

MIX DESIGN OF HIGH-VOLUME FLY ASH ULTRA HIGH PERFORMANCE CONCRETE

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Abstract

The addition of supplementary cementitious materials (SCMs) to replace cement, especially with a high volume (> 50%), is an effective way to reduce the environmental impact due to the CO₂ emissions generated in the production of ultra-high performance concrete (UHPC). Unfortunately, no official guidelines of UHPC using a high volume of SCMs have been published up to now. This paper proposes a new method of mix design for UHPC using high volume fly ash (HVFA), that is referred to the particle packing optimization of the Compressive Packing Model proposed by F. de Larrard. This proposed method also considers the heat treatment curing duration to maximize the compressive strength of HVFA UHPC. The experimental results using this proposed mix design method show that the optimum fly ash content of 50 wt.% of binder can be used to produce HVFA UHPC with a compressive strength of over 120 MPa and 150 MPa under standard curing and heat treatment, respectively. Moreover, the embodied CO₂ emissions of UHPC reduces 56.4% with addition of 50% FA.

Keywords: high volume fly ash; ultra-high performance concrete; mix design; packing density; curing condition.

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1. Introduction

Ultra-high performance concrete (UHPC) is one of the latest advances in concrete technology, and it addresses the shortcomings of concrete today with outstanding properties such as very high compressive strength, high tensile strength (with adding fiber), high ductility, high strength-to-weight ratio, and excellent durability [1–3]. UHPC has been developed from the 1980s to now with potential applications that demand superior strength and corrosion resistance, such as marine applications or seismic structures [2, 4]. More recently, the use of UHPC has expanded to applications requiring its high strength in narrow profiles, such as bridge spans and building facades in which the material's strength, wear resistance, lighter weight, and lower life cycle costs have been the driving determinates.

Typically, UHPC consists of sand, cement, silica fume, superplasticizer, water, and steel fiber. However, the high cement content (about 800-1000 kg/m³) used to produce UHPC [5] gives a drawback regarding technical, environmental, and economic issues. Using high cement content causes high

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energy consumption and CO₂ emission for UHPC that restrict its more comprehensive applications [6]. Additionally, the high silica fume (SF) content used to produce UHPC, i.e. 20 wt.% of binder, also restricts the applications of UHPC as its limited resource and high price, especially in developing countries such as Vietnam. This gives researchers an idea to develop UHPC using supplementary cementitious materials (SCMs) to reduce cement and substitute silica fume with a similar function. Therefore, further research would be required to efficiently produce UHPC in terms of materials and the resulting mix design method.

One of the potential solutions for this is using high-volume fly ash (HVFA) to produce UHPC. The term of HVFA concrete was first introduced by Malhotra at Canada Center for Mineral and Energy Technology (CANMET), Ottawa with a definition that fly ash (FA) replacement above 50% cement content addresses aforementioned issues [7]. FA levels of around 15–20 wt.% of binder in structural concrete have become accepted worldwide in regular practice, even in some recent attempts to apply in producing UHPC [8, 9]. HVFA concrete (HVFAC), defined as FA replacement above 50%, partly addresses this issue.

The mix design of UHPC can be found elsewhere [10, 11], generally based on the optimization of packing density in combination with the absolute volume principle by selecting the water-to-binder (W/B) ratio or the excess paste volume and then evaluating the properties of fresh and hardened concrete. Le [10] proposed the mix design method based on optimizing granular mixture and excess paste volume. The author selected input materials, W/B ratio, adjusted the sand content, and calculated mix proportions based on the absolute volume principle. The properties of mixtures such as workability, compressive strength, flexural strength, and fracture energy were tested and adjusted until meeting the requirements. Meng [11] proposed the mix design method from optimal binder combination for paste based on the properties of the paste, i.e. workability, rheology, and compressive strength, then selecting the W/B ratio, adjusting the binder-to-sand ratio. This method also considers optimizing the fiber content by testing and adjusting the flow ability and compressive strength. After achieving the required properties of concrete, the method considers the cost of UHPC to finalize the UHPC mix proportion. Yu et al. [12] proposed the mix design method based on the optimized particle packing by employing the modified Andreasen & Andersen model. This mix design method also considers fresh behaviour, mechanical properties, water-permeable porosity, and ecological evaluation. Unfortunately, it seems that no officially proposed UHPC mix design method regarding UHPC using high volume SCMs has been considered at all. How to address the solutions to minimize the resulting drawbacks such as low early-age compressive strength when using high FA content for UHPC is really challenging.

