



Effect of elevated temperature on the mechanical properties of concrete produced with finely ground pumice and silica fume

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ABSTRACT

This study investigated the effect of elevated temperature on the mechanical and physical properties of concrete specimens obtained by substituting cement with finely ground pumice (FGP) at proportions of 5%, 10%, 15% and 20% by weight. To determine the effect of silica fume (SF) additive on the mechanical and physical properties of concrete containing FGP, SF has been added to all series except for the control specimen, which contained 10% cement by weight instead. The specimens were heated in an electric furnace up to 400, 600 and 800 °C and kept at these temperatures for one hour. After the specimens were cooled in the furnace, ultrasonic pulse velocity (UPV), compressive strength and weight loss values were determined. The results demonstrated that adding the mineral admixtures to concrete decreased both unit weight and compressive strength. Additionally, elevating the temperature above 600 °C affected the compressive strength such that the weight loss of concrete was more pronounced for concrete mixtures containing both FGP and SF. These results were also supported by scanning electron microscope (SEM) studies.

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1. Introduction

Concrete can be exposed to elevated temperatures during fire or when it is close to furnaces and reactors. The mechanical properties of concrete, such as strength, elastic modulus and volume deformation, decrease remarkably upon heating resulting in a decrease in the structural quality of concrete. High temperature is one of the most important physical deterioration processes that influence the durability of concrete structures [1] and may result in undesirable structural failures. Therefore, preventative measures such as choosing the right materials should be taken to minimize the harmful effects of high temperature on concrete. The high temperature behavior of concrete is greatly affected by material properties, such as the properties of the aggregate, the cement paste and the aggregate-cement paste bond, as well as the thermal compatibility between the aggregate and cement paste [2,3].

When exposed to high temperature, the chemical composition and physical structure of the concrete change considerably. Dehydration, including the release of chemically bound water from calcium silicate hydrate, becomes significant above 110 °C. The dehydration of the matrix and the thermal expansion of the aggregate give rise to internal stresses, and beginning at 300 °C, micro-cracks begin to pierce through the material [4]. $\text{Ca}(\text{OH})_2$, one

of the most important compounds in cement paste, dissociates at around 530 °C, resulting in the shrinkage of concrete [2,5].

Many studies have been carried out to investigate the effect of the elevated temperature on different concrete specimens, for example studying the response of high-strength concrete to elevated temperature [6–10], the effect of high temperature on concrete reinforced with steel and polypropylene fiber [11,12], the comparison of concretes containing lightweight and normal aggregate exposed to high temperature [13,14], and the determination of the effect of high temperature on the mechanical properties of normal concrete at an early age [15].

Additionally, studies have examined the relationship between the compressive strength and the color change in mortars exposed to high temperature [16], the effect of high temperature on the mechanical properties and pore structure of high performance and normal strength concrete, the permeability of concrete subjected to elevated temperature [17,18], and the spalling behavior of structural elements such as beams or columns exposed to high temperature [19,20].

In addition to studies relating to the mineral additives (silica fume, fly ash, blast furnace slag, pumice and metakaolin) used to increase the mechanical and physical properties of concrete, its durability and its workability, previous studies are also available on the performance of the concrete containing such additives after exposure to elevated temperatures [21–26]. However, not a single study has been conducted on the performance of concrete containing both FGP and SF after exposure to elevated temperatures. Therefore, the aim of this study is to investigate the effect of adding SF (10% by weight of

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cement) on the mechanical behavior and microstructure of concrete obtained by substituting cement with FGP at proportions of 5%, 10%, 15% and 20% by weight after exposure to elevated temperature.

2. Materials and methods

An experimental program was designed to investigate the effect of adding SF (10% by weight of cement) on the mechanical behavior and microstructure of concrete obtained by substituting cement with FGP at proportions of 5%, 10%, 15% and 20% by weight. Three main groups of concrete were produced for this purpose and were coded as C, P and PS. The C series represents control concrete with no mineral admixture. In the P series, cement was replaced with FGP at four proportions (5%, 10%, 15% and 20%) by weight. In the PS series, cement was replaced with SF at a constant ratio of 10% by weight in addition to FGP (5%, 10%, 15% and 20%). The effects of 400, 600 and 800 °C were investigated for each group.

