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Effect of silica fume and fly ash on heat of hydration of Portland cement

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Abstract

Results of calorimeter tests on Portland cement–silica fume–fly ash mixtures are presented. Data indicate that silica fume accelerates cement hydration at high water/cementitious ratios and retards hydration at low water/cementitious ratios. On the other hand, fly ash retards cement hydration more significantly at high water/cementitious ratios. When silica fume and fly ash are added together with cement, the reactivity of the silica fume is hampered and the hydration of the cementitious system is significantly retarded. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Heat of hydration; Portland cement; Fly ash; Silica fume

1. Introduction

Investigations into the use of silica fume (SF) in high-strength concrete have been underway for more than 30 years [1]. Cementitious composites containing SF as one of the key ingredients for producing "high-performance concrete" generally have compressive strengths in excess of 100 MPa. Strengths greater than 150 MPa have been reported by a number of researchers [2–4]. Benefits realized when utilizing SF include [5–9]:

1. Substantial increases in compressive strength of concrete while maintaining the same mix design parameters;
2. Reduction in the required cement content for a specific target strength; and
3. Increased durability of hardened concrete when added in optimum amounts.

Fly ash (FA) has been widely utilized in concrete since it reduces cost of the concrete materials, conserves energy and resources and reduces environmental problems. The effects of FA on the various properties of concrete have been the subject of numerous investigations such as reported in ACI Committee 226 [10] Report on the Use of Fly Ash in Concrete in 1987.

Problems are also associated with using this material, as FA has a relatively low surface area and accompanying pozzolanic activity. At normal temperatures, the pozzolanic reaction is slow to start and it does not progress to any significant degree until several weeks after the start of hydration. This results in slow strength development and inadequate strength at the normal age of loading, even though the concrete may have higher strength and durability in the longer term. To achieve the desired concrete properties, some special curing regimes such as prolonged moist curing may have to be used to ensure adequate early strength development [1].

Overcoming the effects of FA on the early age properties of fly ash–cement mixtures is still a challenge. Silica fume appears to be a potential solution to this problem due to its highly reactive nature. Silica fume may provide significant amounts of calcium silicate hydrates (CSH) at an early age which would be expected to increase the early age strength. This research program was initiated to investigate the influence of SF and FA on the hydration of cement based mixtures at early ages.

2. Experimentation

Materials used for this research included a Canadian Standards Association (CAN/CSA-A5) Type 10 Portland

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cement, CAN/CSA-A23.5 Type C fly ash and SF. The chemical compositions and physical properties of all the cementitious materials are given in Table 1.

Type 10 Portland cement was used as the basic cementitious material. Silica fume was added as a partial replacement of the cement at levels of 5% and 10% by weight of the total cementitious material. FA was also added as a partial replacement of the cement at levels of 10%, 20% and 30% by weight of the total cementitious material. Mix designations at each w/c' ratio are based on the amount of fly ash and SF present, e.g. S10A20 indicates 10% SF and 20% fly ash. Calorimeter tests were performed on these mixtures at water/cementitious ratios (w/c') of 0.35, 0.40 and 0.50. Most tests were run for a period of 24 h. However, several were carried on for 72 h to observe any later reactions.

Heat of hydration can be measured under isothermal or adiabatic conditions [11]. Measurement of heat of hydration in this study was carried out under isothermal conditions (25 ± 0.1 °C). The calorimeter used was a thermoelectric conduction type, interfaced to a computer by a data acquisition system.

Each test consisted of a 16-g sample that was placed into the calorimeter cup. The cup was then put into the calorimeter and held for 5–6 h to attain temperature equilibrium. The required amount of water was injected into the cup by means of a hypodermic needle through a hole in the lid of the calorimeter. The data acquisition system was initiated at the same time to record the output voltage from which the heat flow in the system can be calculated.

