










Effects of High Temperature on High-Performance Fine-Grained Concrete Properties

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Abstract. The dense development of high-rise construction in urban areas in Vietnam requires the creation of new concretes with essential properties for fire safety solutions and for high temperature. In this study, the effects of high temperature on high performance fine-grained concrete properties were investigated. Concrete samples were exposed to high temperatures at 300 °C at 4, 5, and 6 h, then cooled to ambient temperature before tests. Two mixtures of Normal Fine-grained Concrete (NFC) and High-Performance Fine-grained Concrete (HPFC) containing 10% silica fume (SF) and 50% bottom ash (BA) were designed in accordance with an absolute volume method. Mass loss, residual compressive strength, and X-ray analysis were performed to investigate the effect of high temperature at different times on the performance of NFC and HPFC. The results of this study showed that the compressive strength of HPFC mixture containing SF and BA obtained is significantly greater than that of the NFC at 300 °C for different curing ages. This can be explained by enhanced reactivity of SiO₂ amorphous in the SF and BA contents in HPFC, which binds more calcium hydroxide at higher temperatures, a percentage of calcium silicate hydrates increases, and the presence of Tobermorite and Xonotlite secondary particles are confirmed through XRD analysis.

Keywords: High temperature · Curing regime · Xonotlite · Tobermorite · Silica fume · Bottom ash · High-performance fine-grained concrete

1 Introduction

The structural stability of concrete and reinforced concrete of high-rise buildings exposed to fire is gaining a significant role in the design process, as users and authorities are increasingly demanding for fire safety solutions [1]. Recent years in Vietnam, with the recent rapid developments of high-rise buildings in urban areas,

where fire safety measures are difficult to implement [2]. In recent times, fires have occurred in high-rise buildings and houses, causing a great deal of damage to property, health, and lives of people. Only in 2017 in this country occurred 4100 fires and is particularly serious fire at Carina Plaza apartment building Ho Chi Minh city in March 2018 [3]. As we know, concrete is a non-combustible material, and, as such, it does not increase the fire load and constitutes a natural barrier preventing the spread of fire [4, 5]. When exposed to fire temperatures concrete does not release any toxic gases or smoke. However, in high-temperature conditions of fires, its internal structure undergoes several physical transformations accompanied with chemical reactions, high result in irrecoverable changes affecting the performance and in the worst case leading to total destruction of the material. Currently, in Vietnam, there is no standardized test methods and is difficult to definitively determine the effect of high-temperature conditions on concrete. The studies [6–8] showed that the fire resistance of concrete is increased with the replacement of brick and steel industry waste such as steel slag, bottom ash, crushed bricks and crushed tiles used as aggregate in concrete. The results of the studies [9–12] show the effects of fly ash, crushed quartz, ground-granulated blast-furnace slag, and other mineral admixture on the mechanical properties of concrete at high temperature. Besides that, some studies [13, 14] the annual amount of industrial waste is more than 150 million tons in Vietnam. In which, metallurgical slag is about $45 \div 55$ million tons, ash and slag TPP is nearly $50 \div 60$ million tons. In 2016, TPP “Vung Ang” produces about 3000 tons of ash and slag waste daily. In addition, an enormous number of gaseous substances and solid particles formed as a result of solid fuel combustion enter the atmosphere through the smokestacks of this power plants, which have caused serious environmental pollution in Vietnam central provinces [15]. The objective of the current study was to investigate the combined effects of high temperature on properties of high-performance fine-grained concrete containing silica fume and bottom ash, which are intended for concrete blocks in the High-Rise Construction.

2 Experimental Details

2.1 Materials

1. The cement used was ordinary Portland cement (OPC) (40 Grade) manufactured at “Tam Diep” factory (Vietnam), the specific weight of 3.15 g/cm^3 . The experimental results of physical and mechanical properties of cement are presented in Table 1 and the results of the chemical compositions are presented in Table 2.

Table 1. Mineralogical composition, physical and mechanical properties of “Tam Diep” Portland cement.