Commercial grade ASTM Type I Portland cement, which is produced in Turkey as CEM I Portland cement, was used in the preparation of all concrete specimens used in this study. The pumice used in this investigation was collected from the Mount Meryem volcano located in the Elazığ province of Turkey. The pumice was very finely ground for hydration reactions and was

then passed through 75 µm sieves for use in concrete preparation. The SF was obtained from the Antalya Electro-Ferrocrome Plant in Turkey. Table 1 compares the chemical and physical properties of the FGP, SF and cement used in the experiments.

High quality river gravel and sand were used as the aggregates, which are widely employed in concrete production (maximum grain size of aggregate=8 mm). The aggregates used in concrete mix proportioning were composed of 65% sand (0–4 mm) and 35% gravel (4–8 mm). Table 2 shows the grading, density and water absorption values of the aggregate. Tap water was used as the mixing water during the preparation of the concrete specimens.

The mixture designs of all the concrete groups were prepared in compliance with ACI 211.1 [27] and are presented in Table 3. Neither plasticizers nor any other chemical admixtures were used.

A total of 180 cube specimens (100 × 100 × 100 mm³) were prepared to determine the mechanical and physical properties of the concretes, such as their compressive strength, porosity, sorptivity and high temperature resistance.

Mixtures prepared according to Table 3 were cast in 100 × 100 × 100 mm³ cube molds. After casting, these specimens were kept in the molds for 24 h at a room temperature of 20 °C. After demolding, these specimens were cured in lime saturated water for 28 days.

2.1. Hardened concrete experiments

Five specimens from each series were used to determine of the compressive strength at 28 days according to ASTM C39 [28]. The compression load was applied at a rate of 3 kN/s using a compression machine with a capacity of 3000 kN.

Three test specimens were used to measure the porosity of each mixture. The specimens were dried in the oven at about 50 °C until a constant mass was achieved and were then placed in desiccators under vacuum for at last 3 h. Finally, the samples were filled with de-aired, distilled water. The porosity was calculated using Eq. (1) according to a previously reported method for measuring the porosity [29,30–32].

$$P = \frac{(W_{sat} - W_{dry})}{(W_{sat} - W_{wat})} 100 \quad (1)$$

where P stands for vacuum saturation porosity (%), W_{sat} represents the weight in air of the saturated specimen, W_{wat} represents the weight in water of the saturated specimen and W_{dry} represents the weight of the oven-dried specimen.

The ultrasonic pulse velocity of three prepared test specimens were automatically determined using a device that measured the amount of time it took for ultrasonic waves to pass between clean specimen surfaces from the wave transmitter to the receiver nozzle, and the wave speed was calculated using Eq. (2) [33]:

$$V = (h/t) \times 10^6 \quad (2)$$

Table 1

Chemical composition of the cement, silica fume and finely ground pumice.

Oxide compounds (mass %)	CEM I 42.5 N	SF	FGP
Silica (SiO ₂)	21.12	93.0–95.0	49.52
Alumina (Al ₂ O ₃)	5.62	0.4–1.4	16.72
Iron oxide (Fe ₂ O ₃)	3.24	0.4–1.0	11.26
Calcium oxide (CaO)	62.94	0.6–1.0	8.26
Magnesia (MgO)	2.73	1.0–1.5	4.54
Sulfur trioxide (SO ₃)	2.30	–	–
Sodium oxide (Na ₂ O)	–	0.1–0.4	–
Potassium oxide (K ₂ O)	–	0.5–1.0	–
Carbon (C)	–	0.8–1.0	–
Sulfur (S)	–	0.1–0.3	–
Loss on ignition	1.78	0.5–1.0	1.68
Density (gr/cm ³)	3.10	2.20	2.8

Table 2

Grading, density and water absorption values of the aggregate.

Sieve size (mm)					
Passage (%)					
8	4	2	1	0.50	0.25
100	65	48	33	19	7
Specific gravity (g/cm ³)			Water absorption (%)		
2.5			3.1		

Table 3

Details of concrete mixes (kg/m³).