This research is aimed to propose a mix design method of HVFA ultra-high performance concrete (HVFA UHPC) comprising 50 wt.% replacement of cement and have a reasonable workability and 28-day compressive strength above 120 MPa and 150 MPa under standard curing and heat treatment, respectively. The challenges addressed in this research is how to overcome low strength development due to HVFA compared with strength development of normal UHPC. The following principles have been employed to tackle the challenges and to gain the required workability as well as compressive strength: the use of very fine fly ash; adding a small percentage of silica fume to optimize the granular mixture; using superplasticizers to reduce the water-to-binder ratio; and applying heat treatment. With this approach, a new method of mix design is proposed for HVFA UHPC which can be produced with over 50% of cement replaced by fly ash.

2. Proposed mix design procedure and experimental program

The HVFA UHPC mix design method is proposed based on some main steps such as the optimization of the granular mixture, the selection of the W/B ratio, and the application of the absolute volume principle to come up with a preliminary mix proportion of UHPC. After that, based on the preliminary mix proportion, an experimental program with different influencing factors and curing conditions is carried out to find the optimum mix proportion. Based on the experimental results, the maximum FA content is determined corresponding to the desired properties of UHPC. The key steps to design concrete mix HVFA UHPC proposed as follows:

- Step 1: Select the raw materials (sand, cement, SF, FA) and determine their particle size distributions.
- Step 2: Optimize the granular composition following the calculation proposed by method of F. de Larrard. Based on the optimal packing density, the ratios of sand-to-(sand+binder) is determined.
- Step 3: Carry out trial tests with different W/B ratios considering the desired workability and compressive strength, and then select a reasonable W/B ratio. The preliminary mix proportion is determined according to the absolute volume principle. The SP dosage is adjusted to control the required workability, i.e. the flow range, of the concrete mixture. However, the setting time of the mixture should be checked carefully as too high SP dosage used to achieve the workability might cause a prolonged setting. In this case, it is necessary to adjust the W/B ratio, and it is necessary to

