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# Synergistic influences of ground granulated blast - furnace slag and silica fume on the viscosity of extremely low W/B cement pastes

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**Abstract.** Particularly with a low water/binder ratio, the synergistic effects of supplemental cementitious materials (SCMs) have drawn a lot of interest in improving the characteristics of cement-based products. The effects of SCMs, such as silica fume (SF) and ground granulated blast furnace slag (BFS) with varying fineness, on the setting time, saturation point, and viscosity of ultra-high performance cement pastes (UHPCP), are examined experimentally in this work. 56 mixes with various SF and BFS substitutions and exceptionally low water/binder ratios (0.16, 0.20) were used in the experiments. This work demonstrates the synergistic effects of SCMs from several angles, including the UHPCP fresh characteristics. It is shown that carefully regulating the water/binder ratio, the BFS fineness, and most significantly the addition of an ideal SF content will have a beneficial synergistic effect on the fresh qualities of UHPCP. However, utilizing high BFS replacement is discouraged as it may have a negative impact on viscosity.

*Keywords:* Ultra-high performance cement pastes, viscosity, silica fume, ground granulated blast furnace slag, synergistic effect.

*Classification numbers*: 3.3.2, 3.3.3.

## 1. INTRODUCTION

Ultra-high performance concrete is a new-generation concrete with exceptional properties in terms of workability, mechanical properties (compressive strength greater than 150 MPa), and durability [1, 2]. However, one of the limitations in the development and application of this type of concrete is the use of large amounts of cement and silica fume, usually around 800 - 1000 kg/m$^3$ and 200 kg/m$^3$, respectively [3]. This also gives a negative impact on sustainable development where the production of Portland cement contributes about 80 % of the total concrete $CO_2$ emissions, and concrete is in charge of about 9.5 % of the global $CO_2$ emissions [4]. This disadvantage has induced a promising solution to use single mineral admixture, i.e. ground granulated blast furnace slag (BFS), pulverized fly ash, metakaolin, limestone powder, and rice husk ash etc., and their combinations as alternatives. It is interesting that each mineral admixture has its pros and cons in terms of filling and pozzolanic effects, and the resulting properties of the concrete, but their combination can offset each other's disadvantages, so-called "synergistic effect" [5].

Silica fume - SF, a highly reactive mineral admixture with an ultra-fine size (about 0.15 μm) and a very high amorphous $SiO_2$ content, has been considered the inevitable constituent in UHPC with a number of positive filler and pozzolanic effects, even improving the flowability of UHPC mixtures when using a reasonable replacement content [6-8]. Therefore, the addition of SF improves both the mechanical properties and durability of UHPC due to its outstanding characteristics and properties. However, a high SF addition will make the mixture too viscous, leading to detrimental effects on the homogeneity of mixtures and resulting in low mechanical properties [9].

One of the potential alternatives is the BFS, which is a by-product of the blast furnaces used to make steel and iron. BFS contains a high percentage of silica and alumina. Actually, the incorporation of BFS has been proven to positively influence the fresh and hardened properties of concrete [10]. The utilization of large volumes of blast-furnace slag, which is considered by-product waste, will not only help to improve the technical and economic aspects of the cement concrete industry but also help to reduce the negative effect of blast-furnace slag on the environment. Furthermore, the modern construction industry requires new material generations including UHPC in which a high amount of BFS is used as a mineral admixture. In fact, research on UHPC has also been implemented since 2011 in Viet Nam, and several results relating to the use of local mineral admixtures in fabricating UHPC have been published elsewhere [7, 11]. Unfortunately, there has been limited research devoted to using the high volume of locally sourced BFS in UHPC, whilst a large number of relevant studies around the world have facilitated the use of BFS in UHPC.

The addition of BFS also results in a negative effect, especially at a high replacement level. It has been observed that the separation and retarded setting occur with concrete mixtures of UHPC with BFS. However, these drawbacks can be compensated with the increase in the viscosity of concrete mixtures by decreasing the w/b ratio and/or combining with highly reactive mineral admixture, for example, SF. Besides, the addition of BFS reduces the hydration rate and thereby the early age strength, especially in UHPC with a high BFS content and cured at a low temperature. In such conditions, incorporating a highly reactive pozzolanic mineral admixture such as SF or rice husk ash could be a promising solution to overcome some technical issues as well as towards a sustainable construction material.