Specimen	Water	Fine aggregate (0–4 mm)	Coarse aggregate (4–8 mm)	Cement	FGP	SF
C	200	1043	560	400	–	–
P5	200	1043	560	380	20	–
P10	200	1043	560	360	40	–
P15	200	1043	560	340	60	–
P20	200	1043	560	320	80	–
PS5	200	1043	560	340	20	40
PS10	200	1043	560	320	40	40
PS15	200	1043	560	300	60	40
PS20	200	1043	560	280	80	40

where V =supersonic wave speed (m/s), h =distance from the surface of the concrete specimen at which the ultrasonic wave is transmitted, and the surface at which the wave is received (m), t =time from transmitting to receiving the wave (μ s).

After measuring the UPV values, these specimens were also used for sorptivity measurements. Measurements of capillary sorption were carried out using specimens pre-conditioned in the oven at about 50 °C until a constant mass was achieved. Then, the concrete specimens were cooled down to room temperature. As shown in Fig. 1, test specimens were exposed to the water on one face by placing them on a pan. The water level in the pan was maintained at about 5 mm above the base of the specimens during the experiment. The lower areas on the sides of the specimens were coated with paraffin to achieve unidirectional flow. At certain times, the masses of the specimens were measured using a balance, and then the amount of water absorbed was calculated and normalized with respect to the cross-sectional area of the specimens exposed to the water at various times, such as 0, 5, 10, 20, 30, 60, 180, 360 and 1440 min. The capillary absorption coefficient (k) was obtained using the following equation:

$$\frac{Q}{A} = k\sqrt{t} \quad (3)$$

where Q represents the amount of water absorbed (cm^3); A represents the cross-section of the specimen that was in contact with water (cm^2); t is time (s) and k is the sorptivity coefficient of the specimen ($\text{cm/s}^{1/2}$). To determine the sorptivity coefficient, Q/A was plotted against the square root of time ($\sqrt{t}$), and then k was calculated from the slope of the linear relationship between Q/A and $\sqrt{t}$. This method of measuring the capillary absorption of concrete specimens has been used in many previous studies [29,30,34,35].

The specimens were dried in an oven at about 50 °C until a constant mass was achieved at the end of 28 days. Then, five specimens for each temperature were heated to 400, 600 and 800 °C using a Protherm HLF 150 electrical furnace. The heating rate was set at 2.5 °C/min based on experience from previous research [36–38]. The specimens were held at these temperatures for one hour to achieve a thermal steady state [39].

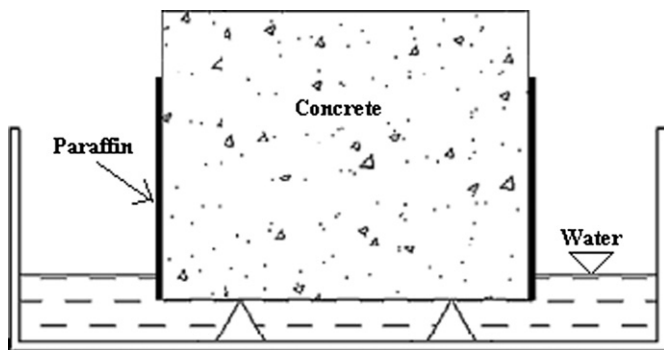


Fig. 1. Measurement of water capillary sorption.

Subsequently, the concrete specimens were cooled down inside the furnace and then tests were conducted one week later to determine the compressive strength and ultrasonic pulse velocity values. The unit weight of the concrete was also determined. The obtained data were compared with the results of the control specimens that were stored at 20 ± 2 °C in the laboratory.

The microscopic analyses of the specimens were performed at the Electron Microscopy Laboratory of Firat University using a Jeol JSM7001F scanning electron microscope.

3. Results and discussion

3.1. Mechanical and physical experiments conducted on the unheated hardened concrete

Table 4 presents the physical and mechanical properties of the hardened concrete samples after 28 days without exposure to high temperatures.

The results in Table 4 indicate that as the amount of the mineral admixtures (FGP and SF) in the concretes increase, the unit weights of the specimens decrease. This outcome is expected because both FGP and SF possess low specific gravities. Therefore, the unit weight of concretes containing FGP or a combination of FGP and SF (double adding) decreases as the percentage of FGP and SF content increases.