Mineral composition (%)					Time of setting (min)		Compressive strength (MPa)			Standard consistency (%)
C ₃ S	C ₂ S	C ₃ A	C ₄ AF	Other	Initial	Final	3 days	7 days	28 days	
56.15	22.47	5.14	12.25	3.99	142	235	35.1	40.4	52.3	29.5

2. Good quality river sand was used as a fine aggregate, which produced from the quartz sand (QS) of “Lo River” (Vietnam). The fineness modulus $MK = 3.1$, specific gravity and dry density are 2.65 g/cm^3 and 1650 kg/m^3 . The particle size distributions details of fine aggregates are shown in Fig. 1.
3. Bottom Ash (BA) TPP “Vung Ang” (Vietnam) class F and Silica Fume SF-90 (SF90) (Vina Pacific). The chemical composition and physical properties of the BA TPP “Vung Ang” and silica Fume SF-90 are presented in Table 2 and their particle size distribution are presented in Fig. 1.

Table 2. Chemical compositions and physical properties of Portland cement, BA TPP “Vung Ang” and Silica fume SF-90.

Chemical components (wt.%)	BA TPP “Vung Ang”	Silica Fume SF-90	Portland cement
SiO ₂	61.22	91.65	20.4
Al ₂ O ₃	21.17	2.25	4.4
Fe ₂ O ₃	5.85	2.47	5.4
SO ₃	2.42	–	3.4
K ₂ O	1.25	–	1.2
Na ₂ O	1.23	0.55	0.3
MgO	0.57	–	2.5
CaO	1.12	0.51	60.2
P ₂ O ₅	1.03	0.03	–
LOI	4.14	2.54	2.2
Average particle size (μm)	6.15	0.243	8.365
Specific gravity (g/cm ³)	2.35	2.15	3.15
Dry density (kg/m ³)	575	760	1250
Surface area (m ² /g)	5.82	14.45	0.365

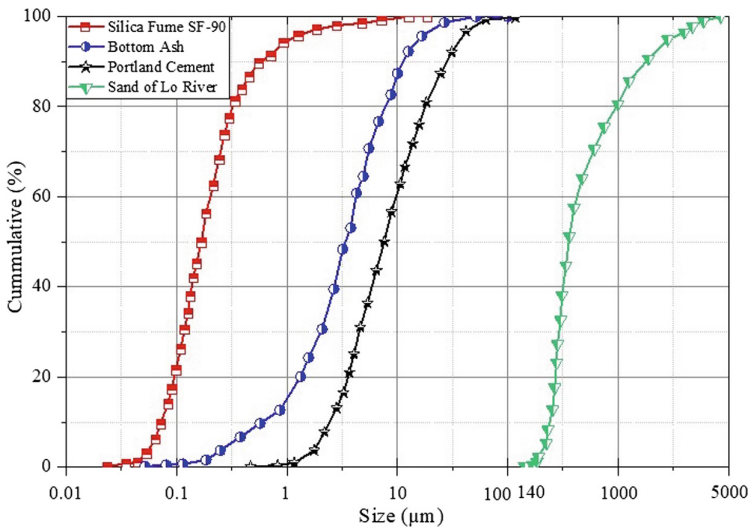


Fig. 1. Sieving analysis of Silica Fume, Portland cement, Bottom Ash and sand of Lo River.

4. Superplasticizer SR 5000F “SilkRoad” (SR5000) (Korea). It is a new generation of chemical additives based on polycarboxylate ethers with a specific weight of 1.1 g/cm^3 at $25 \pm 5 \text{ }^\circ\text{C}$.
5. Ordinary clean tap water (W) was used for both mixing concrete and curing of test specimens.

2.2 Mixture Proportions and Samples Preparation

Absolute Volume Method. The Normal Fine-grained Concrete (NFC) and High-Performance Fine-grained Concrete (HPFC) mixtures were designed in accordance with the absolute volume method and NSC as the control mixture of concrete in this experimental study.

Mix compositions of fine-grained concrete. In the case of this study, the initial ratios of raw materials by weight in concrete mixtures for the production of NFC and HPFC are given in Table 3.

Table 3. Ratios of raw materials used in the preliminary composition.

