechanical properties of concrete. To prevent the phenomena of explosive spalling, added polypropylene, PP, and fibers to concrete mixes to improve their performance under thermal loads [14-16].



(a) Casting stage

(b) Finishing stage

(c) Demolding

Figure 1: Factory of production concrete rings for the tunnel projects under the Suez Canal.

In recent studies, the mechanical properties of concrete were enhanced with new building materials such as Nano-silica at high temperatures; also, its mass loss is evaluated. According to the test results in concrete mixes incorporating Nano-silica, the compressive strength increases as the temperature was increased up to 400 °C, and it decreases as the temperature increases from 400 to 800 °C. Moreover, the mass loss of the concrete mixes with integrating silica fume and Nano-silica was lower than that of the mixes using silica fume except at 800 °C [17]. Ashwini K and Srinivasa Rao P. [18] investigated the strength and durability properties of concrete using Nano-silica and alcofine at elevated temperatures. The results concluded that the effect of high temperature deteriorated the strength and durability of concrete using Nano-

#### S. S. Ahmad et alii, Frattura ed Integrità Strutturale, 62 (2022) 408-425; DOI: 10.3221/IGF-ESIS.62.28



silica and alccofine. Also, severe loss in strength and a significant increase in water absorption and porosity were observed at 800°C. The main objective of this experimental investigation was to study the effect of variables on the mechanical properties of the reference mix to produce a high-performance concrete mixture for rings of the Suez Canal tunnel, as seen in Fig. 1, at a high temperature. These parameters such as the water-cementitious ratio, W/(C+SF), SF contents, and PP fibers. The present paper takes into consideration the measured compressive and splitting tensile strength results at room temperature, RT, and after exposure to temperatures of 400°C and 800°C. Also, SEM micrograph and EDS spot analysis tests were done to evaluate the effect of elevated temperatures on the performance of HPC.

## **EXPERIMENTAL WORK**

n the experimental work, the task was divided into three main parts as follows:

#### Material properties

The cement used was Type III (CEM 42.5 N) in all HPC mixes. The physical properties of the OPC had confirmed by the Egyptian Standard Specifications [19]. Natural sand was used as a fine aggregate with a fineness modulus, FM, of 2.2, and specific gravity of 2.64 g/cm<sup>3</sup>. Crushed dolomite with a maximum size aggregate of 19 mm and specific gravity of 2.60 g/cm<sup>3</sup> was used as coarse aggregate. The grading curves of coarse and fine aggregates are confirmed to E Specification Limits BS.882 [20] as shown in Fig. 2.

Tab. 1 presents the chemical composition of used cement and SF. Cement chemical composition was completed and obtained by Egypt Lafarge Cement Plant, while SF chemical composition was completed and obtained by Egyptian Ferroalloys Company. Fig. 3 shows the used SF. The PP fibers used in this study with a length of 6-7 mm and density of 0.9 gm/cm<sup>3</sup>, as shown in Fig. 4. MasterGlenium ACE 3383 as high range water-reducing superplasticizer was used to obtain constant workability of all HPC mixes in this work. The amount of superplasticizer was considered as a ratio of cementitious materials (cement+silica fume). All the materials used in this work were supplied and supported by the Petroleum Projects and Technical Consultations Company in Egypt (PETROJET). The physical tests for used materials were also completed in the quality control labs of the company during the construction process of the Suez Canal tunnels. Special acknowledgment to PETROJET Company for the support and the great help in this research by offering resources from staff, laboratories, materials, and data needed to complete the experimental work in this research.



(a) Coarse aggregate, dolomite (b) Fine aggregate, sand Figure 2: Grading curves of used aggregates.

Materials	SiO <sub>2</sub>	$\mathrm{Al}_2\mathrm{O}_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	${ m TiO_2}$	$P_2O_3$	MnO	LOI	SUM	Moisture Content
OPC	24.32	7.65	3.93	50.83	3.64	2.60	0.24	0.41	0.00	0.00	0.00	2.01		
SF	88.1	0.00	1.82	0.97	1.55	0.15	2.56	0.81	0.01	0.05	0.09	2.70	99.51	0.46≤3.0
Table 1: Chemical composition for OPC and SF.														



Figure 3: Silica Fume, SF.



Figure 4: Polypropylene, PP, Fibers.

# Concrete mix preparations and experimental parametric study

The typically reference-designed mix used in the production of HPC for rings of tunnel body of Suez Canal was obtained from PETROJET Company. The mix components for  $1m^3$  in Kg are as follows: (cement: 470, water: 188.5, sand: 695, dolomite aggregate 955, silica fume: 40, polypropylene fiber: 2, superplasticizer: 6). The parametric study in this work was divided into five groups depending on the percent of water-cementitious ratio W/(C+SF), SF and PP fiber. The W/(C+SF) ratios were suggested as 0.25, 0.31 and 0.37 while the SF ratios were 5%, 10% and 15% as addition by weight of cement content, 470 kg/m<sup>3</sup>. The PP fibers were added to concrete mixes by the ratios of 0.106%, 0.211%, and 0.317% from concrete volume. Sand and dolomite amounts were considered as in the reference mix. Therefore, the total number of suggested mixes as a result of these parametric studies was found to be 27 mixes. Consequently, Taguchi optimization technique, as reported in [21], was used to optimize these parameters in all suggested mixes. As a result of this technique, fifteen mixes, as in Table 2, were obtained due to the optimizing process. All specimens were tested at RT and after exposure to 400°C and 800°C. Tab. 2 gives, also the proportions of each concrete mix.