The reproducibility of the results was checked. Duplicate tests showed that total heat measurements were within ±3%. Rate of heat evolution did vary somewhat in the first few minutes but after 30 min the rates were essentially identical.

Table 1
Chemical composition and physical properties of Type 10 Portland cement, silica fume and fly ash (%)

	Type 10 cement	Silica fume	Fly ash
SiO_2	20.7	92–94	57.81
Al_2O_3	3.68	0.20–0.30	23.01
CaO	63.0	0.10–0.15	9.90
Fe_2O_3	2.95	0.10–0.50	3.50
MgO	4.21	0.10–0.20	1.50
Na_2O	0.14	0.10 (Na)	2.30
K_2O	0.59	0.10 (K)	0.50
SO_3	2.62	0.10 (S)	0.30
C		3–5	
Free CaO	1.02		
Physical properties:			
Loss on ignition	2.7		0.50
Specific gravity	3.15	2.02	2.04
Average particle size (μm)		0.1	
Bulk density (kg/m^3)		320.4	

3. Results and discussion

3.1. Effect of SF on hydration

The effect of SF varies depending on the w/c' ratio. Fig. 1 shows the effect of a 10% SF addition on the rate of heat evolution of the cement–SF mixtures at three w/c' ratios. The rate of heat evolution was calculated on the basis of a unit weight of plain Portland cement, thus, the rates can be compared with each other and the contributions from SF and FA can be separated. Fig. 1a shows the results for w/c' ratio of 0.35. The difference occurs mainly after 2 h. For the plain cement (S0A0), the acceleration period begins at approximately 1.5–2 h after introduction of the water and ends approximately 5.5 h later. For the 10% SF mixture (S10A0), the acceleration period begins at about 2.0–2.5 h and ends at roughly 8.5 h. The rate of heat of hydration of SF mixtures surpasses that of plain cement at 7.5 h, approximately at the end of the acceleration period. The two curves are similar after 20-h hydration.

These results indicate that the addition of SF alters the hydration process in the period 2–20 h after the start of hydration. The dormant period is prolonged while the rate of the acceleration period is reduced and the rate of the deceleration period is increased.

For the w/c' ratio of 0.40 (Fig. 1b), plain cement exhibits little difference from the w/c' ratio of 0.35. The SF mixture differs in that the rate of heat of hydration surpasses that of plain cement at about 7.0 h, and the second peak (8.5 h) is higher than for plain cement. There is a tendency for the curve to shift to the left, indicating a higher rate of heat of hydration than at the lower w/c' ratio of 0.35.

In Fig. 1c (w/c' ratio of 0.50), the shift is more apparent. Here the rate of heat of hydration of the SF mixtures surpasses that of plain cement at about 6 h. The two curves move closer together as the w/c' ratio increases. In this case the SF does not seem to prolong the dormant period, but increases the rate of heat evolution in the later stages of the acceleration period. This is similar to the findings of Huang and Feldman [12]. For the low w/c' ratios, SF promotes different effects at different periods in the hydration process. It prolongs the dormant period, reduces the rate of heat of hydration during the acceleration period and increases the rate after the acceleration period. Thus, the acceleration effect of SF begins earlier as the w/c' increases.

Accumulative heat of hydration for various periods of hydration of plain cement and 10% SF are shown in Table 2. These results indicate that the presence of SF increases heat evolution during the first 30 min of hydration, and during the period from 8 to 24 h regardless of the w/c' ratio. Heat evolved during the dormant period remained almost constant for all mixtures, while the heat during the period from 2 to 8 h is reduced. Total heat evolved at 1 and 3 days is not changed by the presence of SF at w/c' ratio of 0.35. Total heat at 1 day does increase with an increase in w/c'.