Ratios value	$\frac{QS}{OPC}$	$\frac{SF}{OPC}$	$\frac{BA}{OPC}$	$\frac{SR5000}{OPC}$	$\frac{W}{OPC}$	The volume of air in concrete
NFC	1.2	0	0	0	0.4	2%
HPFC	1	0.5	0.1	0.02	0.28	2%

Based on the ratios of raw materials in Table 3 and combined with absolute volume method, the preliminary of the material compositions in the dry state of the NFC (control mixture) and HPFC mixtures are shown in Table 4.

Table 4. Mix compositions and properties of fresh fine-grained concrete.

Mixture	Compositions of the concrete mixture (kg/m^3)						Slump flow (cm)	Slump (cm)	Average density (kg/cm^3)
	OPC	SF	BA	SR5000	QS	W			
NFC	826	0	0	0	991	330	–	15	2082.1
HPFC	629	62.9	315	6.29	1006	214	65	–	2170.2

2.3 Test Methods

Workability of Concrete Mixtures. Properties of the fresh concrete, including slump, slump flow, which are determined by standard slump cone with dimensions of $100 \times 200 \times 300 \text{ mm}$ according to ASTM C143 and average density were measured right after mixing and its results are shown in Table 4.

Compressive strength. The compressive strength of NFC and HPFC test was performed after 3, 7, 14, and 28 days on cubic samples of $70 \times 70 \times 70$ mm by Russian standard GOST 10180-2012. These cube samples are demolded after 24 h later casting and placed in a 25 ± 2 °C water curing tank until ages of the experimental plan.

High-temperature condition. Fine-grained Concrete cubes were cured under hot air condition as shown in Fig. 3. The effect of high temperature at 300 °C in 4 h, 5 h and 6 h at different ages were studied for normal fine-grained concrete without mineral additives at the age 28 days. In addition, in this paper are studied the effect of normal and high temperatures, respectively, 25 °C and 300 °C on the properties of NFC and HPFC, which contains 10% silica fume and 50% bottom ash by mass Portland cement at the ages of 3, 7, 14, and 28 days.

Test procedures. In this work, uniaxial compressive tests on NFC and HPFC samples (for each concrete sample) were performed with a constant loading rate of 3000 N/s on system Controls Advantest 9. The reason of choosing 3000 N/s is to keep the loading rate to a minimum in the comparison of test NFC and HPFC results (Fig. 2).



Fig. 2. a - Bottom ash TPP «Vung Ang», b - Determine the weight of concrete specimens.

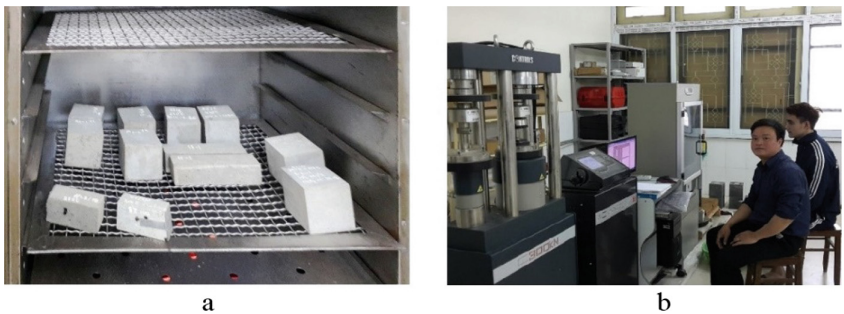


Fig. 3. a - Hot air curing, b - Compressive strength test of Fine-grained concrete.

Characterization of X-ray and of laser granularity. X-ray diffraction was performed with an XRD “Model XDA-D8 Advance” diffractometer of a company “Bruker” sensing with θ/θ configuration and Cu $k\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$). The angular

range from 0 to 90° was performed, to know the different crystalline phases of C-S-H family members developed under high temperature. Laser (Model BT-9300Z, China) granularity analyzer was used to test the granularity distribution of silica fume, bottom ash, and Portland cement.