Group	Mix ID	W/(C+SF)	Silica fume, SF	Polypropylene fibers, PP	Superplasticizer %
	M1	0.25	-	-	2.5%
Group – A	M2	0.31	-	-	2.6%
	M3	0.37	-	-	1%
	M2S1	0.31	5%	-	1.1%
Group – B	M2S2	0.31	10%	-	1.1%
	M2S3	0.31	15%	-	1.1%
	M1S1P1	0.25	5%	0.106	2.29%
Group – C	M2S1P2	0.31	5%	0.211	2.2%
	M3S1P3	0.37	5%	0.317	2.2%
	M1S2P3	0.25	10%	0.317	1.1%
Group – D	M2S2P1	0.31	10%	0.106	1.0%
	M3S2P2	0.37	10%	0.211	1.9%
	M1S3P2	0.25	15%	0.211	0.9%
Group – E	M2S3P3	0.31	15%	0.317	2.0%
	M3S3P1	0.37	15%	0.106	1.1%

Table 2: Proportions of HPC mixes, where M1: W/(C+SF) = 0.25, M2: W/(C+SF) = 0.31, M3: W/(C+SF) = 0.37, SF1 (silica fume) = 5%, SF2 (silica fume) = 10%, SF3 (silica fume) = 15%, PP1 (polypropylene fiber) = 0.106, PP2 (polypropylene fiber) = 0.211, PP3 (polypropylene fiber) = 0.317.



# Casting and testing procedures

The preparation of all tested HPC specimens by mixing, casting, and curing are shown in Fig.5. The cube's dimensions of 100×100×100 mm and cylinders dimensions of 100×200 mm were casted to determine the compressive and splitting tensile strengths of concrete, respectively. Three specimens were casted for each test. The specimens were put for 28 days inside the water tanks for curing. After curing, the RT specimens were dried and tested. While the elevated temperature specimens were dried and then placed in an oven to the required temperatures of 400 and 800 °C, as shown in Fig. 5 (e). After that, the specimens were cooled in the air for 24 hours and then tested. The compression and splitting tensile tests were performed using a compression testing machine of 2000 kN, as shown in Fig 6. The used heating oven (1200°C) and compression testing machine were located at the materials lab of Zagazig University in Egypt. While the SEM test was done in the Electron Microscopy Unit at Mansoura University, the steps of sample preparation are preparing samples, cutting, grinding, polishing, impregnation techniques, drying the specimen (vacuum), coating the specimen, and cleaning the surface of the specimen. Fig. 7 shows the schematic construction and working of various components located within the electron column and specimens chamber of the SEM.



Figure 5: Preparation of test specimen, (a) Mixing, (b) Slump test, (c) Cast and compaction, (d) Curing, (e) heating.



Figure 6: Test setup (a) Compression test (b) Splitting tensile test.



Figure 7: Setup of SEM test.

# **RESULTS AND DISCUSSION**

he experimental results of tested specimens will be analyzed and discussed in the following section depending on the parametric factors. Tab.3 summarized the experimental results of compressive and tensile strengths for 28 days at RT and elevated temperatures.

Group	Mix ID	Compress	sive strength,	Fc, (MPa)	Splitting	Splitting tensile strength, $F_{\rm t}$ , (MPa)		
oroup		RT	400°C	800°C	RT	400°C	800°C	
	M1	48.3	63.4	27.9	4.4	4.3	1.2	
Group – A	M2	52.6	58.7	36.4	4.2	3.8	0.8	
	M3	45.1	55.1	25.2	3.1	2.8	0.5	
	M2S1	49.1	56.5	36.1	3.3	3.3	1.0	
Group – B	M2S2	56.6	67.7	41.8	4.2	3.6	0.6	
	M2S3	53.2	54.9	20.2	3.7	3.5	0.4	
	M1S1P1	60.5	74.0	46.3	5.1	4.5	1.1	
Group – C	M2S1P2	70.1	74.3	36.3	3.5	4.3	0.2	
	M3S1P3	42.0	45.6	27.6	4.0	3.3	0.2	
	M1S2P3	41.5	52.6	28.1	4.0	5.0	0.9	
Group – D	M2S2P1	56.3	57.8	41.3	3.1	3.6	0.3	
Ĩ	M3S2P2	60.7	69.4	38.6	2.6	2.4	0.4	
Group – E	M1S3P2	38.8	63.6	31.1	5.2	4.7	3.1	
	M2S3P3	35.2	45.9	18.3	3.2	4.3	1.6	
	M3S3P1	35.5	38.79	24.7	3.2	2.7	0.7	

Table 3: 28 days compressive and splitting tensile strength results for all tested specimens at different temperatures.