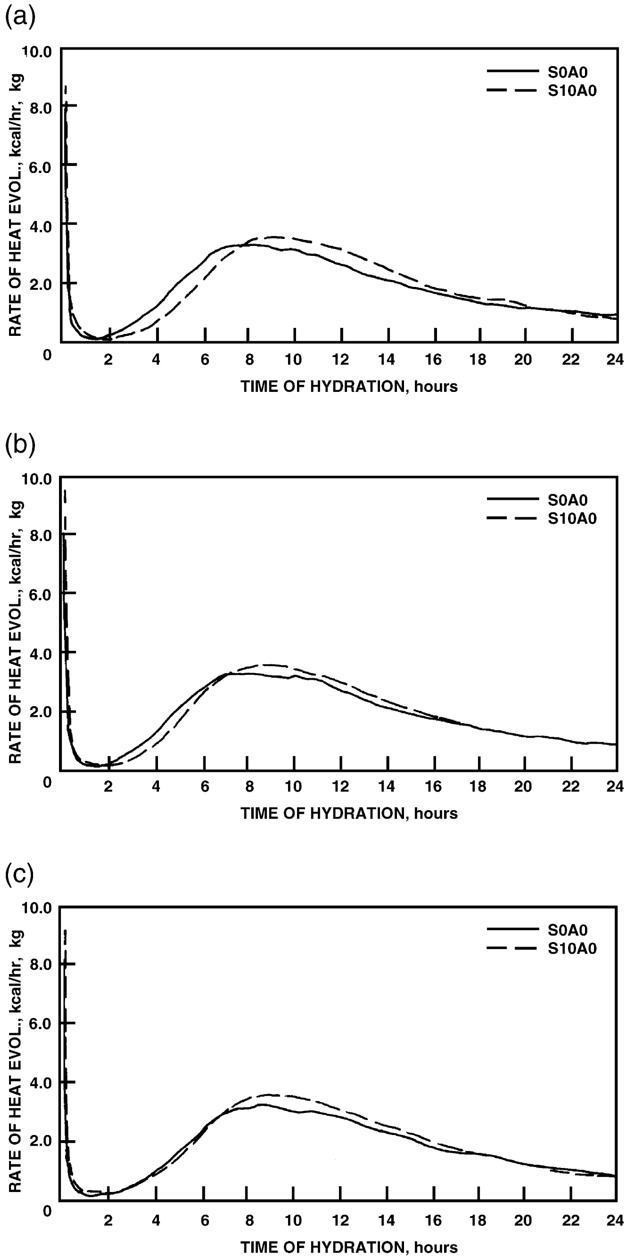

Fig. 1. Heat evolution of cement and silica fume mixture: (a) $w/c'=0.35$; (b) $w/c'=0.40$; (c) $w/c'=0.50$.

Table 3
Early heat evolution characteristics of cement and silica fume mixture

	w/c'	Time of second peak (h)	Rate of second peak (kcal/h, kg)	Time of surpassing (h)
S0A0	0.35	7.3	3.3	–
S10A0	0.35	9.0	3.6	7.9
S0A0	0.40	8.0	3.3	–
S10A0	0.50	8.5	3.7	7.2
S0A0	0.50	8.2	3.2	–
S10A0	0.50	8.5	3.7	6.0

The second peaks for plain cement and SF pastes approach each other as the w/c' ratio increases (Table 3). The rate of the second peak is higher for the SF mixture and the time of surpassing the control decreases as the w/c' ratio increases. This suggests that SF increases the second exothermic reaction and as the w/c' ratio increases, the accelerating effect of the SF begins earlier.

Effects of SF on the rate and amount of heat evolution may be attributed to its chemical and physical effects on the cement hydration process. Grutzeck et al. [13] observed that SF underwent rapid dissolution in the calcium hydroxide solution and formed a new phase on the SF particles in a matter of minutes. This new phase is a calcium poor silica rich layer and may serve as a substrate for the formation of conventional CSH gel. In the first few minutes of hydration there is a rapid release of Ca^{2+} and alkali ions from the cement compounds, the reduction in Ca^{2+} in the solution increases the rate of release and amount of heat evolution, i.e. hydration at this stage is accelerated by SF.