3 Results and Discussion

3.1 Effect of the High-Temperature Condition in Different Times on Weight and Compressive Strength of Fine-Grained Concrete at Age 28 Days

The temperature of curing regime has a vital role in the development of concrete properties. Weight and compressive strength of NFC and HPFC at age 28 of days obtained at 300 °C for 4, 5, and 6 h is shown in Table 5.

Table 5. Weight and compressive strength of NFC and HPFC at age 28-day at 300 °C in different times.

Type of concrete	The weight of NFC at age 28 days (g)				Compressive strength at age 28 days (MPa)			
	25 °C	At 300 °C for 4 h	At 300 °C for 5 h	At 300 °C for 6 h	25 °C	At 300 °C for 4 h	At 300 °C for 5 h	At 300 °C for 6 h
NFC	777.06	686.61	675.50	668.66	54.40	61.74	67.32	68.85
HPFC	789.15	727.99	708.26	699.27	89.50	09.59	111.98	115.23

Weight Loss of Concrete Specimens. For weight loss assessment, the weights of the concrete cubes were measured before (at 25 °C) and after the exposure to elevated temperatures at 300 °C. The impact of high temperature on the mass loss of both NFC and HPFC containing 50% of bottom ash are shown in Fig. 4.

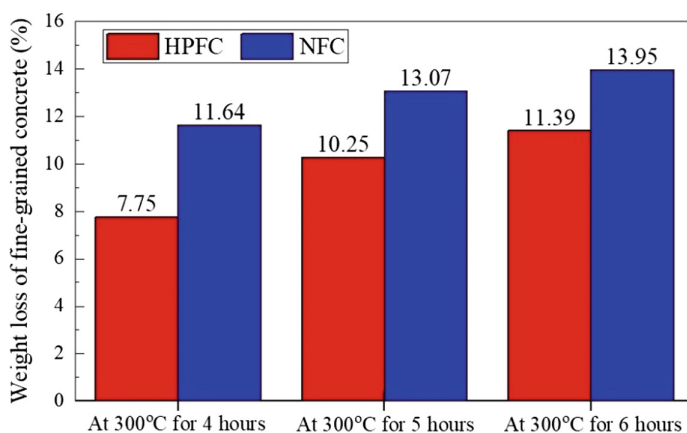
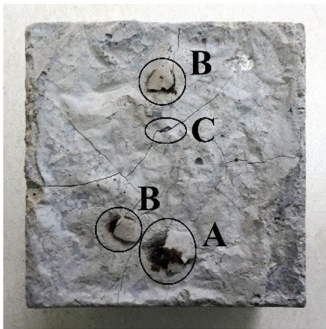


Fig. 4. Weight loss of NFC and HPFC specimens at 300 °C in different times.

The mass loss of all the investigated NFC and HPFC specimens is expressed as a percentage of the original mass at the ambient temperature (25 °C) to the mass after exposure to temperature at 300 °C. Figure 4 further displays that at 300 °C in different times, the weight loss of NFC was the tendency to increase, when compared to HPFC containing bottom ash and silica fume.

In theory, the mass loss in the concrete samples at high temperatures could be attributed to the extra amount of free water, the release of both gel and capillary water, as well as the decomposition of calcareous aggregates, a liberation of carbon dioxide (CO₂) and sloughing off of the concrete surface, which therefore altered the mechanical properties of the concrete [16, 17] (see in Fig. 5).



- A - The decomposition of calcareous aggregates and the liberation of CO₂.
- B - The sloughing off of the concrete surface.
- C - The release of both gel and capillary water.

Fig. 5. The fine-grained concrete surface at 300 °C for 6 h.

Compressive strength development. Data from Table 5 show that as hot air curing at 300 °C for 4, 5 and 6 h, compressive strength is observed to increase, respectively, to 13.49%, 23.75% and 26.56% for NFC, 22.45%, 25.12% and 28.75% for HPFC in comparison with strength at 28 days that obtained at 25 °C. Heat treatment essentially accelerates the pozzolanic reaction of SiO₂ amorphous with Ca(OH)₂, facilitating large development of hydrated products. The strength gain is attributed to well these products under heating environment.