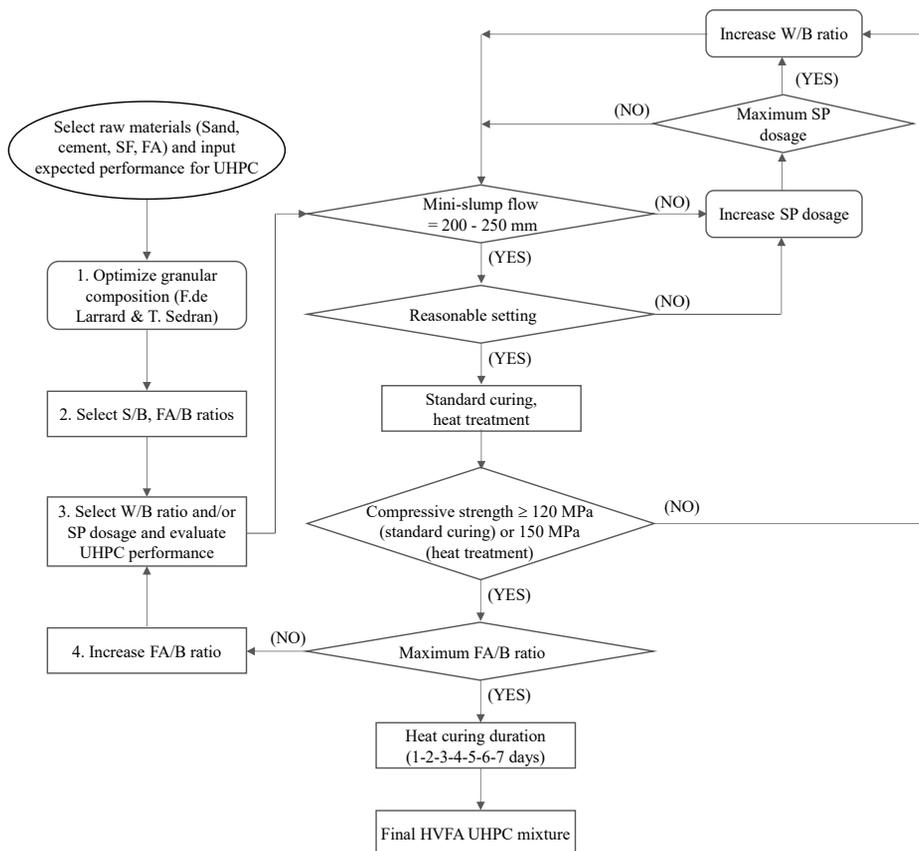


Figure 1. Diagram procedure of mix design methodology for HVFA UHPC

calculate and convert back to concrete mix proportion after achieving workability.

- Step 4: Measure the compressive strength of UHPC under two curing conditions, i.e. standard curing and heat treatment. When the required compressive strength is achieved, i.e. over 120 MPa under standard curing or over 150 MPa under heat treatment, the fly ash-to-binder (FA/B) ratio should be increased and test again from step 2 until the largest FA/B ratio is found and this means that the optimum mix proportion, i.e. the final HVFA UHPC mixture, is obtained. In case that the compressive strength is not attained in either of the two curing conditions, the W/B ratio must be reduced and re-tested from step 2.

- Step 5: Determine the optimal heat treatment duration. When the maximum FA/B ratio is determined with the desired workability and compressive strength, the optimal curing duration is determined by experimental results with the different heat treatment durations from 1 to 7 days after demolding.

The proposed HVFA UHPC mix design method consists main steps as illustrated in Fig. 1.

3. Materials and experimental methodology

3.1. Materials

Portland cement PC50 Nghi Son (according to Vietnamese standard TCVN 2682 [13]), condensed silica fume (SF), and fly ash (FA) conforming to class F specified in ASTM C618 [14] were used for the binder of the UHPC mixture with chemical composition and properties given in Table 1 and Table 2, respectively. Additionally, silica sand with a mean particle size of approximately 300 μm and a density of 2.65 g/cm^3 was used for all mixtures (Table 3). To obtain reliable workability (flow value between 200 mm and 250 mm) of UHPC, a polycarboxylate based-superplasticizer with 30% solid content by mass was employed.

Table 1. Chemical composition of cementitious materials

Material	Chemical composition (%)									
	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	TiO ₂	LOI
Cement	20.30	5.05	3.51	62.81	3.02	-	-	2.00	-	1.83
SF	92.30	1.91	0.86	0.32	0.85	0.38	1.22	0.30	-	1.68
FA	46.82	12.30	25.29	1.20	1.16	1.09	2.50	0.60	0.08	4.04

Table 2. Properties of cementitious materials

Properties	Unit	Cement	SF	FA
Fineness (Blaine)	cm^2/g	4130	N/A	N/A
Mean particle size	μm	10.76	0.15	5.43
Density	g/cm^3	3.15	2.20	2.44
Pozzolanic reactivity index	%	-	111	103
Compressive strength	After 3 days	MPa	36.1	-
	After 28 days	MPa	55.0	-