Though numerous studies on the application of SCMs in UHPC have been carried out as discussed above, the understanding of hydration of SCMs has not yet advanced beyond broad generalities mainly because of the variation in their physical and chemical properties, which depends on the origin of the SCMs. It is acknowledged that the current understanding of binary systems (e.g. OPC with BFS) is reasonably clear. However, the synergistic influence of a combination of several SCMs on fresh properties of UHPC pastes becomes more complicated although the aforementioned advantages of multiple SCMs are expected. This study aims to deepen the understanding of the synergistic influences of SF and BFS produced from the Vietnam steel industry on the fresh properties of UHPC pastes. A series of experiments have been carried out to evaluate standard consistency, setting time, and viscosity of UHPCP in extremely low w/b systems containing both SF and BFS. In addition, the effect of different particle sizes of BFS is also included in this study.

## 2. MATERIALS AND EXPERIMENTAL PROGRAM

### 2.1. Materials

Experiments were performed on cement paste samples, which were made of OPC, BFS, and SF with different replacement levels, and tap water. Polycarboxylate-based superplasticizer (SP, ACE388, BASF, Ludwigshafen, Germany) with 35 % solid content by weight, density of 1.03 g/cm$^3$ was used to control the workability of the mixtures.

### *2.1.1. Ordinary Portland cement*

Type I ordinary Portland cement PC40, conforming to Vietnamese standard TCVN 2682-2009, was used. The cement exhibits a Blaine specific surface of 3870 cm²/g, a mean particle size of 11.4 µm, and a 28-day compressive strength of 52.2 MPa. Chemical compositions determined by X-ray fluorescence (XRF) are shown in Table 1.

*Table 1.* Chemical compositions (wt. %) of cement, SF and BFS.

| | $SiO_2$ | $Fe_2O_3$ | $Al_2O_3$ | CaO | MgO | $Na_2O$ | $K_2O$ | $SO_3$ | $TiO_2$ | 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 |
| BFS | 34.52 | 0.66 | 12.38 | 41.54 | 7.25 | 0.43 | 0.24 | - | - | 0.96 |

### *2.1.2. Mineral admixtures*

Undensified SF contains ultra-fine powders with a mean particle of 0.15 µm and more than 92 % $SiO_2$. The strength reactivity index of SF is 112.5 %.

The SEM images of SF and BFS are shown in Figure 1.

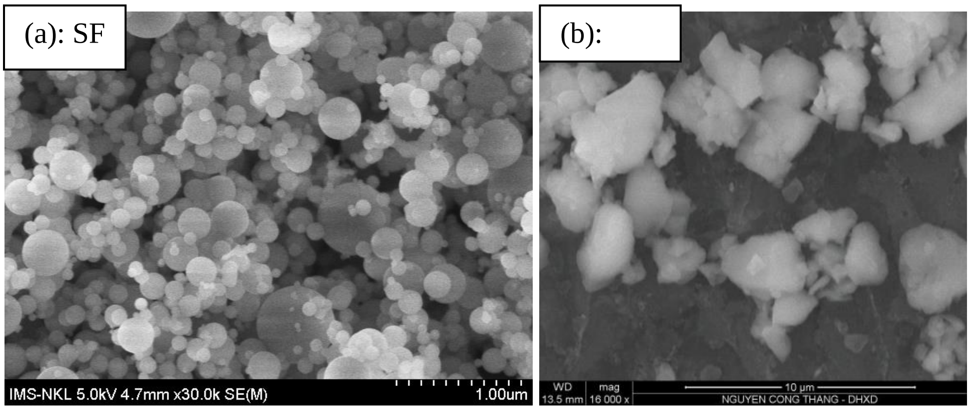

*Figure 1.* SEM image of  mineral admixture: (a) SF particles; (b) BFS 7.2 µm particles.

BFS with the mean particle sizes of 18.4, 10.4, and 7.2 µm, denoted as BFS18.4, BFS10.4, and BFS7.2, respectively, was used in this study. BFS particles with smooth and angled surfaces are observed on SEM images (Figure 1). The BFS has wide particle size distributions, as shown in Figure 2, and its main range of 4 to 20 μm. The strength reactivity index of BFS is 104.3 %. The chemical compositions of BFS determined by XRF are given in Table 1.

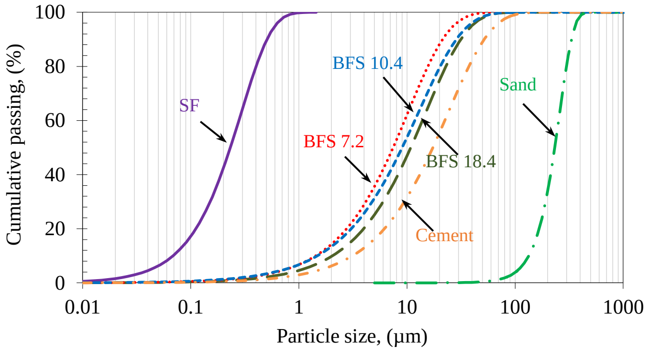

*Figure 2.* Particle size distributions of raw materials used in this study.

