Physical and Chemo/Mechanical behaviors of fly ash and silica fume belite cement pastes- Part I

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Abstract: Physical, chemical and mechanical properties of high belite cement (HBC) blended with high pulverized fly ash (HPFA) with stable ratio of silica fume (SF) in comparison with Portland cement (OPC) were investigated. Results showed that the water of consistency and setting times (Initial and final) tended to increase with the increase of HPFA content. The bulk density and compressive strength were also improved and enhanced with the increase of HPFS content at all hydration times, but only up to 15 % HPFA, and then decreased with further increase. However, the total porosity slightly decreased, but started to increase with further increase of >15 % HPFA. The free lime content of the pure OPC and HBC gradually were increased as the hydration times progressed up to 90 days, while those of blended cements increased only up to 7 days and then decreased onward. The results were confirmed by measuring the heat of hydration and ultrasonic pulse velocity for the optimum cement pastes comparing with those of both OPC and HBC. The heat of hydration of the optimum cement pastes was decreased at all hydration times and become lower than those of OPC and HBC. The ultrasonic pulse velocity test (USPV) proved that the uniformity and quality of the matrix of the hardened cement pastes are good with no cracks.

Keywords: OPC, HBC, HPFA, SF, Setting, Density, Porosity, Strength, Free lime, Heat of hydration.

1. Introduction
1.1 Scope of the problem

Portland cements (PCs) are being the main product for the fabrication of mortars and concretes in the building industry [1,2]. In spite of its ubiquitous usage, PC is a quite environmentally contentious material, where a ton of Portland cement clinker released about ~0.87 tons of CO$_2$ into the atmosphere [3], that produces from the decomposition and burning of limestone and fuel. This reduces into ~7% of the total anthropogenic CO$_2$ emissions with BCs [4]. belite cements (BCs) mainly containing a high content of belite (β-C$_2$S), but a lower contents of alite (C$_3$S) and calcium aluminates (C$_3$A or C$_6$AF), are currently used as low heat cements. These binders produce high amounts of CSH- gel and have very good durability properties [5-7], and some other advantages as a lower limestone level accompanied with lower associated CO$_2$ emissions, a lower energy generation, a lower kiln operating temperature, i.e. a lower CO$_2$ and NO$_x$ emissions due to the burning of fuel and a lower temperature increase at early hydration age. However, early-age strength developments are not competitive with those of Portland cements.

The reduction of CO$_2$ footprint of climate crisis [1,5,8,9]. The production of infrastructures with a longer service life [10,11] would also contribute to the overall decrease of CO$_2$ footprint, as less cement will be needed. This will reduce the production of concrete demolition waste in the future. For these endeavors, it will be needed more durable cements with a lower embodied carbon content, novel techniques to quantify short- and long-term hydration chemistry and micro- and meso-structure developments [12], and also original data correlation tools [13] for better predictions.

High belite cement (HBC) is widely employed as the main material in the construction of dams, canals and other water conservancy engineering structures to control the cracks that may be occurred due to high temperatures [13,14]. Previous research studies have thoroughly investigated the thermal and mechanical properties, crack resistance and deformation performance of HBC cement or concrete, which all proved that HBC is a cementitious material.
with ideal characteristics of a low hydration heat, a high long-term strength, energy saving and environmental protection for hydraulic concretes [15–18]. Due to the influence on local environment and huge investment, the safety and durability for water conservancy engineering must be ensured over hundred years or even longer. It is one of the most significant long-term performances to be considered for the hydraulic cement structures.

The high belite cement (HBC) has a higher content of β-C₃S and lower contents of alite (C₃S) and celite (C₃A). Accordingly, it is commonly expected that the leaching of HBC could be less intensive compared to OPC. In fact, the different mineral compositions lead to the distinctions of Calcium-Silicon ratio (Ca/Si), CH content, hydration product morphology and gypsum content notably. These are the key factors affecting the calcium leaching evolution of cement-based materials [19,20].