Table 4 demonstrates that compressive strength decreased with increasing FGP content, and the strength of the sample with 20% replacement was more than 20% lower than that of the C specimen. This observation is reasonable considering the reduction of cement content in the mix that occurs with increasing FGP content [40,41]. The approximately 50% silica content in FGP (see Table 1) can combine with calcium hydroxide (liberated by the hydrating Portland cement) in the presence of water [42] to form stable compounds such as calcium silicates (C-S-H), which exhibit cementitious properties. Such pozzolanic reactions of FGP contribute to the enhancement of long-term durability and strength. The pozzolanic reaction in pozzolan/cement systems is generally believed to become dominant at ages older than 28 days [43,44]. Therefore, 28 days may not be a sufficient age to observe the pozzolanic effect of FGP.

The addition of SF into cement containing FGP resulted in an increase in the compressive strength. This result may have two underlying reasons, the first being that the silica fume has a much smaller grain size compared to cement and FGP, enabling it to fill in the pores [40]. The other reason is that SF attains strength faster than FGP and cements at early ages such as 28 days. The inclusion of silica fume in the concrete mixture mainly affects the short-term strength of concrete [45]. In their study, Benhood and Ziari [46] explained the resulting increase in compressive strength after entraining SF into cement by emphasizing that SF reacted with calcium hydroxide during the hydration of cement resulting in the formation of fine calcium silicate hydrate (C-S-H), which acted as a filler together with very fine particles of silica fume.

Table 4
Mechanical and physical tests of the specimens.

Experiments	Specimens								
	C	P5	P10	P15	P20	PS5	PS10	PS15	PS20
Unit weight (kg/m^3)	2245	2240	2238	2233	2227	2220	2215	2185	2162
Compressive strength (MPa)	49.71	46.72	44.50	43.51	37.92	46.87	45.84	45.75	38.94
Sorptivity coefficient $\times 10^{-3}$ ($\text{cm/s}^{1/2}$)	1.142	1.184	1.195	1.339	1.356	0.618	0.627	0.695	0.761
Porosity (%)	8.442	8.835	9.362	9.720	9.848	5.385	6.038	6.067	6.649
Ultrasonic pulse velocity (km/s)	4.552	4.396	4.355	4.266	4.139	4.409	4.359	4.300	4.237

As seen in Table 4, the UPV, porosity and sorptivity values support these results as well. In other words, the series with SF, which was less porous compared to the series with FGP, demonstrated lower sorptivity and porosity and higher UPV values. However, even though the silica fume entrained into cements containing FGP led to an increase in the compressive strength of the samples, the compressive strength of the series with SF was still lower than that of the control concrete due to the decrease in the total amount of cement. This result is in good agreement with earlier findings [45].

3.2. Results of mechanical and physical experiments conducted on hardened concrete subjected to high temperature

3.2.1. Weight loss

Fig. 2 shows the weight loss of the concrete specimens resulting from elevated temperature.

As seen in Fig. 2, the unit weights decrease with increasing temperature. Weight reduction takes place in the specimens due to the release of water. Because of the release of bound water from the cement paste, air voids are formed in the concrete. The structural integrity of the specimens deteriorates as confirmed by the increase in weight reduction with increased temperature. The reduction in weight confirms the loss of mass by the concrete material and the increase in the proportion of air voids [3,47]. The average weight loss of the nine samples was 4% at 400 °C, 6% at 600 °C and 8.06% at 800 °C. The results of this study are in agreement with earlier reports [3,13].

In this study, the weight losses in the series containing both SF and FGP were higher than the losses in samples only entrained with FGP especially at 800 °C. Similar to the results of the research conducted by Aköz et al. [48] in which mortars with and without SF were exposed to high temperatures, the concrete samples containing SF experienced higher weight losses than the control concrete samples. Sancak et al. [13] have also reported similar results.

3.2.2. Compressive strength

Fig. 3 presents the relative compressive strength values of the concrete mixtures after exposure to high temperature. The relative strength was calculated as the percentage of strength retained by concrete with respect to the strength of the unheated specimen (20 °C).