#### S. S. Ahmad et alii, Frattura ed Integrità Strutturale, 62 (2022) 408-425; DOI: 10.3221/IGF-ESIS.62.28



# Behavior of mixes without SF and PP fibers at different temperatures

Fig. 8 shows the behavior of 28 days compressive strength,  $F_c$ , for mixes M1, M2, and M3 at RT, 400 °C, and 800 °C. These mixes are without SF and PP fibers. From the figure, we can notice that, at RT, mix M2 (W/(C+SF) = 0.31) gives the highest value of  $F_c$  as compared to M1 and M3. This can attribute to the effect of workability and total water content where M2 shows a high level of workability and sufficient amount of water of hydration in cement past as compared to M1 and M2. This behavior agrees with the vell-known behavior of  $F_c$  with the change in the W/(C+SF) ratio [22]. When these mixes were exposed to a high temperature of 400 °C, similar behavior was found, representing an increase in the value of  $F_c$  as compared to the RT value. The results indicated that the  $F_c$  for M1, M2, and M3 were increased by 31.2%, 11.6%, and 22.1%, respectively, at exposure to 400°C compared to RT. Similar behavior was reported in [17]. On the other hand, when the specimens from these mixes were exposed to a high temperature of  $F_c$  as compared to the RT value. The results of  $F_c$  as compared to the RT value of  $F_c$  as compared to RT. Similar behavior was reported in [17]. On the other hand, when the specimens from these mixes were exposed to a high temperature of  $F_c$  as compared to the RT value. The results of  $F_c$  as compared to the RT value of  $F_c$  as compared to the RT value. The results of  $F_c$  as compared to the RT value of  $F_c$  as compared to RT. Similar behavior was reported in [17]. On the other hand, when the specimens from these mixes were exposed to the RT value. The results of  $F_c$  as compared to the RT value. The results indicated that the  $F_c$  for M1, M2, and M3 were decreased by 42.2%, 30.8%, and 44.1%, respectively, at exposure to 800°C compared to RT. Similar behavior was also found in [22, 23].

The behavior of 28 days splitting tensile strength,  $F_t$ , for the specimens from these mixes is shown in Fig. 9 at temperatures RT, 400°C, and 800°C. Here the  $F_t$  at RT shows higher values as compared to values at 400°C. Moreover, when the specimens were exposed to 800°C, a severe reduction in the value of  $F_t$  was observed as compared to the value at RT. Where the value of  $F_t$  at W/(C+SF) ratio of 0.25, 0.31, and 0.37 decreased from 4.4, 4.2, and 3.1 MPa to reach 1.2, 0.8, and 0.5 MPa, respectively, as the specimens exposed to 800 °C for two hours before testing. Based on the experientially results of this study, the using water - cementations ratio =0.31 has more positive effects on the compressive strength results of high-performance concrete mixtures at temperatures of RT, 400°C, and 800°C compared to other ratios.





Figure 8: Compressive strength of mixes without SF and PP fiber against temperature.

Figure 9: Splitting tensile strength of mixes without SF and PP fiber against temperature.

#### Behavior of mixes without PP fibers at different temperatures

Fig. 10 shows the behavior of 28 days  $F_c$  for mixes M2S1, M2S2, and M2S3 at RT, 400 °C, and 800 °C. These mixes are with SF addition by 5%, 10%, and 15% weight of cement and without PP fibers. From the figure, we can notice that, at RT, mixing M2S2 with 10% SF gives the highest value of  $F_c$  as compared to M2S1 and M2S3. This can attribute to the efficiency of the Pozzolanic reaction resulting from the existence of SF in the mix, which depends on the amount of SF and showed that 10% is an optimum amount. As the percentage of SF decreased to 5%, M2S1 showed less value of  $F_c$  as compared to M2S2 and M2S3. This behavior agrees with the well-known behavior of  $F_c$  with the change in silica fume ratio [24, 25]. When these mixes were exposed to a high temperature of 400 °C, similar behavior as in RT was found, representing an increase in the value of  $F_c$  as compared to the RT value. The results indicated that the  $F_c$  for M2S1, M2S2, and M2S3 were increased by 15.0%, 19.6%, and 3.2%, respectively, when exposed to 400°C compared with RT. Similar



behavior was reported in [26]. On the other hand, when the specimens from these mixes were exposed to a high temperature of 800 °C, similar behavior was found, representing a severe decrease in the value of Fc as compared to the RT value. The results indicated that the Fc for M2S1, M2S2, and M2S3 were decreased by 26.4%, 26.1%, and 62.0%, respectively, when exposed to 800°C compared with RT. Similar behavior was also found in [26].