## 2.2. Mixture compositions

As mentioned, in this study, we primarily focused on the examination of the paste matrix of UHPC to develop and optimize the recipes for UHPC at a later stage. For those reasons, representative UHPC pastes (UHPCP) have been proposed, in which 56 mixtures were produced for various tests as shown in detail in Figure 3. UHPC typically has an extremely low w/b ratio (less than 0.25) to obtain the minimum required strength of 150 MPa. Therefore, the main experimental campaign focussed on two w/b ratios of 0.16 and 0.20. In order to investigate the effects of SCMs on the fresh properties of the mixtures, OPC cement was replaced by SF up to 30 % and/or BFS up to 70 %. Furthermore, the fineness of BFS was also studied (mean particle sizes ($d_{BFS}$) of 7.2, 10.4, and 18.4 μm).

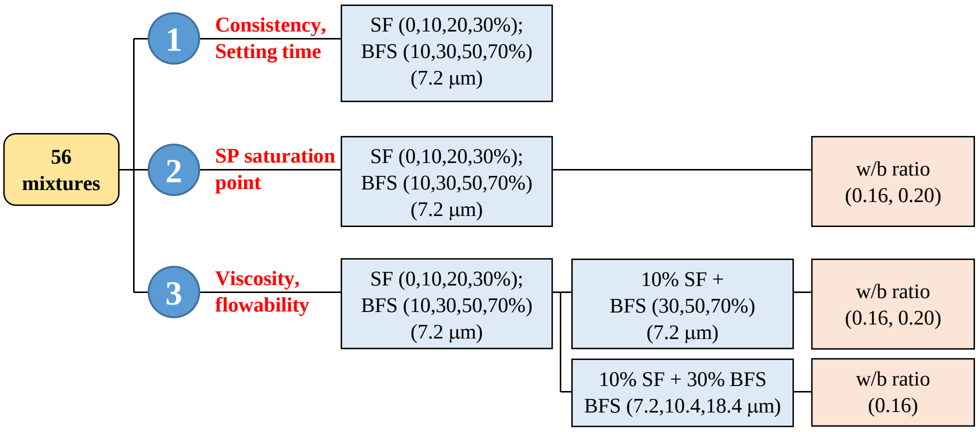

*Figure 3.* Mixture compositions and corresponding testing properties of UHPCP.

## 2.3. Setting time and consistency of UHPCP

The standard consistency of cement paste was determined according to Vietnamese standard TCVN 6017: 2015 by means of a Vicat apparatus. It is evaluated via the amount of water needed when mixed with cement and permitted the Vicat plunger with a diameter of 10 ±

1 mm to penetrate to a distance of 4 to 8 mm from the bottom of the Vicat mould.

The setting time of the cement paste was also determined according to TCVN 6017:2015 by means of the Vicat needle. The Vicat initial setting time is defined as the time elapsed between the initial materials, and the time when the 1 mm Vicat needle penetrates into the cementitious matrix a distance of 31 - 37 mm. The Vicat final setting time is defined as the time until the needle does not leave a complete circular impression on the sample surface.

## 2.4. Flowability of UHPCP and saturation point of superplasticizer

The flowability of cement pastes was determined following ASTM standard C939, which allows evaluating the flowability of the mixtures via the time required for a certain paste volume flowing through the V-shaped flow cone (named as Marsh cone) with a discharge tube diameter of 12.7 mm at a standard temperature of 23 ± 1.7 $^{o}$C. Right after mixing, a paste volume of 1725 ml was introduced into the cone. The time of efflux of the paste was determined from the moment that the paste starts continuously flowing until the entire paste volume discharged.

The flow cone test was also used to determine the saturation point of the superplasticizer (SP). The SP was incremented at a dosage of 0.2 % (by mass of binder), i.e. from 0.6 to 1.4 %. As illustrated in Figure 4, the time of efflux decreased with the increase of SP dosage, and a typical SP saturation point was defined at the dosage at the time of efflux started stabilizing. It should be noted that the steric hindrance effects of PCE-base SP are the dominant stabilizing mechanism resulting in viscosity modification in UHPCP, whereas the repulsive potential resulted from electrostatic interactions is negligible.