According to [18] reactive powder concrete samples when cured in the temperature range of 100 °C ÷ 300 °C have shown the presence of needle-like the fibrous structure of Tobermorite and Xonotlite, which is a hydrated calcium silicate mineral (Ca₅.Si₆.O₁₆(OH)₂.4H₂O) and (Ca₆.Si₆.O₁₇(OH)₂), respectively. The results of the study [19] also have reported the presence of Tobermorite and Xonotlite at this temperature. It is known that compressive strength, porosity, and permeability of the crystalline calcium silicate hydrate matrix formed at temperatures from 150 °C to 200 °C is dependent in part on the phase formed, for example, Truscottite, Tobermorite, Xonotlite. These crystals become stable and continue their growth, leading to the development of higher strengths of fine-grained concrete, when the heat curing is continued. The relationship between the compressive strength and weight of NFC and HPCF at age 28 days at 300 °C in different times. The given data in Table 5 was observed that the cube compressive strength values could be correlated with the corresponding mass of

concrete. Figure 6 displays a good relationship amongst the ratio (%) of residual compressive strength at 300 °C to original compressive strength at 25 °C and ratio (%) of the weight of concrete at 300 °C to original weight at 25 °C.

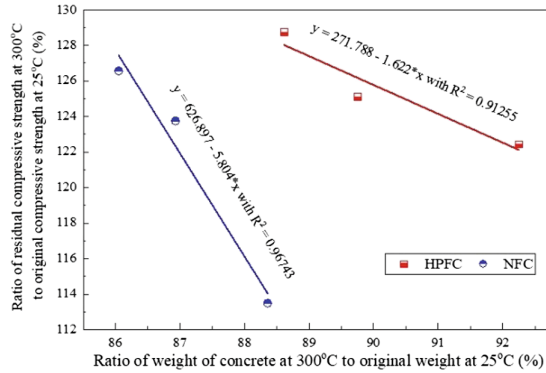


Fig. 6. The relationship between relative compressive strength and weight of NFC and HPCF at age 28-day at 300 °C in different times.

To correlate the experimental data, a linear regression method was applied, resulting in Eqs. (1) and (2) with a correlation coefficient values (R^2) of between approximately 0.91255 and 0.96743 for all samples of NFC and HPFC, which signified good confidence for the relationships. They are as the following:

$$+ \text{ For NFC: } y = 626.897 - 5.804 \cdot x \text{ (with } R^2 = 0.96743) \tag{1}$$

$$+ \text{ ForHPFC : } y = 271.788 - 1.622 \cdot x \text{ (with } R^2 = 0.91255) \tag{2}$$

Where: y is the ratio of residual compressive strength at 300 °C to original strength at 25 °C (%) and x signifies the ratio of the weight of concrete at 300 °C to original weight at 25 °C (%).

3.2 Effect of High-Temperature Condition on Properties of NFC and HPFC at Different Ages

The compressive strength of NFC and HPFC obtained at 25 °C and 300 °C for 6 h and at different curing, ages are shown in Table 6.

Table 6. Compressive strength of NFC and HPFC at different ages at 25 °C and 300 °C for 6 h.

Curing age (days)	The compressive strength of NFC (MPa)			The Compressive strength of HPFC (MPa)		
	At 25 °C	At 300 °C for 6 h	% at 300 °C with at 25 °C	At 25 °C	At 300 °C for 6 h	% at 300 °C with at 25 °C
3	25.7	40.6	157.98	42.2	85.3	202.13
7	41.2	55.3	134.22	71.6	103.3	144.27
14	49.6	63.2	127.42	84.1	111.2	132.22
28	54.4	68.85	126.56	89.5	115.23	128.75

From Table 6, it can be seen that as the temperature of 300 °C for 6 h, the strength of NFC and HPFC observed to quickly increase at early age (3-day) and to slowly increase at late ages (14 and 28 days) in comparison with the strength of concrete that obtained at ambient temperature curing (at 25 °C). For 3 days of hot air curing at 300 °C for 6 h, the HPFC compressive strength was 102.13% higher, when compared to 3 days strength obtained at 25 °C, but for NFC only of 57.98%. However, at 28-day age, compressive strength for HPFC was 28.75% and for NFC was 26.56% higher than those of ambient temperature curing regime.