Table 3. Particle size distribution of raw materials used in this study

Sieve size (mm)	Mean size, d_i (μm)	Fraction retained (%)			
		Sand	Cement	FA	SF
0.5	561				
0.4	447	1			
0.25	316	46.9			
0.18	212	27.6			
0.125	150	23.4			
0.09	106	0.9			
0.063	75.3	0.2	3.2	7.5	
0.032	44.9		6.7	17.9	0
0.016	22.6		19.9	16.3	0.15
0.008	11.3		26.6	22.2	0.24
0.004	5.66		21.7	16.3	0.58
0.002	2.83		4.1	12.8	0.44
0.001	1.41		6.8	5.36	2.31
0.0005	0.707		5.1	1.34	3.8
0.00025	0.354		5.9		26.22
0.00012	0.173				38.57
0.00006	0.085				21.79
0.00003	0.042				5.9

Note: $\lg(d_i) = 0.5(\lg(d_{upper}) + \lg(d_{lower}))$ with d_i : the converted sieve size dupper, d_{lower} : upper and lower sizes after sieving, respectively

3.2. Experimental methodology

All UHPC mixtures were prepared using a 60-liter capacity mixer. To produce UHPC with high-workability, the following mixing procedure depicted in Fig. 2 used: (1) cementitious materials and sand were dry-mixed for 5 min; (2) 70% of the designed unit water was added and then mixed for another 4 min; and (3) the other 30% of the unit water including superplasticizer was added and mixed for 9 min.

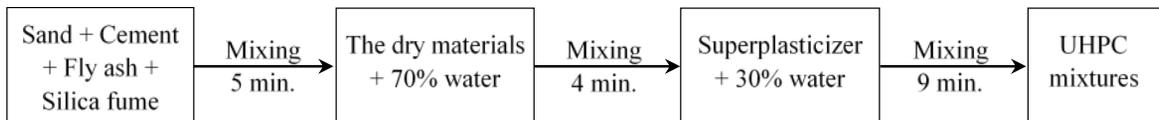


Figure 2. Mixing procedure of UHPC mixtures

The fresh UHPC mixtures were cast into 100 mm cubic molds and stamped 30 times per mold to make samples for examining the compressive strength at different ages. All specimens were cured at a standard curing room with the temperature of $27 \pm 2^\circ\text{C}$ and relative humidity (RH) of exceeding 95%, and then demolded after casting of 24 h. Note that some mixtures using a high SP dosage might cause a longer setting time, these samples should be checked carefully before demolding. After demolding, the specimens were cured under two different curing conditions:

(1) Standard curing condition ($27 \pm 2^\circ\text{C}$, $\text{RH} \geq 95\%$) until testing.

(2) Heat curing condition: in hot water ($90 \pm 3^\circ\text{C}$) for 48 h followed by the standard curing condition until testing.

The compressive strength of specimens cured at the above conditions was tested at ages of 3, 7, 28 complying with ASTM C109 [15]. To examine the effect of heat curing age on 28-day compressive strength of UHPC specimens, more specimens were cured in the hot water condition at different ages varied from 1 to 7 days.

4. Experimental validations

4.1. Select raw materials

The raw materials including sand, cement, SF and FA with their particle size distributions are shown in Table 3.

4.2. Optimize the granular composition

Up to now, some studies for optimizing UHPC granular mixture have been published such as Yu et al. [16] and the F. de Larrard [17]. Yu et al. found a simple calculation when applying the modified Andreasen and Andresen equation, whereas Larrard [17] proposed a new method with a consideration of wall- and loosening-effects of granular classes and a relationship between virtual and real packing. This calculation procedure is complicated, but the packing density can be determined. Therefore, the F. de Larrard method was employed to optimize particle packing densities of sand and cementitious materials in this study.

This model establishes the calculation of the virtual packing density γ_i for a mixture of one particle size class i with unit virtual packing density β_i values is defined by the following equation. It should be noted that the loosening effect a_{ij} and the wall effect b_{ij} where the smaller and bigger particle size influences on the size class i , respectively, are considered.

$$\gamma_i = \frac{\beta_i}{1 - \sum_{j=1}^{i-1} \left[1 - \beta_i + b_{ij}\beta_i \left(1 - \frac{1}{\beta_j} \right) \right] y_j - \sum_{j=i+1}^n \left[1 - a_{ij} \frac{\beta_i}{\beta_j} \right] y_j} \quad (1)$$

where the value y_i represents the volume fraction retained in each size class i .