In the following stages, the use of supplementary cementitious materials is undoubtedly the strategy with the lowest economic and performance impacts [1,8,21–23]. The typical supplementary cementitious materials used by the cement industry are industrial wastes as blast furnace slags, fly ashes and silica fume. The long term availability of the industrial wastes will accordingly decrease [21] as in case of fly ash in the United States and Europe. For instance, calcium sulfoaluminate cements or high belite-cements of alkaline activation [7,24–26] are facing tremendous challenges worldwide. This is essentially contributed to practice codes, availability of raw materials and costs.

Only one way to reduce CO₂ footprints, cements with the same components of PC, but with lower limestone demand which is the belite cements (BCs). These binders are mainly ettringite-based and focused on Ordinary Portland and active, CSH gel-based belite binders.

1.2. Objectives of the study

The essential target of the current investigation is to evaluate and follow the effect of fly ash (HPFA) in combination with a certain ratio of silica fume (SF) on the physical, chemical and mechanical properties of belite cement in comparison with OPC cement. The obtained results were confirmed measuring the heat of hydration and ultrasonic pulse velocity tests.

2 Experimental Section

2.1 Raw materials

The raw materials which were used in the present research study are Ordinary Portland cement (OPC Type I- CEM I 42.5R), High belite cement (HBC), high pulverized fly ash (HPFA) and silica fume (SF). The blaine surface areas of the four raw materials were 3400, 3763, 4150 and 19230 cm²/g, respectively. The blaine surface area was applied by the “Air Permeability Apparatus”. The OPC and HBC samples were provided by the new cement factory, Sohag, Egypt, while HPFA sample was provided from a local plant, Giza, Egypt. The SF sample was supplied by the ferrosilicon alloys company, Edfo, Egypt. The chemical composition of the OPC, HBC, HPFA and SF samples that measured by X-ray florescence technique (XRF) is shown in Table 1. The mineralogical phase composition of the used OPC and HBC as received from the factory is given in Table 2, while the mix composition is illustrated in Table 3.

| Oxide Materials | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO  | MgO  | Na₂O | K₂O  | SO₃  | Insoluble residue | LOI  | Total, % |
|-----------------|------|-------|-------|------|------|------|------|------|-------------------|------|---------|
| OPC             | 20.41| 5.51  | 3.64  | 61.53| 1.60 | 1.01 | 1.16 | 2.07 | 1.63              | 1.44 | 100     |
| HBC             | 23.62| 3.45  | 4.28  | 57.54| 4.89 | 0.07 | 0.13 | 2.43 | 0.77              | 1.82 | 100     |
| FA              | 50.53| 31.64 | 6.05  | 5.13 | 1.47 | 0.76 | 3.14 | 0.17 | 0.42              | 1.51 | 100     |
| SF              | 93.15| 0.11  | 0.15  | 1.12 | 0.32 | 0.08 | 0.41 | 0.41 | ----              | 0.71 | 100     |
Table 2. Mineralogical constituents of OPC and HBS cement samples, wt. %.

| Phase | Cement | C_3S | β-C_2S | C_3A | C_4AF |
|-------|--------|------|--------|------|-------|
| OPC   | 48.37  | 24.72| 8.28   | 11.45|       |
| HBC   | 29.12  | 42.87| 2.03   | 12.57|       |

Table 3. Mix composition from the used raw materials, wt. %.

| Materials Mixes | OPC | HBC | HPFA | SF |
|-----------------|-----|-----|------|----|
| P0              | 100 | ----| ---- | ----|
| B0              | 100 | ----| ---- | ----|
| B1              | ----| 95  | 5    | ----|
| B2              | ----| 90  | 5    | 5   |
| B3              | ----| 85  | 10   | 5   |
| B4              | ----| 80  | 15   | 5   |
| B5              | ----| 75  | 20   | 5   |