The relationship existing between the compressive strength and curing age for NFC and HPFC at 25 °C and 300 °C is shown in Fig. 7.

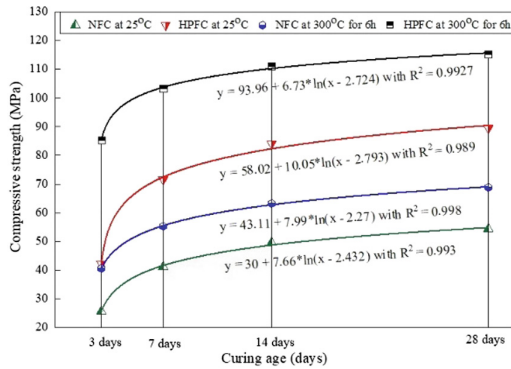


Fig. 7. Compressive strength variation of NFC and HPFC for different curing ages.

The cement concrete strength level and rate of gain, they are a complex function of many different factors. Hydration rate and percentage are two factors related to the used Portland cement [20]. Besides mixture compositions, aggregate type and properties, curing time, different curing regime and method are some factors among the factors affecting both strength level and the gain rate at different ages of concrete [21]. According to data experimental results, for all NFC and HPFC specimens, the relationship between curing age and the compressive strength given by the following formulas:

$$+ \text{ For NFC at } 25^{\circ}\text{C} : y = 30 + 7.66 \cdot \ln(x - 2.432) \text{ with } R^2 = 0.993 \quad (3)$$

$$+ \text{ For HPFC at } 25^{\circ}\text{C} : y = 58.02 + 10.05 \cdot \ln(x - 2.793) \text{ with } R^2 = 0.989 \quad (4)$$

$$+ \text{ For NFC at } 300^{\circ}\text{C} \text{ in } 6 \text{ hours} : y = 43.11 + 7.99 \cdot \ln(x - 2.27) \text{ with } R^2 = 0.998 \quad (5)$$

$$+ \text{ For HPFC at } 300^{\circ}\text{C} \text{ in } 6 \text{ hours} : y = 93.96 + 6.73 \cdot \ln(x - 2.724) \text{ with } R^2 = 0.9927 \quad (6)$$

Where: y is the compressive strength of concrete (MPa) and x is curing age (days). These various equations for prediction of compressive strength derived from curing age also have been proposed by published study [22].

Based on the results of this study, the correlation between curing times and the ratio of residual compressive strength at 300 °C to original compressive strength at 25 °C of NFC and HPFC at different curing times was illustrated in Fig. 8 and shown by Eqs. (7) and (8).

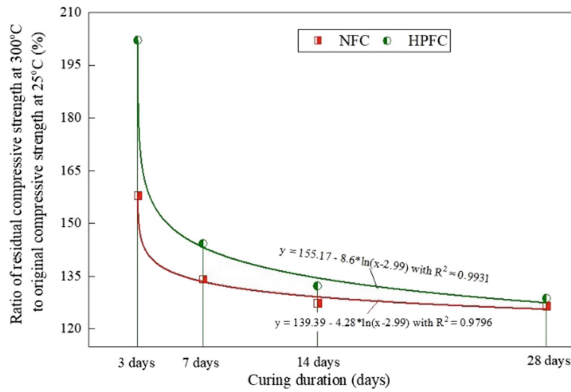


Fig. 8. Effect of high temperatures on compressive strength variation of NFC and HPFC at different curing ages.

$$+ \text{ For NFC : } y = 139.39 - 4.28 \cdot \ln(x - 2.99) \text{ with } R^2 = 0.9796 \quad (7)$$

$$+ \text{ For HPFC: } y = 155.17 - 8.6 \cdot \ln(x - 2.99) \text{ with } R^2 = 0.9931 \quad (8)$$

In Eqs. (7) and (8), x is curing age (days) and y is the ratio (%) of residual compressive strength at 300 °C to original compressive strength at 25 °C of NFC and HPFC. The values R^2 of Eqs. (7) and (8), respectively 0.9796 and 0.9931 represents a very strong negative correlation between the two compared parameters of the curing age and compressive strength variation for the NFC and HPFC, which included amounts of SF-90 and BA.