During the dormant period, the main process underway is the diffusion of calcium ions through the film of hydrate products to the liquid phase. Silica fume adsorbs the calcium ions to its surface and reduces the concentration of Ca^{2+} in the solution. This effect may accelerate the process of diffusion, as shown by the results at the higher w/c' ratio and as reported by others [12,14]. The prolonged dormant period at the low w/c' ratio can be explained by the water adsorption behaviour of SF and the formation of a SF layer on the cement particles. Silica fume is composed of very small particles which when mixed with water may form agglomerations and are soon covered with a gel-like layer. Water may become enclosed in this layer, which has been suggested as the cause of the rapid water consumption of SF mixtures [13,15]. If the w/c' ratio is low, cement and water do not come into full contact initially as the voids may not be full of water. Adsorption decreases the water content available for cement hydration resulting in less cement taking part in the hydration process. Thus, SF acts

Table 2
Effects of silica fume on heat evolution of Portland cement hydration (kcal/kg, cement)

	w/c'	0.0–0.5 h	0.5–2.0 h	2.0–8.0 h	8.0–24 h	Total at 1 day	Total at 3 days
S0A0	0.35	2.6	0.4	11.7	31.3	45.9	56.9
S10A0	0.35	3.1	0.4	8.7	34.5	46.6	56.1
S0A0	0.40	2.6	0.5	11.8	31.8	46.7	–
S10A0	0.40	3.2	0.4	10.3	33.6	47.4	–
S0A0	0.50	2.6	0.4	10.2	33.3	46.4	–
S10A0	0.50	3.2	0.5	9.7	35.3	48.7	–

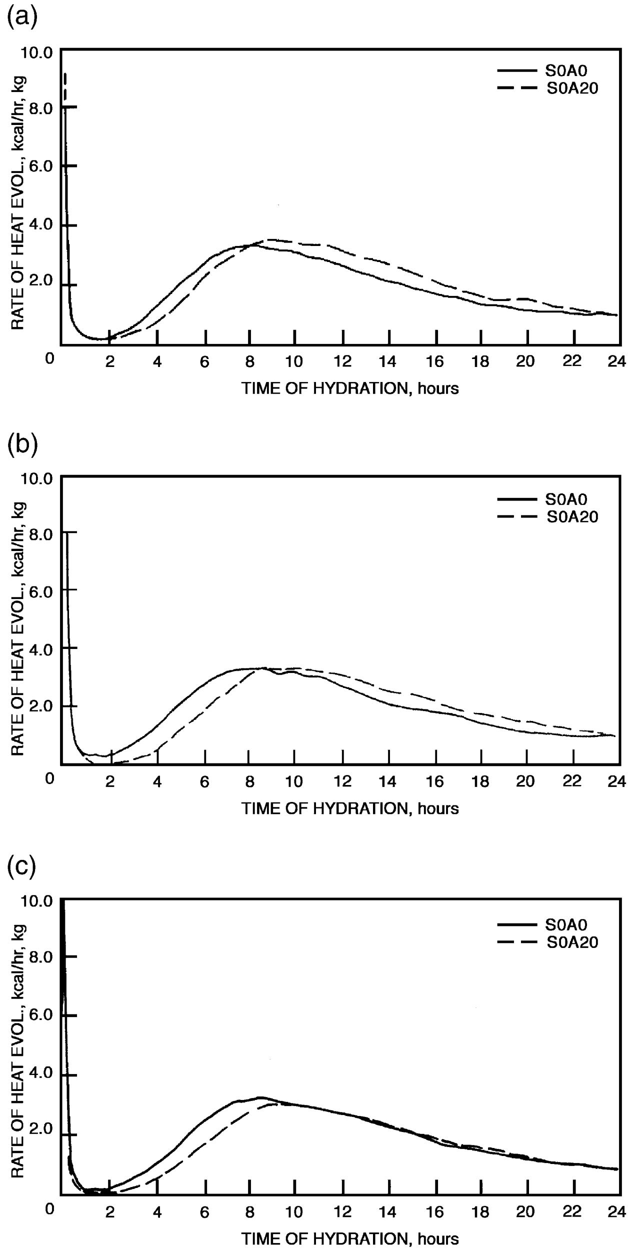

Fig. 2. Heat evolution of cement and fly ash mixture: (a) $w/c' = 0.35$; (b) $w/c' = 0.40$; (c) $w/c' = 0.50$.