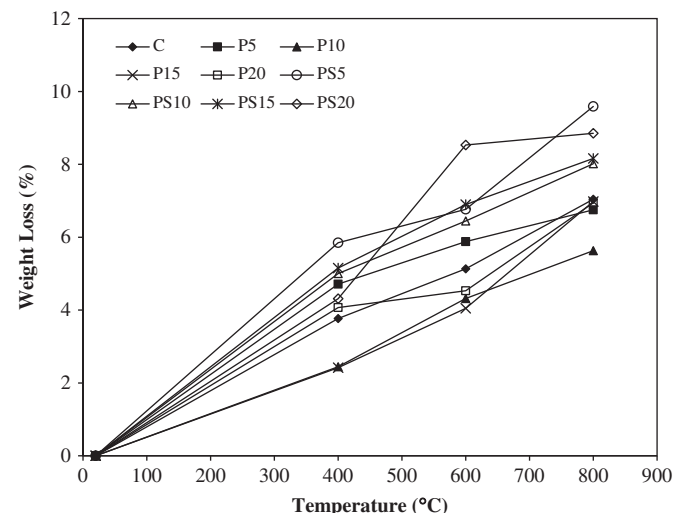


Fig. 2. Weight losses of the concrete specimens.

As seen in Fig. 3, the relative compressive strength increased slightly up to heating at 400 °C (with the exception of the control specimen) and then slightly decreased between 400 and 600 °C.

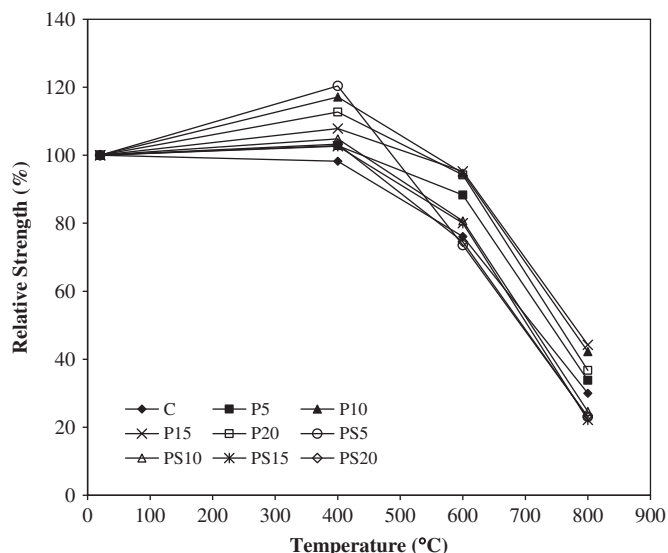


Fig. 3. Relative strength of the concrete specimens subjected to elevated temperatures.

Table 5
Relative strengths of the specimens.

Specimen code	T (°C)	Compressive strength (MPa)	Relative strength (%)
C	20	49.71	100
	400	48.83	98.24
	600	37.82	76.09
	800	14.95	29.99
P5	20	46.72	100
	400	48.05	102.84
	600	41.25	88.30
	800	15.80	33.81
P10	20	44.50	100
	400	52.12	117.12
	600	42.14	94.70
	800	18.80	42.24
P15	20	43.51	100
	400	46.92	107.84
	600	41.45	95.26
	800	19.25	44.26
P20	20	37.92	100
	400	42.75	112.74
	600	35.74	94.24
	800	13.93	36.74
PS5	20	46.87	100
	400	56.43	120.39
	600	34.49	73.59
	800	10.79	23.02
PS10	20	45.84	100
	400	47.96	104.82
	600	36.91	80.68
	800	11.28	24.64
PS15	20	45.75	100
	400	47.06	102.66
	600	36.69	80.04
	800	10.15	22.14
PS20	20	38.94	100
	400	40.22	103.30
	600	29.02	74.53
	800	8.95	22.97

Finally, a sharp reduction in relative strength occurred beyond that point due to the loss of crystal water, leading to the reduction of the $\text{Ca}(\text{OH})_2$ content and changing the morphology and formation of microcracks. For example, the relative concrete strengths were about 80% and 30% when the concrete was exposed to 600 and 800 °C, respectively. The decomposition of calcium hydroxide does not generally occur below 350 °C. The conversion of calcium hydroxide into lime and water vapor during heating is not critical in terms of loss of strength. Nevertheless, this conversion may lead to serious damage due to the expansion of lime during the cooling period. The detrimental effects of $\text{Ca}(\text{OH})_2$ can be eliminated using mineral admixtures such as fly ash and ground granulated blast furnace slag. The pozzolanic reaction between $\text{Ca}(\text{OH})_2$ from cement and reactive SiO_2 from

these mineral admixtures decreases the amount of $\text{Ca}(\text{OH})_2$ in the system [22]. Therefore, the compressive strengths of the concrete specimens containing FGP at 400 °C are higher than those of the concrete specimens containing FGP at 20 °C.