The *loosening effect* (a_{ij}) describes an effect whereby the introduction of small particles pushes apart larger particles.

$$a_{ij} = \sqrt{1 - \left(1 - \frac{d_j}{d_i} \right)^{1.02}} \quad (2)$$

where d_i, d_j represent the average particle diameters of the i^{th} and j^{th} size class, respectively, in which d_i is larger than d_j ($d_i > d_j$).

The *wall effect* (b_{ij}) describes an effect that larger particles cause the voids in the system. These voids are too small to be filled by other particle classes.

$$b_{ij} = 1 - \left(1 - \frac{d_i}{d_j} \right)^{1.50} \quad (3)$$

After that, the real packing density Φ is established through the compaction index K , a relationship between virtual and real packing, or between the known fractions of each class y_i , the packing density

β_i, γ_i and the real packing density of the mixture. The compaction index K relates each packing density, the real packing density of the mixture increases with the value of K . Thus, for each K index, a real maximum packing density Φ determined in the mixture can be achieved.

$$K = \sum_{i=1}^n K_i = \sum_{i=1}^n \frac{\frac{y_i}{\beta_i}}{\frac{1}{\Phi} - \frac{1}{\gamma_i}} \quad (4)$$

In this study, the compaction index K of the granular mixture was taken as 12.5 [18]. For the mixed granular mixture consisting of sand - cement - SCMs, SCMs including FA and SF, in which the addition of 10% SF (by weight of binder) aimed to improve both fresh and hardened properties of UHPC [19]. Besides, the FA-to-binder (FA/B) ratio ranging from 0 to 0.80 was calculated from each sand-to-binder ratio in the range from 0 to 0.85, the increasing cement-to-binder (C/B) ratios with a step of 0.1, and the fixed SF-to-binder ratio of 0.1. The correlation between the packing density of the granular mixture and the sand-to-(sand+binder) (S/(S+B)) ratio is shown in Fig. 3.

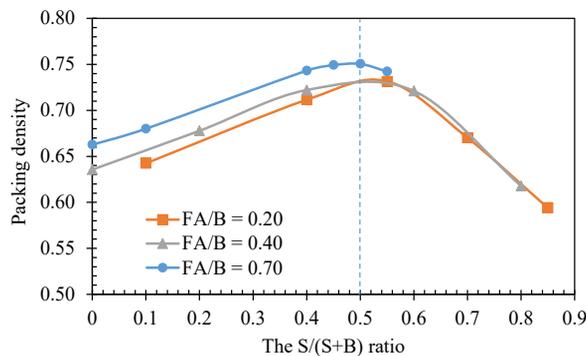


Figure 3. The relationship between packing density and the S/(S+B) ratio

It can be seen that the optimized packing density of granular mixtures is achieved with the S/(S+B) ratio of 0.5 and different the C/B and FA/B ratios. Furthermore, the relationship between packing density of granular mixtures and the FA/B ratio presented in Fig. 4 shows that increasing the FA/B ratio will improve the packing density of granular mixtures at a certain S/(S+B) ratio. However, it is clearly observed that the packing density of granular mixtures achieves the highest values with the S/(S+B) of 0.50.

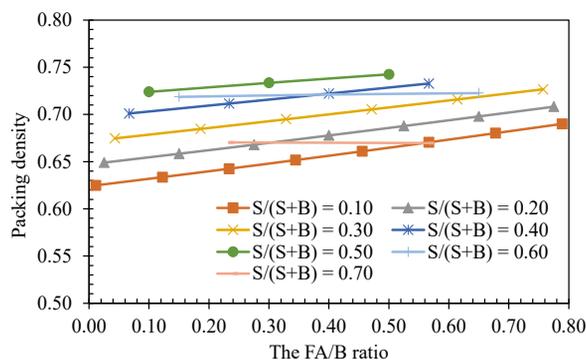


Figure 4. The relationship between packing density and the FA/B ratio

In this study, the S/(S+B) ratio of 0.5 was adopted and the FA-to-binder (FA/B) ratio of (0-70%) was selected to further study. Having combined with 10% SF, consequently, the total amount of SF and FA was investigated in the range of 10% to 80% of binder to optimize the UHPC composition.