3.3 X-Ray Diffraction (XRD)

The results of XRD of the NFC and HPFC exposed to high temperatures of 300 °C for 6 h are illustrated in Fig. 9. The XRD analysis shows that at the high temperatures the compositions of Normal Fine-grained Concrete and High-Performance Fine-grained were significantly affected by hot air curing.

From the data given in Fig. 9, it can be seen at 3 days at 300 °C, the presence of calcium hydroxide ($\text{Ca}(\text{OH})_2$) in both NFC and HPFC specimens, but the content of calcium hydroxide in NFC was higher than of HPFC. This can be explained by enhanced reactivity of SiO_2 amorphous in the bottom ash and silica fume contents in HPFC, which binds more calcium hydroxide at higher temperatures.

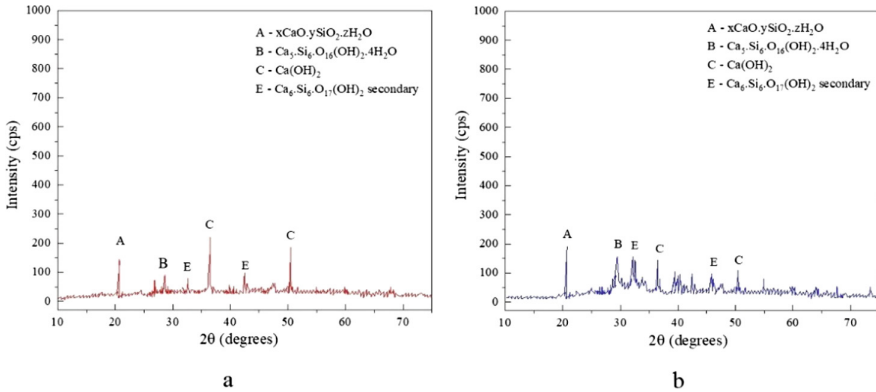


Fig. 9. a - XRD analysis of NFC samples at 3 days at 300 °C, b - XRD analysis of HPFC samples at 3 days at 300 °C.

The reaction of SiO_2 amorphous is consistent with portlandite decreases, the percentage of secondary calcium silicate hydrates increases and the presence of small sponge ball shape of Tobermorite and Xonotlite secondary particles were observed in 3 days at 300 °C of Fine-grained Concrete samples, which are compressive strength enhancing compound in HPFC higher than those of NFC.

4 Conclusions and Future Work

The experimental study was carried out in the laboratory of Hanoi University of Mining and Geology to find out the effect of high temperatures on high performance fine-grained concrete properties. The results of the present study support the following conclusions:

1. Under hot air curing at 300 °C in 4, 5 and 6 h, compressive strength is observed to increase, respectively, to 13.49%, 23.75% and 26.56% for NFC, 22.45%, 25.12% and 28.75% for HPFC in comparison with strength at 28 days that obtained at 25 °C. Still, in that curing regime, weight loss of specimens was also increased, respectively, to 7.75%, 10.25% and 11.39% for HPFC, 11.64%, 13.07% and 13.95% for NFC.
2. At 300 °C for 6 h, the strength of NFC and HPFC observed to quickly increase at early age and to slowly increase at late ages in comparison with the strength of concrete that obtained ambient temperature curing. For 3 days of hot air curing at

300 °C for 6 h, the HPCF compressive strength was 102.13% higher, when compared to 3 days strength obtained at 25 °C, but for NFC only of 57.98%. However, at 28-day age, strength for HPFC was 28.75% and for NFC was 26.56% higher than those of ambient temperature curing regime.

- The results of this study showed that during the increase in curing temperature from 25 °C to 300 °C at different curing times, the strength of NFC and HPFC is also increasing, which shows a promising positive influence of heat treatment to improve microstructure of Fine-grained Concrete in the future work. In addition, in the microstructure of NFC and HPFC, the presence of secondary hydrated products such as Tobermorite and Xonotlite secondary particles are confirmed through XRD analysis.

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