An earlier study by Hertz [49] made similar observations, indicating that the average residual compressive strength of concrete produced with silica fume increased with temperature up to 350 °C and then decreased sharply. Another study showed that concretes enriched with pumice and fly ash do not suffer any loss in strength after exposure to temperatures up to 600 °C [22]. Ariöz [2] observed that the relative compressive strength decreased between 400 and 800 °C. Furthermore, Lin et al. [50] reported that all types of concrete showed severe deterioration at 800 °C due to the decomposition of the C-S-H gel.

As seen in Table 5, the loss of compressive strength in SF entrained series are higher at 800 °C compared to the series without any SF admixtures. This finding is in accordance with the literature. For example, Aköz et al. [48] reported that at temperatures exceeding 600 °C, the proportion of compressive strength loss in mortars containing silica fume is higher than that of the mortars without any SF admixture, while Sancak et al. [13] stated that the loss of compressive strength in the lightweight concrete series containing silica fume increased at 800 °C.

Samples exposed to heating were subjected to UPV tests again after removal from the furnace, and these results were compared with the UPV results taken from the samples at 20 °C. Fig. 4 plots the UPV results.

As seen in Fig. 4, the microstructures of the specimens that were used in the study deteriorated due to increasing temperature, resulting in decreasing UPV values, especially at 800 °C. This decrease in UPV values observed in concrete samples exposed to high temperatures has been explained by Topçu and Demir [51] as follows: degeneration of the C-S-H gel at temperatures above 600 °C increases the amount of air voids in the specimens and decreased the transmission speed of sound waves through the

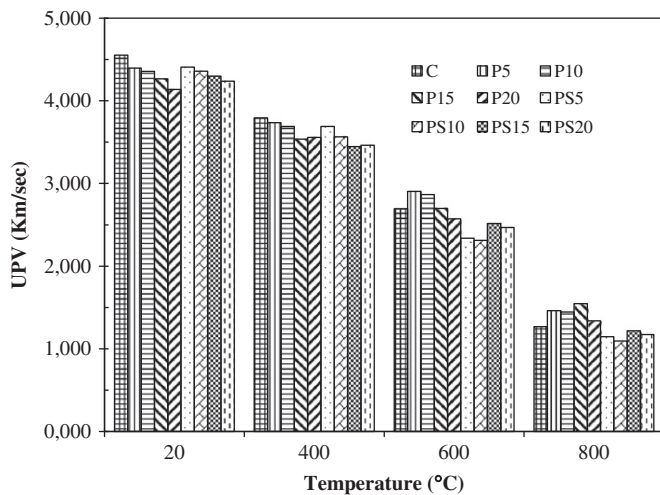


Fig. 4. Change in UPV in the concrete exposed to elevated temperatures.