4.3. Calculate the mix proportion

The selection of the W/B ratio depends on the desired compressive strength. Based on the suggestions of several researches using available local materials in Vietnam [20–23], some trial tests were conducted, and the W/B ratio was adjusted to attain the desired compressive strength of 120 MPa. It should be noted that the very high SCMs content is used, the W/B ratio should be decreased to offset the reduction of compressive strength of UHPC. Finally, the W/B ratio of 0.16 was selected in this study. The concrete mix proportion was then calculated according to the absolute volume principle.

$$\frac{S}{\rho_S} + \frac{C}{\rho_C} + \frac{SCM}{\rho_{SCM}} + \frac{W}{\rho_W} + \frac{SP}{\rho_{SP}} + \varepsilon = 1000 \quad (5)$$

where S , C , SCM , W , SP is the weight of sand, cement, supplementary cementitious material, water, superplasticizer (kg/m^3); ρ_S , ρ_C , ρ_{SCM} , ρ_W , ρ_{SP} is the specific gravity of sand, cement, supplementary cementitious material, water, superplasticizer (kg/l); ε is the air content of mixture (2% in this study).

Based on the results of the optimized packing density of granular mixtures, eight UHPC mixtures were designed with a sand-to-binder ratio (S/B) of 1.0, i.e. S/(S+B) ratio of 0.5, by weight and the FA ranging from 0 to 70% by weight of binder. The water-to-binder (W/B) ratio was selected at 0.16 by weight. It should be noted that the SF content was fixed at 10% by weight of binder. The flow measurements were controlled in the range of 200–250 mm by adjusting the superplasticizer (SP) dosage, as listed in Table 4.

Table 4. Mix proportions of HVFA UHPC mixtures

Mix. No	W/B (by weight)	S/B (by weight)	FA (wt.% of binder)	SF (wt.% of binder)	SP (wt.% of binder)	C, kg/m^3	FA, kg/m^3	SF, kg/m^3	S, kg/m^3	Water, kg/m^3	SP, kg/m^3
1	0.16	1	0	10	0.58	1036	0	115	1151	182	22.3
2	0.16	1	10	10	0.56	911	114	114	1139	180	21.3
3	0.16	1	20	10	0.52	789	225	113	1127	180	19.5
4	0.16	1	30	10	0.49	669	335	112	1116	179	18.2
5	0.16	1	40	10	0.40	552	442	110	1104	179	14.7
6	0.16	1	50	10	0.27	437	547	109	1093	181	9.8
7	0.16	1	60	10	0.24	325	649	108	1082	180	8.7
8	0.16	1	70	10	0.19	214	750	107	1072	179	6.8

Note: Water considering the additional water content in both the saturated sand with a moisture content of 1.15 wt.% and the liquid superplasticizer with a solid content of 30 wt.%.

4.4. Effect of the FA content on the development of compressive strength of HVFA UHPC

In this step, the development of compressive strength with time of UHPC using different FA contents from 0% to 70% by weight of binder was determined. The experimental results were compared with the desired compressive strength, i.e. over 120 MPa and 150 MPa under standard curing and heat treatment, respectively (Fig. 5). It should be noted that the W/B ratio was kept constantly at 0.16, and the SF content was fixed at 10% by weight of binder.