as a retarder to the process. The formation of SF layers on the surface of the cement particles also contributes to the delay in hydration [15–17].

Chatterji et al. [17] stated that around each cement particle there were approximately 100,000 grains of SF. Clearly, this number depends on the size of the cement and SF particles, and the percentage of SF in the mixture. Water required for the hydration of the cement grains will have to percolate through the layers of SF. The resistance to this water flow would increase with an increase in the thickness of the SF layer, i.e. with a lowering of the w/c' ratio. In the dormant period, the SF layer inhibits the diffusion of water to the cement particle surface. This mechanism does not seem to apply at higher w/c' ratios. At these higher ratios, the voids between particles are originally filled with water and the particles may be fully separated by a water film. In this case SF will dissolve and move into the solution instead of adhering to the cement particle surfaces as both surfaces have a silica rich layer (negative charge). Silica fume, even though it adsorbs some water, does not reduce the amount of water available for cement hydration. The higher the w/c' ratio, the more the SF is dissolved and remains in solution for rapid dissolution reaction. Therefore, the retarding effects reduce as the w/c' increases.

The breakdown of initial hydration products on the surface of cement particles and exposure of additional new surface area to water initiates the acceleration period. In this stage, the main hydration component is C_3S. This is accomplished by the crystallization of CH from the supersaturated solution. The influence of SF on this stage is that the previously dissolved surface of SF or the previously formed gel-like, silica-rich, calcium-poor intermediate phase [13] provides nucleating sites for the precipitation of hydration products, especially lime. This process dominates during the acceleration period even though reaction of CH with SF may occur. At the higher w/c' ratio where SF particles are all well dispersed in the liquid phase as explained above, the increased number of fine particles in the solution provide nucleation sites for deposition of CH and alumina silica hydrates, thus, hydration is accelerated. These results agree with the results obtained by other researchers [12,14–16,18]. Thus, SF starts to accelerate the reaction earlier at higher w/c' ratios. At lower w/c' ratios, this effect is not evident in the test results. Whether or not there is a contribution to heat evolution from the reaction between SF and CH at this stage is not known.

The rate of heat of hydration of SF mixes surpasses that of plain cement mixtures indicating the accelerated hydration of cement by SF. By carefully examining the mech-

Table 4
Effects of fly ash on the heat evolution of Portland cement hydration (kcal/kg, cement)

	w/c'	0.0–0.5 h	0.5–2.0 h	2.0–8.0 h	8.0–24 h	Total at 1 day	Total at 3 days
S0A0	0.35	2.6	0.4	11.7	31.3	45.9	56.9
S0A20	0.35	3.2	0.4	8.91	35.4	47.9	59.1
S0A0	0.40	2.6	0.5	11.8	31.8	46.7	–
S0A20	0.40	2.8	0.3	7.81	35.8	46.6	–
S0A0	0.50	2.6	0.4	10.1	33.2	46.4	–
S0A20	0.50	2.9	0.2	7.11	33.4	43.5	–