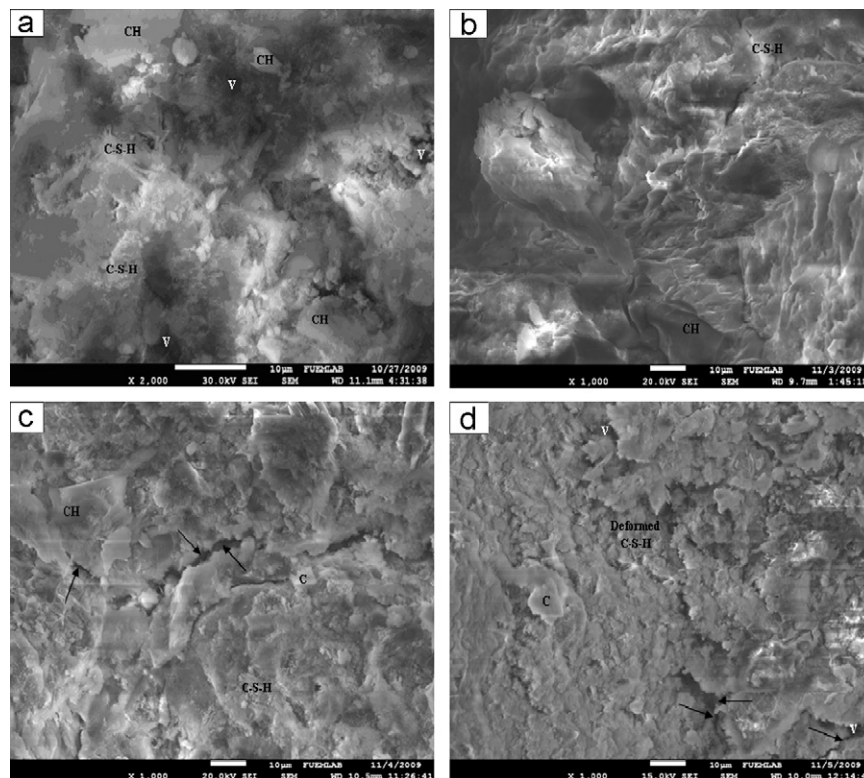


Fig. 5. SEM micrographs of C specimens exposed to (a) 20 °C, (b) 400 °C, (c) 600 °C and (d) 800 °C.

specimens. The decrease in UPV values was observed to be higher for SF entrained concrete samples in our study, especially at 600 and 800 °C. This decrease results from the formation of a more porous structure due to the decomposition of the CSH gel, which is more abundant in samples containing SF.

3.2.3. SEM studies

SEM investigation of hardened concrete demonstrates the distinct morphological changes that originated as a consequence of exposure to elevated temperatures.

Fig. 5(a–d) show, SEM micrographs illustrating the microstructure characteristics of type C concrete at 20, 400, 600 and 800 °C, respectively.

As shown in Fig. 5(a), well-developed hydrated phases exist such as $\text{Ca}(\text{OH})_2$ crystals (marked “CH”) intermixed with C-S-H (marked “CSH”) and voids (marked “V”). Similar findings were previously reported by Handoo et al. [52]. As the temperature increases, Fig. 5(c) and (d) demonstrate the deformation of $\text{Ca}(\text{OH})_2$ crystals and the C-S-H gel, as well as the transformation of $\text{Ca}(\text{OH})_2$ into CaCO_3 (marked “C”) and the formation of microcracks (as indicated by an arrow mark). Along with the increase in temperature, both the density and length of microcracks increased, and the microcracks intermingled with voids due to the increasing porosity.

SEM investigation of concrete specimens revealed massive changes in the morphology of the concrete exposed to 600 and 800 °C. This finding is probably due to the predominance of microcracks, the increased porosity of the concrete due to voids, the deformation of $\text{Ca}(\text{OH})_2$ crystals, and finally disrupted C-S-H phase boundaries. Therefore, the loss of strength observed at higher temperatures may be attributed to the loss of bound water, increased porosity, and consequently, the increased permeability.

Fig. 6 presents SEM observations of the P20 and PS20 concrete specimens, which possess the highest FGP ratios, following treatment 800 °C.

As seen in Fig. 6, because the addition of silica fume into the concrete samples containing FGP leads to the formation of even more CSH gel, the quantity of decomposed CSH at 800 °C is higher in PS20 specimens compared to P20 specimens. After exposure to 600 °C, all the hydrated phases including C-S-H and CH appeared to have amorphous structures due to the loss of their characteristic crystal structures [22]. The SEM results of the microstructures of the concrete specimens containing only FGP or both FGP and SF following exposure to high temperatures are consistent with the results of the mechanical and physical tests performed on these specimens.

4. Conclusions

The following conclusions can be drawn based on the experimental studies presented in this paper.

1. The unit weight of the concrete decreased due to the fact that certain proportions of mineral admixtures (FGP and SF) had been added to the concrete as cement substitutes. This finding is an expected outcome due to the low specific gravity of FGP and SF.
2. Greater amounts of FGP not only resulted in a decrease in compressive strength and ultrasonic pulse velocity values of the concrete but also led to an increase in the porosity and sorptivity values. This result occurred because in the concrete with FGP, a greater reduction occurs in the proportion of cement as the FGP content increases. The pozzolanic reaction in pozzolan/cement is known to become dominant at ages after 28 days; therefore, the 28 day observation in this study may not be sufficient to observe a pozzolanic effect of the FGP.