Fig. 11 presents splitting tensile strength at 28 days for mixes at temperatures RT, 400°C, and 800°C. The results showed that the tensile strength for mixes M2S1, M2S2, and M2S3 recorded the highest values at temperature RT as compared to values at 400 °C and 800 °C. On the other hand, the specimens prepared with SF=10% showed the highest tensile strength value of 4.2 MPa for specimen M2S2 compared to 3.3 MPa and 3.7 MPa for specimens M2S1, SF =5%, and M2S3, SF =15%, respectively when tested at room temperature. Also, the tensile strength value records of 3.6 MPa with SF=10% for specimen M2S2 compared to 3.3 MPa and 3.5 MPa for specimens M2S1, SF=5%, and M2S3, SF=15%, respectively when

exposed to temperature of 400°C. This behavior was also found in mixes without SF and was reported in several previous works [26].





Figure 10: Effect of SF on compressive strength of mixes at different temperatures.

Figure 11: Effect of SF on splitting tensile strength of mixes at different temperatures.

## Effect of PP fiber

The effect of the existence of polypropylene, PP, fibers on the behavior of 28 days  $F_e$  at RT, 400 °C, and 800 °C are shown in Fig. 12. The data was plotted for mixes with percentages of 0.106, 0.211, and 0.317 PP fibers and corresponding mixes without PP fibers. From the figure, we can notice that at RT, the addition of PP fibers improves 28 days  $F_e$  for mixes containing 0.211 PP fibers, M2S1P2, by about 42.7%, as compared to M2S1, the corresponding mix without PP fibers. While for the mix which contains 0.317 of PP fibers, M2S3P3,  $F_e$  value decreased by about 33.8% as compared to mix M2S3. This change can be due to the increase in polypropylene fibers which leads to segregation and is not evenly distributed, causing weak points that reduce the strength of the concrete. When these mixes were exposed to a high temperature of 400 °C, from the figure, we can notice that the addition of PP fibers improved 28 days  $F_e$  for mixes containing 0.211 of PP fibers, M2S1P2, by about 31.5%, as compared to the corresponding mix without PP fibers, also, it's improved  $F_e$  as compared to 0.106 and, 0.317 by about 21.6% and 61.8%, respectively. It is clear that the mix contained P3 = 0.317 due to the expansion and shrinkage, which led to water being withdrawn from the mix needed for the hydration of cement, unable to complete the process of hydration of cement [27]. This led to a decrease in the compressive strength of concrete. On the other hand, at 800 °C, we can notice that there is no significant improvement in  $F_e$  as compared to corresponding mixes without PP fibers.

The effect of the existence of polypropylene fibers on the behavior of 28 days  $F_t$  at RT, 400 °C, and 800 °C are shown in Fig. 13. The data were plotted for mixes with percentages of 0.106, 0.211 and 0.317 PP fibers and corresponding mixes without PP fibers. From the figure, we can notice that, at RT, mix M2S1P2, PP2 = 0.211, gives the highest value of  $F_t$  as

compared to M2S2P1 and M2S3P3 by 12.9% and 9.3%, respectively, also, by about 6.0% as compared to M2S1. When these mixes were exposed to a high temperature of 400 °C, we can notice that mixes M2S1P2 and M2S3P3 give the highest value of  $F_t$  about 4.3MPa; also,  $F_t$  of mixes M2S1P2 and M2S3P3 were improved about by 30.3% and 22.8%, respectively, as compared to corresponding mixes without PP fibers. On the other hand, when these mixes were exposed to a high temperature of 800 °C, similar behavior was found, representing a severe decrease in the value of  $F_t$  as compared to the RT value. The results indicated that the Ft for mixes M2S1P2, M2S2P1, and M2S3P3 were decreased by 94.2%, 90.3%, and 50.0%, respectively, at exposure to 800°C compared to RT.



Figure 12: Effect of PP fiber on compressive strength of mixes at different temperatures.

Figure 13: Effect of PP fiber on splitting tensile strength of mixes at different temperatures.

# Optimizing the effect of W/(C+SF) ratio, SF, and PP fiber at different temperatures

The test results of mixes are optimized in Tab. 4, in which compressive strength and splitting tensile strength were obtained from the present experimental work. The optimum possible mix proportion levels, W/(C+SF) ratio, SF content, and PP fiber content were investigated for the  $F_c$  and  $F_t$  using the Taguchi method [29-31]. Figs. 14-19 show the best parameters series for the designed mixes at temperatures RT, 400°C, and 800°C.

Mix	Temperature °C	Strength	SF content	PP fibers content	W/(C+SF) ratio
M2S1P1	RT	Compressive	5%	0.106	0.31
M1S1P2	400	Compressive	5%	0.211	0.25
M2S3P3	800	Compressive	15%	0.211	0.31
M1S1P3	RT	Splitting tensile	5%	0.317	0.25
M1S1P3	400	Splitting tensile	5%	0.317	0.25
M2S1P2	800	Splitting tensile	5%	0.211	0.31

Table 4: Optimal mix design proportions for high performance concrete.