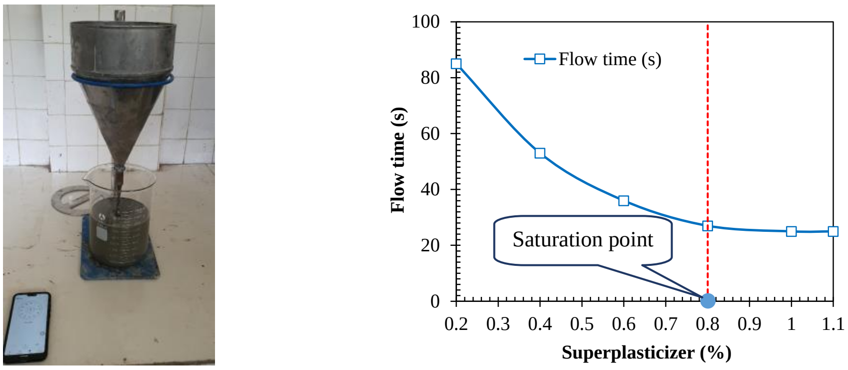

*Figure 4.* A photo of Marsh cone to measure the flowability of UPPCP (left) and an example of the determination of SP saturation point using Marsh cone for w/b ratio of 0.20 and 30% BFS (right).

## 2.5. Viscosity measurement

The viscosity of the pastes was determined using a commercial viscometer Vibro (A&D company, Viet Nam), as shown in Figure 5. The viscosity is calculated based on the current needed to vibrate two sensors immersed in the pastes with a constant frequency of 30 Hz and amplitude of less than 1 mm. Because the friction between the vibration sensors and the paste matrix is directly proportional to the viscosity, the current needed to maintain the constant frequency of vibration sensors can be used to calculate the viscosity of the pastes.

The viscosity measurement was performed right after mixing, in which a paste volume of 35 - 45 ml was poured into a cup located under the viscometer. The viscosity was determined at the time of 15 seconds after starting the sensor vibration.

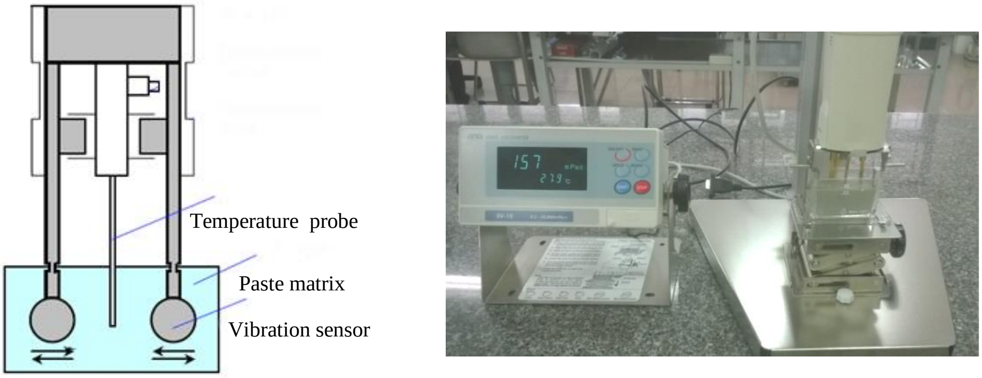

*Figure 5.* Viscometer based on the vibration of 2 sensors.

## 3. RESULTS AND DISCUSSION

### 3.1. SP saturation points

The effects of the w/b ratio and mineral admixture substitution level (i.e. BFS and SF) are shown in Figure 6.

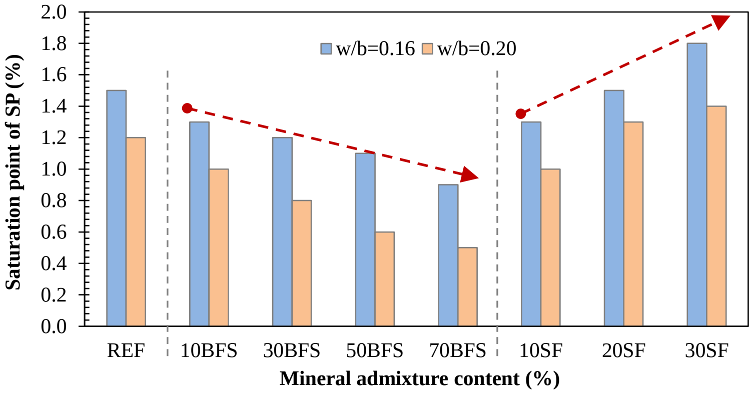

*Figure 6.* Effect of mineral admixture content and w/b ratio on the SP saturation point of pastes.