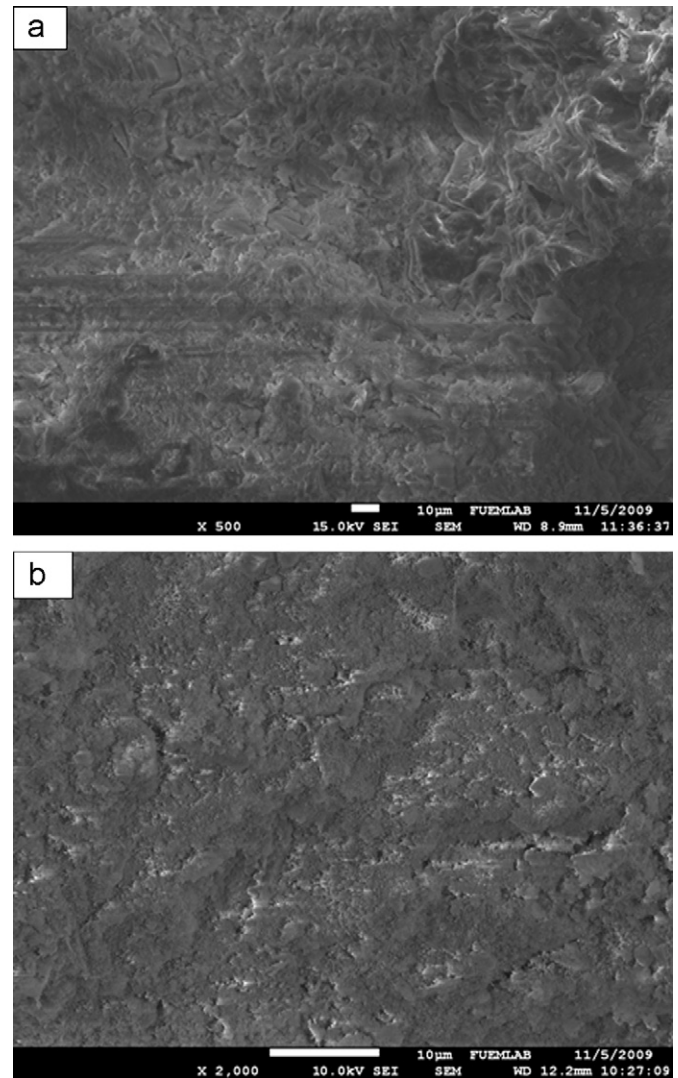


Fig. 6. SEM micrographs of (a) P20 and (b) PS20 specimens exposed to 800 °C.

3. The unit weight of the concrete decreased when it was exposed to elevated temperature. This finding is due to the release of bound water from the cement paste and the occurrence of air voids in the concrete. The highest weight loss occurred in specimens with FGP plus SF that were subjected to 800 °C.
4. The reduction in the compressive strength of concrete was significantly larger for samples exposed to temperatures higher than 600 °C. This result is due to the lost water of crystallization resulting in a reduction of the $\text{Ca}(\text{OH})_2$ content, in addition to the changes in the morphology and the formation of microcracks.
5. The decomposition of $\text{Ca}(\text{OH})_2$ and C-S-H gels, especially at 800 °C, resulted in the total deterioration of concrete. This finding is one of the results of the decreasing compressive strength.
6. The drastic reduction in ultrasonic pulse velocity values between 400 and 800 °C indicates that the physical state of the concrete samples deteriorated rapidly beyond 400 °C.
7. SEM investigations conducted on the specimens confirmed the deformation of well-developed $\text{Ca}(\text{OH})_2$ crystals and the C-S-H gel at temperatures beyond 600 °C. This study demonstrates that the critical temperature for concrete specimens containing FGP or FGP and SF is 600 °C because all the hydrated phases including C-S-H and $\text{Ca}(\text{OH})_2$

appeared to have amorphous structures at this temperature instead of their characteristic crystal structures.

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