Table 5
Early heat evolution characteristics of cement and fly ash mixtures

	w/c'	Time of second peak (h)	Rate of second peak (kcal/h, kg)	Time of surpassing (h)
S0A0	0.35	7.3	3.3	–
S0A20	0.35	9.0	3.5	8.0
S0A0	0.40	8.0	3.3	–
S0A20	0.50	10.0	3.3	8.1
S0A0	0.50	8.2	3.2	–
S0A20	0.50	10	3.0	12.0

anism of the pozzolanic reaction proposed by Greenberg [19] and the dissolution of SF in an alkaline environment, establishing the contribution to the heat of hydration from the pozzolanic reaction between the SF and CH is possible. According to Greenberg [19], when finely divided materials are present in an aqueous solution, the silica will react with water to form a saturated solution of monosilicic acid as shown in Eq. (1).

$$SiO_2(\text{solid}) + 2H_2O(\text{liquid}) \rightarrow H_4SiO_4(\text{aqueous}) \qquad (1)$$

In SF-blended systems, alkalis go into solution causing more silica to dissolve. Ca^{2+} and OH^{-} in the pore solution become chemically adsorbed by the silanol groups to form CSH, as shown in Eqs. (2) and (3):

$$H_2SiO_4(\text{aqueous}) + nCa^{2+}(\text{aqueous}) + 2nOH^{-}(\text{aqueous}) \rightarrow nCaO \cdot SiO_2 \cdot (n+2)H_2O \qquad (2)$$

$$SiO_2(\text{solid}) + nCa^{2+}(\text{aqueous}) + 2nOH^{-}(\text{aqueous}) \rightarrow nCaO \cdot SiO_2 \cdot nH_2O \qquad (3)$$

where n is the CaO/SiO_2 ratio.

Dissolution of silica, increases with pH and chemisorption of Ca^{2+} can only occur after silica has gone into a solution following the attack by OH^{-} ions. Grutzeck et al. [13] observed a rapid dissolution of SF in the first few minutes after mixing the SF with CH solution and the formation of a silica rich layer on its surface. Labri et al. [18] indicated that dissolution of silica becomes significant at pH values greater than 12. It is well known that this pH value can be achieved during the dormant period (calcium supersaturated state). Therefore, chemisorption or chemical reaction can occur both at this stage because such a reaction can occur between CH previously deposited on the SF surface and the gel-like intermediate phase [13].

3.2. Effects of fly ash

Results of heat of hydration of FA mixtures are illustrated in Fig. 2. As with SF, the effects of FA also differ at different w/c' ratios.

At a w/c' ratio of 0.35 (Fig. 2a), the FA mixture begins its acceleration period at about 3.0 h. The rate of heat of hydration of FA mixtures surpasses that of plain cement at approximately 8.0 h, about the end of the acceleration period. The peak occurs at 9.0 h and the maximum is a slighter higher than the plain mix. The results indicate that FA retards hydration mainly in the dormant and acceleration periods.

At a w/c' ratio of 0.40 (Fig. 2b), the retarding effect appears more significant than at the lower w/c' ratio. The curve of the FA mixture departs from that of the plain cement in the period from 2.0 to 8.0 h. This mixture begins its acceleration period at about 2.5 h and ends at 9.5 h. The