As expected, the higher the w/b ratio, the lower the SP saturation point. In the case of adding BFS, the SP saturation point became lower compared to the reference sample. Regardless of the w/b ratios, the larger the BFS replacement, the lower the SP saturation point. The SP saturation point was still lower for the sample with 10 % SF replacement compared to the reference sample. However, if the substitution level of SF increased to 20 % and 30 %, the SP saturation was also increased and to values higher than the one of the reference sample. The high SP saturation point in the SF mixtures is attributed to the ultra-fine particle size of SF (0.15 µm) with an extremely large specific surface area of around 18 - 20 $m^2$/g. Therefore, a significant amount of SP could be absorbed on the surface of SF particles, which increases the saturation point of SP. On the other hand, the particle size distribution of BFS is just a bit finer than the one of cement (Figure 2). Consequently, the SP absorption effect could be negligible. Whereas, the required water for BFS is much lower than for cement. Note that the hydration rate of cement is much faster than BFS, especially at an early age [12] leading to the lower required dosage of SP to obtain the same flow time determined by the Mash cone method, thereby lowering the SP saturation point for BFS mixtures.

### 3.2. Effects of BFS and SF on setting time and standard consistency of UHPCP

Figure 7 shows the setting time and standard consistency of UHPCP with various BFS and SF replacement levels. It is clearly seen that the standard consistency decreased with the increase of BFS content and vice versa with SF. In the case of SF, a high amount of silica fume tends to increase the consistency due to the relatively higher surface area of SF, i.e. 18 $m^2/g$ calculated from the mean SF particle size of 0.15 μm in this study. A delay can also be observed in the initial setting time of the SF paste by about 30 minutes, similar to the observation of Didamony *et al*. [13]. However, the final setting time was not significantly delayed with the increase of the SF addition. Note that the effect of SF on the setting times is not consistently reported in the literatures. The SF is very reactive at very early hydration (few hours) as SF accelerates the $C_3A$ and $C_3S$ hydration at the first few hours as reported by Cheng Yi and Feldman [14]. On the other hand, Swamy found that the addition of a small amount of SF (about 10 % by weight of cement) had no significant or slight alteration of the initial setting time [15], whereas SF significantly influenced the final setting time, which is contrary to the observation of Rao [16].

The positive effect of the BFS addition is because the required water needed for BFS hydration at the first few hours is lower compared to the one for the OPC system as explained before. Both initial and final setting times of UHPCP increased with the increase of BFS replacement levels. For the mixture with 70 % BFS, the initial setting time was doubled compared to the reference mixture. The increase of final setting time with the increase of BFS content was slower compared to the increase of initial setting time. However, if the BFS content was more than 50 %, the final setting time was significantly increased, which brought the final setting time to 245 and 315 minutes for 50 % and 70 % BFS replacement, respectively. Whereas the final setting time of the reference mixture was only 200 minutes. BFS is known as a slow reactive hydraulic binder resulting in a longer setting time compared to the pure OPC system.

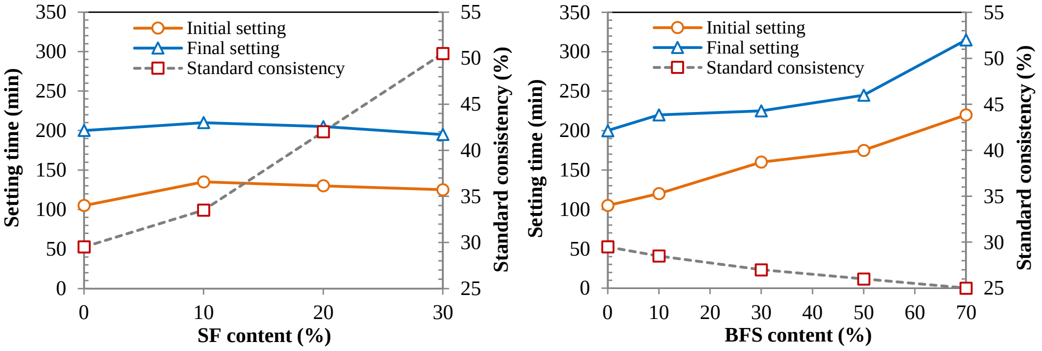

*Figure 7*. Effect of SF and BFS ($d_{BFS}$ = 7.2 μm) on standard consistency and setting time of UHPCP.