2.2 Preparation and methods

There are five blended cement batches composed from Belite cement (BC), high pulverized fly ash (HPFA) and silica fume (SF). The belite cement batches were composed of BC, HPFA and SF as 100:0:0, 95:5:0, 90:5:5, 85:10:5, 80:15:5, and 75:20:5 having the symbols: B0, B1, B2, B3, B4 and B5, respectively (Table 3). The blending process of the various cement batches was firstly done in a porcelain ball mill using three balls for two hours to assure the complete homogeneity of all cement batches. The standard water of consistency and setting times [5,6,27,28] of the various cement pastes were directly measured using Vicat Apparatus. The water of consistency could be calculated from the following relation:

\[ WC, \% = \frac{A}{C} \times 100 \]  

Where, A is the amount of water taken to produce a suitable paste, C is the amount of cement mix (300 g). During mixing, the correct predetermined w/c-ratio (water of consistency) was poured into the cement portion inside the mixer gradually. Then, the mixer was run for 5 minutes at an average speed of 10 rpm in order to have perfect homogenous pastes. Before casting, the moulds were deld with a thin film of a motor engine oil to facilitate the release of the cement cubes from the moulds when the moulds were de-moulded. The cement pastes were then moulded into one inch cubic stainless steel moulds (2.5 x 2.5 x 2.5 cm³) using about 500 g from the cement powder, vibrated manually for three minutes, and also on a mechanical vibrator for another three minutes to remove all air bubbles tapped inside the cement pastes. The moulds were filled to the top surface and smoothed with a flat stainless steel trowel or a suitable spatula to obtain a flat and smooth surface [29-32]. After casting of samples, they were covered with a wet sheet during the first 24 hours to prevent moisture loss. Hence, the moulds were kept in a humidity chamber for 24 hours under 95 ± 1 relative humidity (RH), and room temperature (23 ± 1), demoulded in the next day and soon cured and totally immersed in water at an ambient laboratory temperature till the time of testing for heat of hydration, bulk density, apparent porosity, compressive strength, combined water and free lime contents at 1, 3, 7, 28 and 90 days. This is necessary for the cement cubes as it facilitates the proper hydration of cement phases.

The bulk density (BD) and total porosity (δ) of the hardened cement pastes [29-32] were calculated from the following equations:

\[ BD, (g/cm^3) = \frac{W1}{(W1-W2)} \times 1 \]  

\[ \delta, \% = \frac{(0.99 \times We \times BD)}{(1+Wt)} \]  

Where, 0.99 is the specific volume of free water, We is the free or evaporable water content, BD
is the bulk density, g/cm³ and Wt is the total water content which is equal to the sum of We and Wn contents, where Wn is the combined water. The compressive strength (CS) of the various hardened cement pastes [33-36] was measured and calculated from the following relation:

\[
CS = \frac{L}{S} \frac{(KN)}{(cm^2)} \frac{(KN/m^2 \times 102)}{(Kg/cm^2)/10.2 (MPa)}
\]

Where, L is the load taken, Sa is the surface area. Thereafter, about 10 grams of the broken specimens were first well ground, dried at 105°C for 30 min. and then were placed in a solution mixture of 1:1 methanol: acetone to stop the hydration [37-40]. The free lime content (FLn) of the hydrated samples pre-dried at 105°C for 24 hours was also determined. About 0.5 g sample + 40 ml ethylene glycol → heating to about 20 minutes without boiling. About 1–2 drops of pH indicator were added to the filtrate and then titrated against freshly prepared 0.1N HCl until the pink color disappeared. The 0.1 N HCl was prepared using the following equation:

\[
V_1 = N \times V_2 \times W \times 100/D \times P \times 1000
\]

Where, V1 is the volume of HCl concentration, V2 is the volume required, N is the normality required, W is the equivalent weight, D is the density of HCl concentration and P is the purity (%). The heating and titration were repeated several times until the pink color did not appear on heating. The free lime content [41-43] was calculated from the following relation:

\[
FLn \% = \frac{V \times 0.0033}{1} \times 100
\]

Where, FLn and V are the free lime content and the volume of 0.1 N HCl taken on titration, respectively.