For the W/(C+SF) ratio, 0.25 gave the highest compressive and splitting tensile strength at room temperature and 400 °C because of the fresh state of the mix. It was homogeneous, and there was no excess water. It is well known that there is an inverse relationship between W/(C+SF) ratio and strengths. The results coming from this research agree with the aforementioned fact that for the compressive strengths and splitting, tensile strengths increased with the reduction of the W/(C+SF) ratio, such as [21, 28]. PP fiber of 0.317 gave the optimum mix at room temperature and 400 °C for the



splitting tensile strength. This is a rational result; also, it is well known in the literature that the fibers contribute to the tensile strength more than the compressive strength for most of the mixes tested. The increase of PP fiber content reduced compressive strengths and splitting tensile strengths. Silica fume as pozzolanic material needs more time to cure to be tested on ages more than 28 days. So, using percentages like 10% and 15%, the silica fume contents inside the mix contribute to the mechanical properties of the concrete as filler. In this study, silica fume of 5% was optimum, while the increase of SF content reduced the compressive strength.



Figure 14: Main Effects Plot for SN ratios for Compressive Strength at RT.



Figure 15: Main Effects Plot for SN ratios for Compressive Strength at 400 °C.



Figure 16: Main Effects Plot for SN ratios for Compressive Strength at 800 C.





Figure 17: Main Effects Plot for SN ratios for Splitting Tensile Strength at RT.



Figure 18: Main Effects Plot for SN ratios for Splitting Tensile Strength at 400 C.



Figure 19: Main Effects Plot for SN ratios for Splitting Tensile Strength at 800 C.

# Mass loss

Fig. 20 displays the relationship between the mass loss and temperatures under the influence of changing the parameters (W/(C+SF), SF, and PP) studied in this study. The loss of moisture resulted from the evaporation of the chemical and physical bond water used to evaluate the mass loss (%). Mass loss (%) of cubic specimens of high-performance concrete mixes by three water - cementations ratios (0.25, 0.31, and 0.37) as shown in Fig. 20(a). The results indicated that the mass



loss for the specimen (W/(C+SF) = 0.25) of the M1 concrete mixes at 400°C and 800 °C were 4.42% and 7.66%, respectively. While, the mass losses of M2 concrete mixes (W/(C+SF) = 0.31) at 400°C, and 800 °C were 4.55%, and 7.0% respectively. In contrast, the mass losses were 4.09% and 7.90% for specimen M3 with W/(C+SF) =0.37 at the same temperature. The results of the mass loss of HPC mix prepared W/(C+SF) ratios of 0.31 with SF replacements by 5%, 10%, and 15% weight of cement at temperatures are presented in Fig. 20 (b).

The results indicated that the mass loss for the specimen of M2S1 with SF1=5% at 400°C and 800 °C were 4.73% and 6.76%, respectively. While the mass loss of specimen M2S2 (SF2=10%) at 400°C and 800 °C were 3.31% and 6.88%, respectively. In contrast, the mass losses were 3.26% and 10.37% for specimen M2S3 with SF=15% at the same temperature. Fig. 20 (c, d, and e) displays the effect of polypropylene (PP) fibers on the mass loss at different temperatures. The results indicated that the mass loss for specimen M2S3P3 of HSC mix decreased using PP=0.317% and SF=15% at tests to 400°C and 800. While the mass loss for the specimen M2S2P1 of HSC mix using PP=0.106% and SF=10% was high at exposure to temperatures of 800°C.



Figure 20: Mass loss (%) of concrete mixes at different elevated temperatures.

## SEM with EDS to analyze the cement paste

SEM and EDS tests were focused on analyses of surfaces of the samples after being crushed at subjected to temperatures RT, 400 °C, and 800 °C. The chemical mixes consisted mainly composed of elements C, O, Mg, Si, and Ca. The Ca (OH)2 and C–S–H gels forms structure for all element. In this work, the study was affected to analyze crushed specimens surface obtained from the M1S2P3 and M3S3P1mixes against different temperatures (RT, 400°C, and 800 °C).

Figs. 21 and 22 present EDS spot analysis and SEM micrographs of specimen M1S2P3 at different temperatures. While the weight (%) of all elements for the specimen M1S2P3 is shown in Fig. 21. The percentage of elements O and Ca increased as the temperature increased up to 400 °C; after that, it decreased at a temperature of 800 °C. It has already been noted that the Si element at 400 °C increased while it decreased at a temperature of 800 °C resulting in a reduction in



# S. S. Ahmad et alii, Frattura ed Integrità Strutturale, 62 (2022) 408-425; DOI: 10.3221/IGF-ESIS.62.28

increasing the concrete strength until failure. Using EDS analysis for M1S2P3 was obtained on the Ca/Si atomic ratio to analyze the chemistry of C–S–H formed in the hydrated cement paste matrix. The chemistry of the C–S–H figuration was significantly dependent on the chemical activity of elements Ca and Si produced during the hydration process. The low Ca/Si ratio was causing the compact and densified microstructure of the cement matrix, as seen in Fig. 22 (b). Hence, the M1S2P3 mix had a slightly decreased Ca/Si ratio at T > 400 °C, which led to an increase in the compressive strength compared with that at RT.