### 3.3. Effects of BFS and SF on the viscosity of UHPCP

#### *3.3.1. Main effects*

The effect of BFS and SF content on the viscosity and flow time of UHPCP with different w/b ratios is shown in Figure 8. Regardless of w/b ratios, the minimum viscosity was obtained with UHPCP containing 10 % SF. However, adding more SF would increase the viscosity.

Especially for the UHPCP with the lowest w/b ratio of 0.16, the viscosity was significantly increased to the value which is higher than the one of reference mix when adding 30 % SF. A similar trend was observed for the flow time determined by the Marsh cone. However, the flow time was more sensitive with the SF content, meaning that even adding 20 % SF, the flow time was already higher compared to the reference mixes for all w/b ratios. Reasonable use of SF would not only help to reduce the required water and SP for the mix, but also enhance the viscosity and flow time of the UHPC paste. The density of SF (2.2 g/cm$^3$) is much smaller compared to the density of cement (3.15 g/cm$^3$), thus when replacing the cement with SF, the paste volume is increased, resulting in an increase in the workability. It should also be noted that the finely spherical particles of SF are covered by a layer of the surface-active compound during mixing with a superplasticizer. Therefore, there is an electrostatic repulsive force between the cement and SF particles. The SF with an ultra-fine spherical shape, which is about 75 times finer than the one of cement, could reduce the friction between different particles in the paste matrix, also known as the ball-bearing effect [9]. In addition, according to Bache [17], for concrete with a low w/b ratio and using SP, the SF particles would occupy the space in which the water is typically located between the cement particles. As a result, more free water is provided for the paste matrix to increase workability. Nevertheless, excessive SF could absorb a significant amount of water due to its extremely high specific surface area, around 18 - 20 m$^2$/g. Therefore, the water needed for hydration and creating workability is reduced, resulting in a decrease in viscosity (or flow time) in such cases.

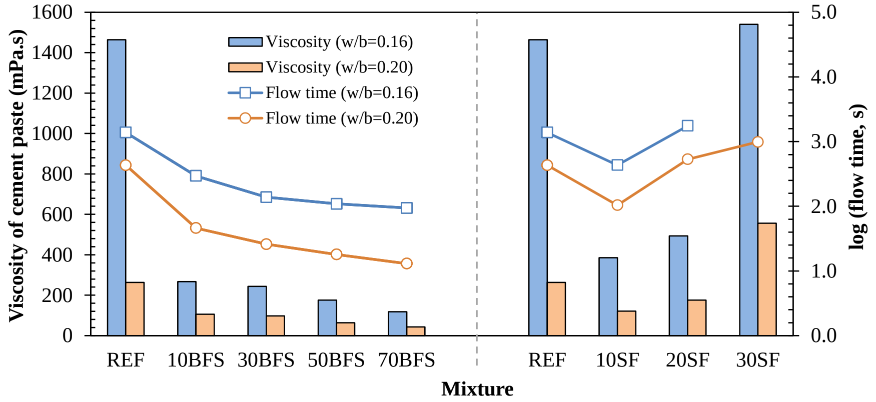

*Figure 8.* Effect of BFS ($d_{BFS}$ = 7.2 μm) and SF on viscosity and flow time of UHPCP.

The addition of BFS resulted in an opposite effect on viscosity and flow time compared to the addition of SF, as shown in Figure 8. The addition of BFS significantly reduced the viscosity and flow time of the UHPCP regardless of w/b ratios. The more BFS addition, the larger reduction in viscosity and flow time. However, adding more than 50 % BFS would induce bleeding in the mix with a w/b ratio of 0.2. The decrease in viscosity and flow time is attributed to the smooth dense slip surface of BFS particles, which would make the required water of BFS to be absorbed on its surface less compared to cement or SF. Therefore, more water could be used for enhancing workability. Furthermore, the water content needed for early age hydration of BFS is smaller compared to the one for cement because the hydration of BFS is much slower compared to the one of cement [12, 18], which makes more available water to increase the flowability of concrete. Besides, slags induce a negative surface charge due to the highly alkaline conditions (silanolate group formation). Under a high pH solution, calcium and subsequently sulfate ions present in the pore solution of cement are adsorbed on the BFS surface resulting in a double ion layer formation, i.e. $Ca^{2+}$ positively charged layer and $SO_4^{2-}$ negatively charged layer. Therefore, the final surface charge of slag dispersed in the pore solution of cement is always negative. The addition of superplasticizer induces a competitive adsorption between the sulphate anions and superplasticizer. The use of highly anionic superplasticizer will desorb $SO_4^{2-}$ and adsorb $Ca^{2+}$ ions resulting in a strong dispersing effect. In addition, more plasticizers could be absorbed on the BFS surface because of its large surface energy [9], resulting in a more dispersing effect between the particles and improving the filling effect of BFS leading to an increase in the paste's fluidity.