The heat of hydration of the prepared blended cement pastes has been experimentally investigated to confirm the obtained results [44-46]. Ultra-sonic pulse velocity (UPV) method (Figure 1) is one of the prominent non-destructive testing (NDT) methods. It is mainly carried out to assess the uniformity and relative quality of existing structures and specimens. The test was carried out on 1, 3, 7, 28 and 90 days.

The mineral composition of PFa specimen was investigated by X-ray diffraction patterns (XRD) and diffraction thermal analysis (DTA). The XRD analysis was achieved by a Phillips X-ray diffractometer (XRD), PW 1710 powder with an anticathode copper radiation and Cu-Kα radiation, wavelength of 1.54178 Å and a graphite monochromator. The tube working voltage was 40 kV and current strength was 30 mA, in the range 5–50° 2θ with a step of 0.02 and 0.5 seconds retention time for each step. The DTA analysis was carried out using NETZSCH Geratore Selb, Bestell-Nr. 348472c at a heating rate 10 °C/min up to 1000 °C. The SEM images of the fractured surfaces, coated with a thin layer of gold, were obtained by JEOL-JXA-840 electron analyzer at accelerating voltage of 30 KV.

**Figure 1. Ultrasonic Pulse Velocity Tester.**
3. Results and Discussions

3.1. XRD, DTA analyses of HPFA

The XRD diffraction patterns of the used fly ash sample are shown in Figure 2. Crystalline and amorphous phases are detected and also the differences in the amounts of amorphous phases. The crystalline phases were identified according to JCPDS standards. The content of any individual mineral phases cannot be easily identified. The FA sample contains a significant amount of amorphous matter but low amounts of crystalline phases, quartz (Q) and feldspar (F). In most cases, hematite (H), anhydrite (A) and mullite (M) are detected. The amorphous phase minerals are more reactive if it is compared to the crystalline phases. This confirms the exploitation of FA as an alternative substitute for normal raw mixture used to produce Portland cement clinker.

The DTA thermograms of FA sample is shown in Figure 3. The exothermal peak at about 500-550 °C proves that carbon does not burned completely. The existence of unburned carbon in FA sample make it to be used successfully as a suitable raw material in the raw mixture but with smaller amounts of fuel. Hence, it is in turn lead to the reduction of the energy needed for the sintering process [47]. The DTA curve of the studied FA sample up to 1000 °C did not show any other exo- and/or endothermic peaks that could eventually correspond to the formation of new mineral phases during heating and/or sintering.

3.2. SEM of BC and HPFA

The SEM images of the used belite cement and HPFA is shown in Figure 4. The cement minerals are made of small particles (<100 nm). The grain size of
The clinker was less than 2 μm. The smaller particle size, looser structure, higher porosity, and crystal structure of C12A7 revealed that this clinker had higher hydration activity when compared with that calcined at a high temperature. In addition, unreacted mineral particles originating from the FA were observed. The porous particles increased the water requirement and hydration rate of the cement. The smaller grain sizes improved the grindability of all clinkers, which reduced the energy consumption in the cement grinding process, while there are several particulates in HPFA sample with various shapes and sizes as flocculants or almost globulars.

### 3.3. Water of consistency

The water of consistency (water/cement ratio) of belite cement pastes incorporating HPFY and SF comparing with those of Portland cement (OPC) were graphically represented in Figure 5. The water of consistency of OPC pastes was 29 %, while that of belite cement was 29.25 %, i.e. the water of consistency of belite cement was slightly higher than that of Portland cement [38-40]. This ratio was enhanced with the addition of both HPFA and SF. This is mainly attributed to that the addition of either PHFS or SF increased the blaine fineness of the cement batches. So, the water of consistency increased, in addition that the PHFA and/or SF like water [48-50].

On the other side, Figure 5 also shows the setting times (initial and final) of belite cement batches containing different ratios of HPFY and a constant ratio of SF (5 wt. %) comparing with those of Portland cement. The setting times behaved as in case of water consistency, i.e. the setting times increased with gradual addition of HPFA and SF [5,6,15-18,23,31,36,38,50]. This is essentially due to the higher surface area caused by replacement of cement powder by HPFA and SF.