Element	Wt. %	At. %	Ca/Si %
СK	35.43	46.93	3.37
ΟK	43.43	43.43	
Mg K	3.83	2.51	
Si K	2.99	1.69	
Ca K	14.33	5.69	

Element	Wt. %	At. %	Ca/Si %
ΟK	44.88	65.77	1.118
Al K	4.18	3.63	
Si K	15.30	12.78	
КК	2.37	1.42	
Ca K	24.45	14.30	
Fe K	1.82	0.76	
Sn K	3.28	0.65	
ΙL	3.71	0.69	

¢			
00	69	1	
0	2	4	6

(a) At 25°C



(b) At 400°C

Element	Wt. %	At. %	Ca/Si %
СK	35.57	50.67	1.7
O K	31.28	33.46	
Si K	9.64	5.87	
Ca K	23.22	9.91	
Fe K	0.29	0.09	

(c) At 800°C

Figure 21: EDS analysis for specimen M1S2P3 at different temperatures.

For specimen M3S3P1, EDS tests analysis and SEM micrographs at temperatures RT, 400, and 800 are shown in Figs. 23 and 24, respectively. The percentage of elements Ca and Si increased as the temperature increased up to 400 °C; after that, it decreased at a temperature of 800 °C; the result of the reduction is an increasing the concrete strength to failure as with the specimen M3S3P1. EDS analysis for the M3S3P1 appears the Ca/Si atomic ratio to analyze the chemistry of C–S–H formed in the hydrated cement paste matrix. The increasing Ca/Si ratio at 400 °C caused the compact and densified microstructure of the cement matrix, as seen in Fig. 24 (b).



Also, the exposure of the M3S3P1 mix to the elevated temperature of 800 °C caused the highest deterioration in its microstructure, as shown in Fig. 24(c). The SEM image indicates the creation of CSH and CH peaks as key hydration products. Other images of wollastonite (CaOSiO2) also seemed in this image. As the beginning curing time increases, the CH images decrease with increasing hydration time; this is credited to the pozzolanic interaction between CH produced by cement hydration with SF making a marked consumption of the CH with increasing the curing time until 28 days hydration. Also, there is a slight decrease in the SEM of CSH images (CSH is amorphous and ill-crystalline in nature) with increasing hydration ages which is mainly attributed to the that 28-days of curing, particular peaks are identified for some specific hydration products (CSHs, CASHs, CH) in addition to traces of unreacted silicates like C3S and  $\beta$ -C2S. Moreover, distinct peaks for CaCO3 and free quartz are also detected.

On the other hand, the SEM images for OPC with free Al<sub>2</sub>O<sub>3</sub> in FA via internal autoclaving reaction produce extra amounts of CASHs. Firing OPC-FA or SF composite at 400°C leads to thermal destruction for all binding yields, and so sharp depression in the images of SEM of CASHs, CSHs, and CH were observed. After firing at 800°C, the SEM image for CH, CSHs, and CASHs completely vanished, and C3S and b-C2S were extended again, confirming the complete destruction of the binding phases at 800°C that converted into unreacted silicates. Due to the de-carbonation process at 750°C CaO phase is detected. At this temperature, some traces of akermanite (Ca2MgSi2O7) and larnite (Ca2SiO4) phases are also identified in the chemical reactions of free oxides (CaO, SiO2, MgO).





(a) At 25°C





(c) At 800°C

Figure 22: SEM micrographs for specimen M1S2P3 at different temperatures.



Element	Wt. %	At. %	Ca/Si %
СК	27.12	38.92	0.795
ΟK	41.73	44.92	
Al K	2.45	1.57	
Si K	12.84	7.88	
Ca K	14.57	6.27	
Fe K	1.30	0.40	



(a)	At 25°C
(4)	111 25 0

Element	Wt. %	At. %	Ca/Si %
СK	32.41	46.90	1.03
ΟK	31.66	34.39	
Al K	3.07	1.98	
Si K	13.33	8.25	
Ca K	19.53	8.47	



(b) At 400°C

Element	Wt. %	At. %	Ca/Si %
СK	43.23	57.95	0.75
ΟK	27.45	27.63	
Al K	1.93	1.15	
Si K	13.22	7.58	
Ca K	14.16	5.69	



(c) At 800°C Figure 23: EDS analysis for specimen M3S3P1 at different temperatures.







Figure 24: SEM micrographs for specimen M3S3P1 at different temperatures.

# **CONCLUSIONS**

he present experimental study was conducted, including testing of high-performance concrete specimens with the influence of changing the parameters as W/(C+SF), SF, and PP fiber on compressive, tensile strengths and mass loss after exposure to temperatures of RT, 400°C and 800°C. The studies can arrange conclusions as the following:

- 1. The compressive strength values of the present high-performance concrete mixes increased significantly when tested after exposure to 400°C. While compressive strength decreased for all mixes when tested after exposure to 800°C.
- 2. The HPC mixes with a water-cement ratio of 0.31 have positively affected a remarkable increase of the compressive strength values at different temperatures.
- 3. Inclusion of SF=10% as replacement of cement to high-performance concrete mixes, the results indicated that there were remarkable improvements in the compressive strength at RT, 400°C, and 800°C, in the case of the water-cement ratio of 0.31.
- 4. The highest effect of the presence of polypropylene fibers by PP2=0.211 in the case using 5% of SF and W/(C+SF) = 0.31 at temperatures 400°C. While at T = 800°C, the best result of the effect of polypropylene fibers was PP1=0.106%, and W/(C+SF) = 0.25.