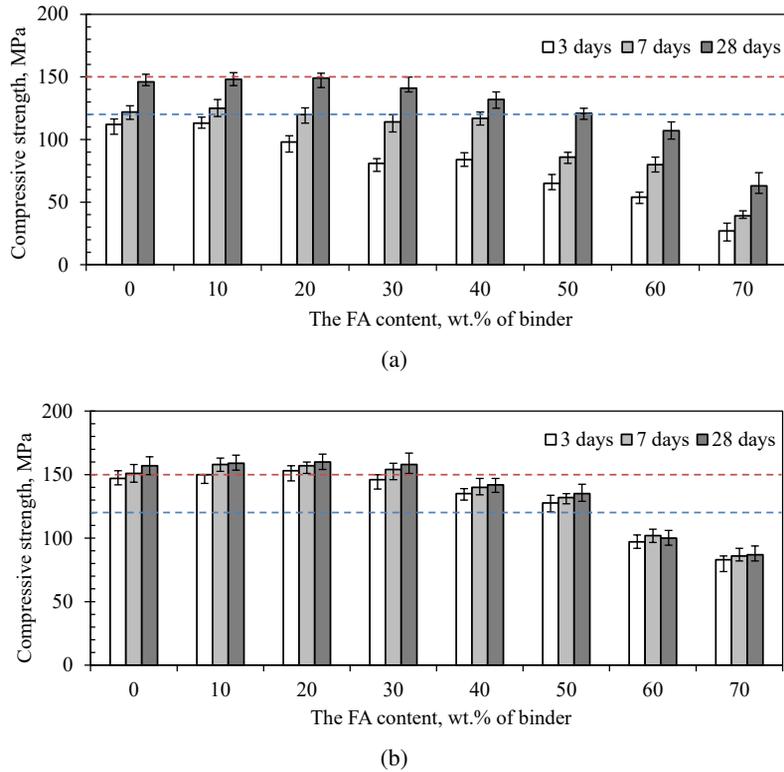


Figure 5. Effect of the FA content on 28-day compressive strength of UHPC with time, W/B = 0.16, 10% SF, under (a) standard curing condition, (b) heat treatment

Experimental results show that the enhancement of the compressive strength of HVFA UHPC requires the early heat treatment. The maximum 28-day compressive strengths of 149 MPa and 160 MPa were achieved when using a combination of 10%SF and 20%FA under standard curing and heat treatment, respectively. In both curing conditions, the compressive strength of concrete reached over 120 MPa with the FA content up to 50%. It means that the total amount of SF and FA, in this case, is 60% by weight of binder, which contributes a great significance to use SCMs to replace cement to produce UHPC.

4.5. Effect of heat treatment duration from 1 to 7 days on the 28-day compressive strength

After the desired compressive strength of HVFA UHPC was obtained, the effect of heat treatment duration on the 28-day compressive strength was studied to determine the maximum FA content to produce HVFA UHPC because the heat curing condition can enhance the microstructure and result in increasing strengths [1, 24, 25]. In this study, the desired compressive strength was either 120 MPa under standard curing or 150 MPa under heat treatment, and the achieved experimental results are shown in Fig. 6.

It can be observed from Fig. 6 that extension of heat treatment time enhances the compressive strength of UHPC but not significant after 1-2 days. Compared with the desired compressive strength, it is recommended as at least two days under heat treatment from the experimental results.

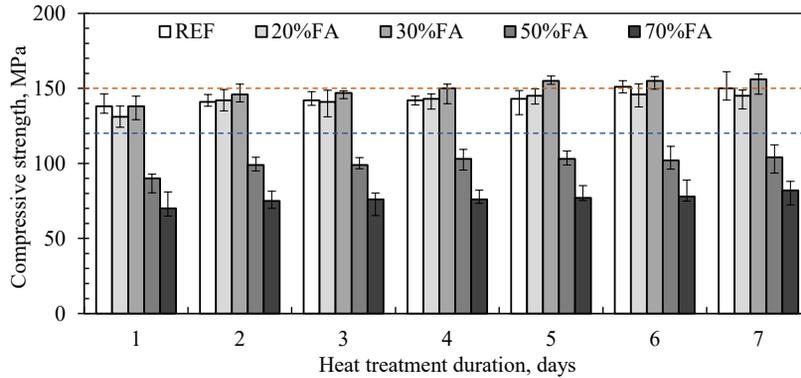


Figure 6. Effect of the heat curing duration on 28-day compressive strength of HVFA UHPC, W/B = 0.16, SF fixed at 10%