### *3.3.2. Synergistic effect*

As discussed in the previous section, without SF, the bleeding would occur if adding more than 50 % of BFS. The use of SF blended with BFS could help to prevent bleeding. Therefore, the synergistic effect of SF and BFS on the viscosity and flow time was investigated by varying the BFS content from 10 to 70 %, while keeping SF at a constant content of 10 %. Figure 9 proves the efficiency of using both SF and BFS to slow down the decrease of viscosity when increasing the BFS content in the mixtures. Compared to samples with 10 % SF, the average viscosity (of two w/b ratios) only decreased by 13 % when using 10 % BFS. The extent of viscosity decrease was larger if a higher amount of BFS was added to the mixtures, i.e. a viscosity decrease of 45 % and 75 % for 50 % and 70 % BFS, respectively. However, no bleeding was observed, which was the case for mixes containing more than 50 % BFS but without SF as shown above. Similarly, the flow time was decreased with the increase of BFS content, while keeping a constant SF content of 10 %. Nevertheless, the rate of flow time decrease was not significant, especially at high BFS replacement levels.

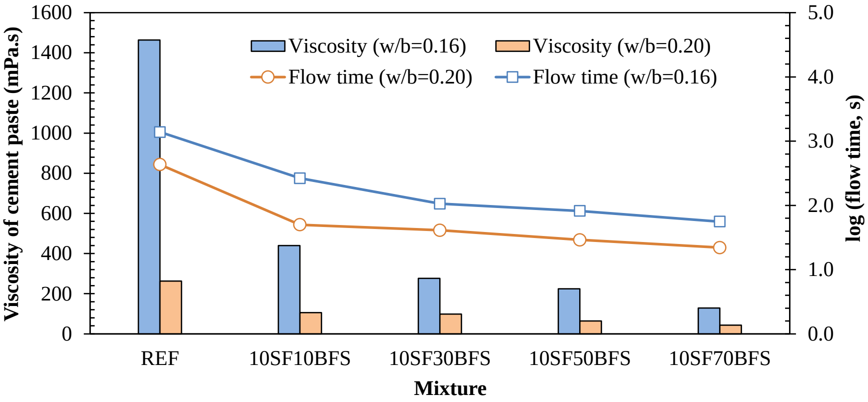

*Figure 9.* Synergistic effect of the combination of 10 % SF and BFS ($d_{BFS}$ = 7.2 μm) on the viscosity and flow time of UHPCP.

Four main forces determining the rheological nature of the binder paste containing particles with a size from several nm to 100 μm have been identified by Roussel *et al*. [19]. These include the Brownian force, surface force (i.e. colloidal interactions), hydrodynamic force, and various particle contact forces. Cement particles smaller than a few μm are significantly controlled by the random (Brownian) motion, which causes them to diffuse through the suspending liquid. Usually, most mineral admixture particles are far smaller than the particle size of cement. The addition of SF with a particle size far from both cement and BFS highly increases the Brownian motion. Therefore, it helps to increase diffusion, form a more stable suspension, and reduce the risk of bleeding.

### *3.3.3. Effect of BFS fineness*

Not only the BFS content but also the BFS fineness relatively affected the viscosity and flow time, as shown in Figure 10. Both viscosity and flow time were increased when the particle sizes of BFS increased for the mixtures containing 30 % BFS and a w/b ratio of 0.16. For mixtures without SF, increasing the particle size from 7.2 µm to 10.4 µm slightly increased the viscosity and flow time. However, if the size was increased to 18.4 µm, the extent of the increase in the viscosity and flow time was much larger. The BFS particles possess a smooth surface, thus the water absorption is not changed significantly with the increase of the BFS particle size from 7.2 to 10.4 and 18.4 µm. Therefore, it is expected only a slight increase in viscosity with the increase of BFS particle size due to the effect of surface water absorption. However, it should be noted that the mean particle size of cement is 11.4 µm in this study, which means that only a smaller size, i.e. BFS7.2 with a mean particle size of 7.2 µm, might have a filler effect and release the trapped water resulting in a reduced viscosity. The filler effect would not exist in the BFS paste with the mean BFS particle size of 18.4 µm (larger than that of cement, i.e. 11.4 µm in this study), but also larger BFS particle size could induce an increase in viscosity as observed in this study.