- 5. The highest effect of the presence of polypropylene fibers by PP2=0.211 in the case using 10% of SF and W/(C+SF) = 0.37 at temperatures 400°C. While at T = 800°C, the best result of the effect of polypropylene fibers was PP1=0.106%, and W/(C+SF) = 0.31.
- 6. The highest effect of the presence of polypropylene fibers by PP2=0.211 in the case using 15% of SF and W/(C+SF) = 0.25 at temperatures 400°C.
- The results of mass losses were convergent for the M1, M2, and M3 mixes at elevated temperatures of 400°C and 800 °C. At adding silica fume to the mix of M2, the highest mass loss was recorded for specimen M2S3 at a temperature of 800°C.
- 8. Adding polypropylene fibers to the mix of M2 decreased mass loss by PP3 and SF3 at elevated temperatures of 800°C. While mass loss increased using PP1 and SF2 to mix M2 at temperatures of 800°C compared to 400°C.
- 9. The results appeared that the tensile strength for specimens M1, M2, and M3 of HPC mixes recorded the highest values of 4.4 MPa, 4.2MPa, and 3.1MPa, respectively, at temperature RT, in comparison to 400°C and 800°C.
- 10. The tensile strength values were improved for specimens with W/(C+SF) =0.25 that; the best results were at room temperature, 400°C, and 800°C.
- 11. The Ca/Si for specimen M1S2P3 remarkably decreased as temperature increased from RT to 400°C. Then, it increased to 800°C. While the Ca/Si for the specimen M3S3P1slightly increased from RT to 400°C, thereafter, it decreased at T = 800 °C.

#### REFERENCES

- Phan, L. T., Lawson, J. R. and Davis, F. L. (2001). Effects of elevated temperature exposure on heating characteristics, spailing, and residual properties of high performance concrete, Materials and Structures, 34, pp. 83-91. DOI: 10.1007/BF02481556.
- [2] Balakrishnaiah, D., Balaji, K. V. G. D., Srinivasa, Rao. P. and Satyanagh, M. (2017). Study on the Effect of Elevated Temperatures on Residual Compressive Strength of Ternary Blended Concrete using Fly Ash and Micro Silica, International Journal for Research in Applied Science & Engineering Technology, 5, pp.468-471.
- [3] Kulkarni, K. S., Yaragal, S. C. and Babu Narayan, K. S. (2011). Effect of elevated temperatures on mechanical properties of microcement based high performance concrete, International Journal of Applied Engineering and Technology, 1, pp. 24-31.
- [4] Kodur, V. (2014). Properties of Concrete at Elevated Temperatures, Hindawi Publishing Corporation, pp. 1-15. DOI: 10.1155/2014/468510.
- [5] Anupama Krishna, D., Priyadarsini, R. S. and Narayanan, S. (2019). Effect of elevated temperature on the mechanical properties of concrete, Procedia Structural Integrity, 14, pp. 384-394. DOI: 10.1016/j.prostr.2019.05.047.
- [6] Gencel, O. (2011). Effect of elevated temperatures on mechanical properties of high-strength concrete containing varying proportions of hematite, Fire and Materials, 36, pp. 217-230. DOI: 10.1002/fam. 1102.
- [7] Chowdhury, S.H. (2014). Effect of elevated temperature on mechanical properties of high strength concrete, 23rd Australasian Conference on the Mechanics of Structures and Materials, pp. 1077-1082.
- [8] Matsudo, M., Nishida, H., Ohtsuka, T., Hirashima, T. and Ave, T. (2008). Mechanical properties of high strength concrete at high temperatures, Journal of Structural and Construction Engineering, 73(624), pp. 341-347. DOI: 10.3130/aijs.73.341.
- [9] Hager, I., Tracz, T., Choinska, M. and Mróz, K. (2019). Effect of Cement Type on the Mechanical Behavior and Permeability of Concrete Subjected to High Temperatures, Materials, 12, pp. 1-14. DOI: 10.3390/ma12183021.
- [10] Balendran, R. V., Nadeem, A., Maqsood, T. and Leung, H. Y. (2003). Flexural and Split Cylinder Strengths of HSC at Elevated Temperatures, Fire Technology, 39, pp. 47-61. DOI: 10.1023/A:1021727226913.
- [11] Khodja, N. and Hadjab, H. (2018). Effects of Elevated Temperatures on Mechanical's concrete specimen behaviour, 12th International Fatigue Congress, 165, pp. 1-8. DOI: 10.1051/matecconf/201816522010.
- [12] Poon, C.-S., Azhar, S., Anson, M. and Wong, Y.-L. (2001). Comparison of the strength and durability performance of normal and high-strength pozzolanic concretes at elevated temperatures, Cement and Concrete Research, 31, pp. 1291-1300. DOI: 10.1016/S0008-8846(01)00580-4.
- [13] Bhanjaa, S. and Sengupta, B. (2005). Influence of silica fume on the tensile strength of concrete, Cement and Concrete Research, 35, pp. 743-747. DOI: 10.1016/j.cemconres.2004.05.024.