4.6. Embodied CO₂ emissions of HVFA UHPC

The replacement of HVFA for cement in producing UHPC gives a positive influence on the environmental impact due to that UHPC consumes a high amount of cement as aforementioned. In this study, the environmental benefit of using HVFA to produce UHPC can be assessed by the embodied CO₂ emissions of concrete mixtures. The calculation method following to the life cycle assessment (LCA) approach presented by Yang *et al.* [26]. The LCA procedure is specified and meets the minimum requirements, i.e. from the cradle to the gate of concrete plant, of the ISO 14040 series. In this calculation, CO₂ inventories of raw materials, i.e., sand, cement, SP, water, SF, FA, were taken from studies by Shi *et al.* [27] and King [28], as given in Table 5. Note that the transportation of each component was not considered in this calculation.

Table 5. Embodied e-CO₂ of components [26, 27] (Unit: CO₂-kg/kg)

Cement	FA	SF	Quart sand	Water	SP
0.83	0.009	0.028	0.01	0	0.72

From the UHPC mix proportions in Table 4 and embodied e-CO₂ of components in Table 5, embodied CO₂ emissions of UHPC using different FA contents under standard curing were calculated and the results are shown in Fig. 7.

Regarding the heat treatment, the total heat treatment duration of 54 h was applied, including the gradual heating time of 6 h and the 48 h constant temperature time. The CO₂ emission for heat treatment of 2.49 CO₂/(m³.h) was chosen [27] in this study. Therefore, the total CO₂ emission of 134.46 kg/m³ was added to the CO₂ emission under the standard curing condition for all samples under the heat treatment condition.

It can be observed in Fig. 7 that progressively increasing FA was found to decrease the embodied CO₂ emissions of UHPC. The replacement of 50% FA reduces 56.4% embodied CO₂ emission in producing UHPC while still reaching the desired 28-day compressive strength of 120 MPa under the standard curing condition. This contributes to a significant environmental impact benefit for the sustainable development of UHPC.

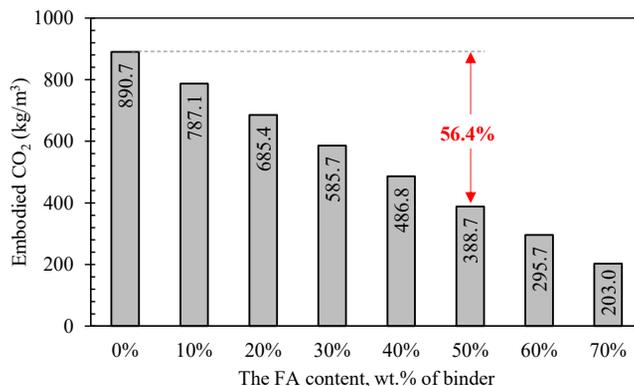


Figure 7. Embodied CO₂ emissions of UHPC using different FA contents under standard curing condition

5. Conclusions

This paper presents a proposed mix design verified by the experiments of properties evaluation for high volume fly ash ultra-high performance concrete (HVFA UHPC). From the results obtained, the following conclusions can be drawn:

- The mix design method based on the optimization of granular composition calculated by the method of F. de Larrard, selection of a reasonable W/B ratio, and application of absolute volume principle can be used effectively to fine optimum mix proportions of HVFA UHPC. The effect of curing condition, and heat treatment duration were considered in this proposed mix design method.

- At least two days under heat treatment is required for HVFA UHPC to achieve the desired compressive strength.

- The total SCMs content of 60% (10%SF + 50%FA), or only 437 kg cement per m³, can be used to produce HVFA UHPC with a desired compressive strength over 120 MPa and 150 MPa under standard curing and heat treatment, respectively.

- The embodied CO₂ emissions of UHPC reduces 56.4% with addition of 50% FA.

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