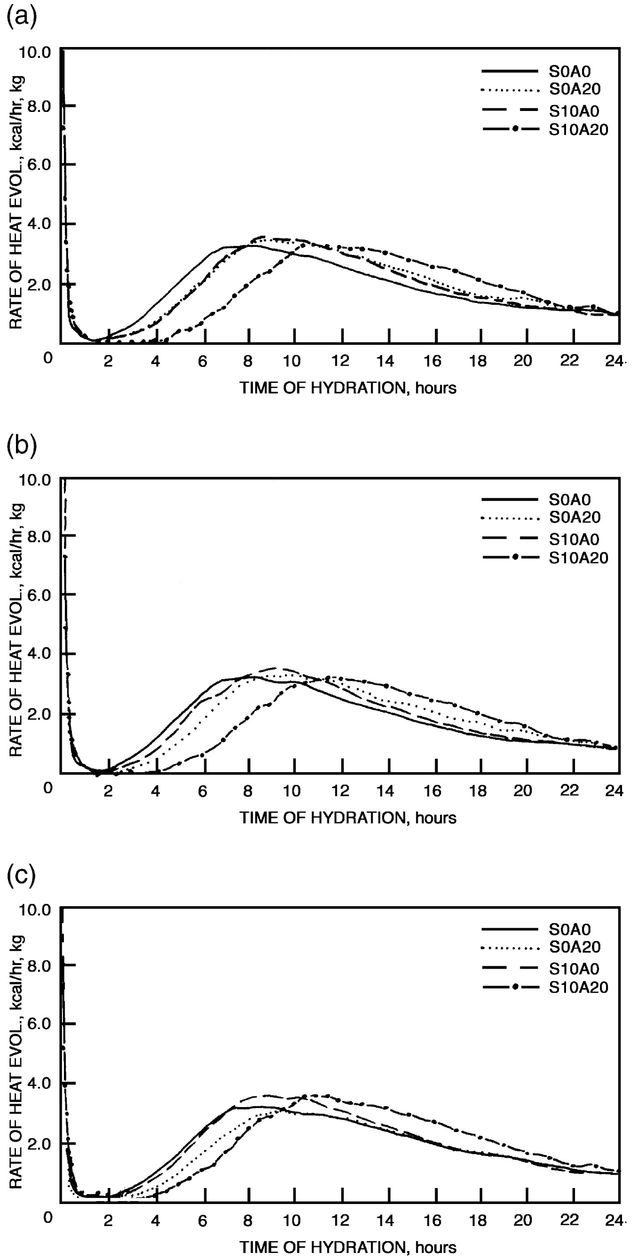

Fig. 3. Heat evolution of cement, silica fume and fly ash mixtures: (a) $w/c' = 0.35$; (b) $w/c' = 0.40$; (c) $w/c' = 0.50$.

Table 6
Effects of silica fume and fly ash on heat evolution of cement hydration (kcal/h, kg cement)

	w/c'	0.0–0.5 h	0.5–2.0 h	2.0–8.0 h	8.0–24 h	Total at 1 day	Total at 3 days
S0A0	0.35	2.6	0.4	11.7	31.3	45.9	56.9
S10A0	0.35	3.1 (2.8)	0.4 (0.4)	8.7 (7.8)	34.5 (31.1)	46.6 (41.9)	56.1 (50.5)
S0A20	0.35	3.2 (2.6)	0.4 (0.3)	8.9 (7.1)	35.4 (31.9)	47.9 (38.3)	59.1 (47.3)
S10A20	0.35	2.4 (1.7)	0.3 (0.2)	3.1 (2.2)	37.1 (26.0)	42.9 (30.0)	53.2 (37.2)
S0A0	0.40	2.6	0.5	11.8	31.8	46.7	–
S10A0	0.40	3.2 (2.9)	0.4 (0.4)	10.3 (9.3)	33.6 (30.2)	47.4 (42.7)	–
S0A20	0.40	2.8 (2.2)	0.3 (0.2)	7.8 (6.2)	35.8 (28.6)	46.6 (37.3)	–
S10A20	0.40	1.9 (1.3)	0.5 (0.4)	3.7 (2.6)	39.1 (27.4)	45.1 (31.6)	–

Data in brackets are normalised to per unit weight of total mixture.

time of surpassing is delayed to a little more than 9.0 h and its peak height is reduced marginally.

At a w/c' of 0.50 (Fig. 2c), the retardation effect becomes much more apparent. The FA mix begins its acceleration period at about 2.5 h and ends at 9.5 h, and the height of the second peak is reduced significantly.

These results seem to be contrary to that observed with SF mixtures where the retarding effect of SF is reduced, but the accelerating effect increases as the w/c' ratio increases. The retarding effect of FA is more significant at higher w/c' ratios.