- [14] Shariq, M., Soltanzadeh, F., Masood, A. and Baqi, A. (2013). Tensile strength of normal and high strength concrete with polypropylene fibers at elevated temperature, Conference on Advances in Civil, Structural and Environmental Engineering, pp. 44-48. DOI: 10.3850/ 978-981-07-7965-8\_29.
- [15] Huismann, S., Weise, F., Meng, B. and Schneider, U. (2012). Transient strain of high strength concrete at elevated temperatures and the impact of polypropylene fibers, Materials and Structures, 45, pp. 793-801. DOI: 10.1617/s11527-011-9798-6.
- [16] Bošnjak, J., Sharma, A. and Grauf, K. (2019). Mechanical Properties of Concrete with Steel and Polypropylene Fibres at Elevated Temperatures, Fibers, 7(2), pp.13. DOI: 10.3390/fib7020009.
- [17] Sharaky, I. A., Ahmad, S. S., El-Azab, A. M. and Khalil, H. S. (2021). Strength and Mass Loss Evaluation of HSC with Silica Fume and Nano-Silica Exposed to Elevated Temperatures, Arabian Journal for Science and Engineering, 47, pp. 4187-4209. DOI: 10.1007/s13369-021-06006-7.
- [18] Ashwini K. and Srinivasa Rao, P. (2021). Effect of elevated temperature on strength and durability properties of concrete using nano-silica and alccofine, Research on Engineering Structures & Materials, 8(1), pp. 101-115. DOI: 10.17515/resm2021.281st0419.
- [19] ESS 2421/2005: Egyptian standard specification cement-physical and mechanical tests, (2005).
- [20] BSI 1992 BS-882: Specification for aggregates from natural sources for concrete. BSI, London, UK.
- [21] Warda, M. A., Khalil, H.S., Ahmad S.S.E., Mahdi, I.M. (2020). Optimum sustainable mix proportions of high strength concrete by using Taguchi method, Frattura ed Integrità Structural, 54, pp.211-225. DOI: 10.3221/IGF-ESIS.54.16.
- [22] Hager, I. (2013). Behaviour of cement concrete at high temperature, Bulletin of the polish academy of sciences, 61(1), pp. 145-154. DOI: 10.2478/bpasts-2013-0013
- [23] Rahmouni, Z. EL. and Tebbal, N. (2020). Mechanical Behavior of High-Performance Concrete under Thermal Effect, IntechOpen, pp. 1-22. DOI: 10.5772/intechopen.89916.
- [24] Khajuria, A., Bhardwaj, S., Lalotra, S. (2020). Study on Mechanical Properties of Silica Fume and Alccofine based High Performance Concrete, International Journal for Research in Applied Science & Engineering Technology, 8, pp. 380-383. DOI: 10.22214/ijraset.2020.2058.
- [25] Mansor, A. M., Hamed, A. M. M. and Borg, R. P. (2016). Effect of Silica Fume on High Performance Concrete Strength, Europe and the Mediterranean towards a sustainable built environment, Malta, pp. 65-70. Available at: https://www.um.edu.mt/library/oar/handle/123456789/84499.
- [26] Abdi Moghadam, M. and Izadifard, R. A. (2019). Experimental investigation on the effect of silica fume and zeolite on mechanical and durability properties of concrete at high temperatures, SN Applied Sciences, 1, 682, DOI: 10.1007/s42452-019-0739-2.
- [27] Matar, P. and Zehil, G.-P. (2019). Effects of Polypropylene Fibers on the Physical and Mechanical Properties of Recycled Aggregate Concrete, Journal of Wuhan University of Technology-Mater, 34, pp. 1327-1344. DOI: 10.1007/s11595-019-2196-6.
- [28] Warda, M. A., Ahmed, S.S.E., Mahdi, I.M., Sallam, H.M. and Khalil, H.S. (2022). The Applicability of Topsis- and Fuzzy Topsis-Based Taguchi Optimization Approaches in Obtaining Optimal Fiber-Reinforced Concrete Mix Proportions, MDPI Buildings, 12. DOI: 10.3390/buildings12060796.
- [29] Roy, R.K. (2010). A primer on the Tuguchi method. Society of Manufacturing Engineering, USA.
- [30] Phadke, M.S. (1995). Quality engineering using robust design, Prentice Hall PTR, New York, United States.
- [31] Taguchi, G. (1993). Taguchi on Robust Technology Development, The American Society of Mechanical Engineers, DOI: 10.1115/1.800288.