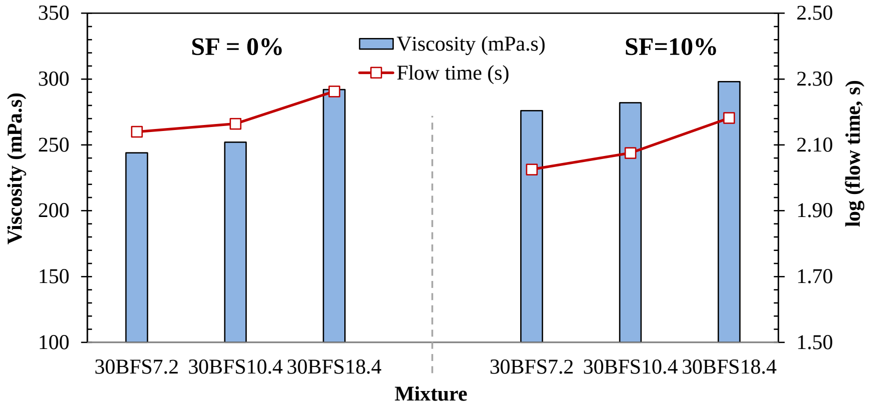

*Figure 10.* Effect of BFS fineness on the viscosity and flow time of UHPCP with a w/b ratio of 0.16 and a fixed 30 % BFS.

In the case of mixtures containing both 30% BFS and 10% SF, the effect of BFS fineness was not significant. The addition of SF with a mean particle size of 0.15 µm (corresponding to a surface area of 18 $m^2/g$) increased the viscosity of the paste. Typically, the effect of SF addition on viscosity is twofold. On one hand, if a small amount is added (i.e. < 5 %), the SF particles with spherical shape reduce the viscosity of the paste due to the “ball-bearing” and “filler” effects. These effects result in a reduction in the water demand because SF fills the voids between cement grains and releases the trapped water. On the other hand, if a high amount of SF is added, the excessive silica fume increases the total surface area of the system, thereby increasing the water demand and viscosity. In this study, adding 10 % SF exceeds the optimal level resulting in an increase in viscosity, which is even stronger than the viscosity increase due to the increase in BFS particle size.

In summary, the combination of both BFS and SF gives a positive effect on the viscosity and flow time. Reasonable viscosity and flow time were obtained with mixtures containing 10 % SF and a maximum of 50 % BFS. This range was then chosen as a basic mix design to further study the hydration of UHPCP.

## 4. CONCLUSIONS

The synergistic effects of SCMs on the standard consistency, setting time, and viscosity of UHPCP with incredibly low w/b ratios are thoroughly examined in this work. 56 mixes with various binder combinations were used in a thorough experimental program to look into any potential synergistic effects of SCMs. Following are some of the major findings of this study about the synergistic effects:

- The viscosity and flow time of UHPCP were enhanced by the addition of SF with ultra-fine spherical particles and BFS with smooth dense slip surface. With a w/b ratio of 0.2, however, adding more than 50 % BFS would cause bleeding in the mixture. In the case of SF and BFS addition, the maximum contents of SCMs are recommended to be 20 % and 5 0 % of binder weight, respectively, to achieve a satisfactory viscosity and flow time for UHPCP.
- Even at relatively low w/b ratios between 0.16 and 0.20, the addition of 10 % SF had a positive impact on the viscosity and flow time of UHPCP. Additionally, the viscosity and flow time of UHPCP were enhanced by the synergistic combination of 10 % SF with various BFS contents. In particular, the inclusion of SF reduced the danger of bleeding when UHPCP was made with a large volume of BFS, or more than 50 %.
- For mixes including BFS but without SF, the increase in BFS fineness resulted in an increase in viscosity and flow time. The inclusion of 10 % SF, however, resulted in a decrease in the water requirement, making the effect of BFS fineness insignificant. Because the usage of BFS with high particle size decreases the OPC hydration degree as well as the pozzolanic reaction, the bound water was also noticeably reduced with an increase in BFS size.

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***CRediT authorship contribution statement.*** Nguyen Cong Thang: Conception, Methodology, Investigation, and Interpretation of data, Drafting the article. Nguyen Van Tuan: Methodology, Investigation, Drafting the article, Funding acquisition. Quoc Tri Phung: Conception, Critical reviewing, and Final approval of the version to be submitted.

***Declaration of competing interest.*** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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