### 3.4. Bulk density and total porosity

The bulk density and total porosity of belite cement batches incorporating HPFY and a constant ratio of SF comparing with those of Portland cement pastes (OPC) were graphically represented in Figures 6 and 7, respectively. The bulk density of either Portland or Belite cements increased little by little with the addition of HPFA and SF, while the total porosity decreased. This was taken place at all curing times up till 90 days. This firstly is contributed to the normal hydration process of the major cement phases. Moreover, the addition of HPFA or SF further increased the bulk density and decreased the total porosity. This is due to the pozzolanic reactions that could be occurred between the constituents of these waste materials (HPFA and SF) with those of Portland or Belite cements [50-52].

During the hydration process or even the pozzolanic reactions, a large amount of hydration products could be formed, and soon deposited into the pore structure of the hardened cement pastes. This was leasing to the decrease of total porosity, which reflected positively on the bulk density, i.e. the bulk density was improved and enhanced [5,6,23,36,40,44]. This was continued up to B4 that was containing 15 % HPFA and 5% SF, but then decreased with further addition of HPFA (B5), due to the deficiency of the main cementing and hydrating material [5,6,44,52,53]. Moreover, the bulk densities of the belite cement batches containing HPFA up to 15 % were higher than those of the blank cement batches. Hence, the incorporation of HPFA into the belite cement activated the best hydration process [36-38,54]. Thereby, it was selected to be the optimum cement batch.

![Figure 4. The SEM image of the used PFa sample](image-url)
Figure 5. Water of consistency and setting times of the various blended cement pastes

Figure 6. Bulk density of the various blended cement pastes hydrated up to 90 days.
Figure 7. Total porosity of the various blended cement pastes hydrated up to 90 days.

Figure 8. Compressive strength of the various blended cement pastes hydrated up to 90 days.
3.5. Compressive strength

The compressive strength of belite cement batches incorporating HPFY and a stable ratio of SF comparing with those of Portland cement (OPC) were graphically plotted in Figure 8. It is obvious that the compressive strength of all cement pastes improved and enhanced with the hydration times up to 90 days. This is mainly contributed to the normal hydration process of all cement phases, particularly the silicate phases of the cement (C₃S and β-C₂S) [5,6,39,55-57]. The pozzolanic reactivity of HPFA and SF further improved and in turn increased the compressive strength much more than in case of its absence. So, the compressive strength of cement batches B1, B2, B3 and B4 were slightly higher than those of the blank (B0) at all hydration times. With cement batch B5 containing 20 % HPFA, the compressive strength was adversely affected, i.e. it decreased and were becoming lower than those of the blank [55-58].

The increase of compressive strength is essentially attributed to the pozzolanic reactivity of HPFA and SF with the resulted free lime, Ca(OH)₂ from the hydration of silicate phases of the cement forming hydration products that often deposited in the pore system of the hardened cement pastes. This would decrease the total porosity and increase the bulk density. This in turn would be reflected positively on the compressive strength, in addition to the filling action of the very fine HPFA and SF particles [59-61]. Furthermore, the compressive strength of the belite cement batch (B4) are higher than those the OPC pastes itself. Therefore, it was choice to be the optimum cement batch. The results of compressive strength are in good agreement with those of bulk density and total porosity.

3.6. Free lime contents

Generally, the hydration reaction of C₃S and β-C₂S in Portland cement (OPC) or belite cement (BC) can be expressed and simplified in the following relation:

- \[2C₃S + 6H₂O \rightarrow C₃S₂H₃ + 3CH\] (8)
- \[2C₂S + 4H₂O \rightarrow C₃S₂H₃ + CH\] (9)

Where, the amount of free lime, CH generated by β-C₂S hydration is only one third of that produced by C₃S [2,5,6]. The free lime contents of the hydrated OPC and BC cements (P0 and B0) and the optimum belite cement pastes containing 15 % HPFA and 5 % SF (B4) is drawn versus the hydration ages up to 90 days in Figure 9. The free lime contents of OPC and BS cement pastes increased continuously as the hydration times progressed up to 90 days. This is evidently due to the normal hydration process of calcium silicate.
phases of the cement [2,5,6,12,15,49,62]. The free lime contents of OPC were higher at the early times of hydration up to 7 days than those of BC. In contrast, the free lime contents of OPC pastes were lower at older ages of hydration if compared with those of BC. The free lime contents of belite blended cements increased only up to 7 days of hydration, and then started to decrease onward. This is mainly attributed to the pozzolanic abilities of both HPFS and SF with a part of the released free lime resulting from the hydration of silicate phases of both cements, which responsible for the decrease of free lime contents [16-18,23,31,36,38,42,50,52,63].

3.7. Heat of hydration

The heat of hydration of the pure OPC and Belite hardened cement pastes (P0 and B0) and the optimum belite cement pastes containing 15 % HPFA and 5 % SF (B4) is graphically plotted versus the hydration ages up to 90 days in Figure 10. As soon as the cement powders become in contact with water, a heat due to hydration was generally generated. The amount of the released heat increased as the hydration time progressed up to 90 days. The same trend was displayed by all cement pastes. This is mainly contributed to the continuous and gradual increase of the rate of hydration [5,6,63-66]. Furthermore, the rate of the generated heat of hydration was sharply enhanced at early ages up to 7 days. This is essentially attributed to the activation effect of the hydration reaction mechanism of $\text{C}_3\text{S}$ by the very fine HPFA and SF particles. At later ages (28-90 days), the rate of hydration reaction was reduced, and therefore the evolved heat of hydration slightly increased. This may be due to the activation action mechanism of $\beta\text{-C}_2\text{S}$ by the fine HPFA and SF particles [38,65,66]. The heat of hydration of the OPC cement at later ages of hydration (28-90 says) seemed to be stable, while that of BC increased more than that of OPC pastes. This was attributed to the fact that the hydration of The OPC phases started as soon as the cement powder became in contact with water, whereas in BC pastes it begins after 28 days of hydration [5,6]. The heat of hydration of the optimum cement pastes (B4) are the lowest, while that of the OPC pastes are the highest.

![Figure 10](image-url). Heat of hydration of the pure OPC and Belite hardened cement pastes (P0 and B0) and the optimum belite cement pastes (B4)
3.8. Ultrasonic velocity test

Ultra-sonic pulse velocity method (USPV) is mainly carried out to assess the uniformity and relative quality of structures and specimens. Hence, the ultrasonic pulse velocity (USPV) of the OPC (P0) and belite hardened cement (B0) as well as the optimum belite cement pastes containing 15 % HPFA and 5 % SF (B4) is graphically drawn versus the hydration ages up to 90 days in Figure 11. The USPV of the blank cements (OPC and B0) slightly increased only up to 7 days, and then seemed to be stable, i.e. the various constituents of blank cements (P0 and B0) did not affect by the ultrasonic pulse velocity [47,60]. It is good mention that the same trend was displayed by the compressive strength. The addition of 15 % HPFA with 5% SF (B4) to belite cement could be successfully applied without any adverse effects on the physical, chemical, mechanical and durable properties of the cement. Therefore, it was selected to be the optimum batch. The free lime content of the blank OPC and BC cements (P0 and B0) gradually increased as the hydration time progressed up to 90 days, while the free lime contents of all blended cement pastes containing different ratios of HPFA (B0-B5) increased only up to 7 days and then suddenly decreased. The heat of hydration of the optimum belite/Fly ash cement batch (B4) was lower than those of the belite cement control (B0) at all hydration times. This means that the belite blended cement release a lower rate of heat of hydration. The USPV test demonstrated that B4 assess the best conformity of the matrix. Moreover, all the obtained results are in a good agreement with each other.

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Conflict of interest
The author declares that there is no conflict of interest anywhere.

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