The cumulative heat of hydration in the first 3 days is summarized in Table 4. Heat evolved during the first 30 min is increased in the presence of fly ash. Heat evolved in the period from 0.5 to 2.0 h is reduced at high w/c' ratios, while heat evolved in the period from 2.0 to 8.0 h is significantly reduced as the w/c' ratio increases. Heat evolved during the period from 8.0 to 24.0 h is increased at low w/c' ratios (0.35, 0.40). Total heat at 1 day and 3 days increased at low w/c' ratios but decreased at high w/c' ratios.

The time of the second peak is increased at all w/c' ratios (Table 5). The height of the second peak is increased a small amount at the low w/c' ratio and reduced at the high w/c' ratio. This means that as the w/c' ratio increases, the retarding effect of FA increases. Therefore, the addition of FA accelerates hydration in the first few minutes, reduces hydration in the dormant periods, which is followed by accelerated hydration.

FA is believed to be inert at this very early stage. Its effect on the 24-h hydration seems to be the result of a change in the amount of water available for cement hydration and the different mechanisms at the different stages of hydration. During the first minutes, heat of hydration tends to increase with the addition of FA as more water is available for the initial hydrolysis. This is consistent with Wei et al. [20]. In the dormant period, the increase in water reduces the calcium concentration in the pore solution. Therefore, it prolongs the dormant period by increasing the time when the supersaturated state can be reached. The reduction in Ca^{2+} is usually attributed to the presence and the condition of the FA [19]. It has been suggested that FA acts like a Ca sink in that it removes calcium ions from the solution. This depresses the calcium concentration in the first hours and delays the CH and CSH nucleation and crystallization, thus, retarding hydration [21,22]. Cabrera and Plowman [23] observed similar results where C_3S and C_3A hydration were retarded. In the following periods, the increase in water may help the hydration process because more water is available to react with the newly exposed surfaces. The increased heat of hydration may also be attributed to the increased number of nucleation sites by FA for deposition of cement hydrates. This effect enhances the hydration after the dormant period. These results agree with those observed by other researchers [16,24].

3.3. Effects of fly ash and SF combined

Fig. 3 presents results when 10% SF and 20% FA were added to Portland cement. Tables 6 and 7 show heat of hydration results at different periods of hydration and the early heat evolution characteristics. From these results, it can be seen that SF–fly ash mixtures have the lowest heat evolution rates and the longest dormant period, i.e. hydration is significantly retarded. The second peak is the latest to arrive and the decreased heat evolution results from the hindered reaction of the SF with the calcium hydroxide. In the presence of both pozzolans, the pH value is greatly reduced due to the chemisorption of Ca^{2+} on both SF and FA particles, which reduces the calcium ion concentration in the solution. The dissolution of SF, which dominates the early reactivity of the SF was significantly depressed. It can be seen by comparison that the time for the second peak to occur is decreased and the height is increased as the w/c' ratio increases. This results from the increased influence of the SF. Thus, in the presence of FA, the reactivity of the SF is delayed and the hydration of the cement is further retarded.

Table 7
Early heat evolution characteristics of cement and silica fume and fly ash mixtures

	w/c'	Time of second peak (h)	Rate of second peak (kcal/h, kg)	Time of surpassing (h)
S0A0	0.40	8.0	3.3	–
S10A0	0.40	9.0	3.7	7.2
S0A20	0.40	10.0	3.3	8.1
S10A20	0.40	12.0	3.2	10.0

4. Conclusions

Based on the results obtained in this study, it has been shown that SF accelerates cement hydration at high w/c' ratios. At low w/c' ratios, SF retards cement hydration and prolongs the dormant period, followed by enhanced hydration of the cement. Initial hydration of the cement is usually accelerated by the presence of SF. The higher the w/c' ratio, the higher accelerating effect of the SF.

FA also increases the initial hydration of cement. However, it retards hydration in the dormant and acceleration periods. It also accelerates hydration after the acceleration period. The higher the w/c' ratio, the greater the retardation effect.

When SF and fly ash are incorporated together in cement, the hydration of the cement is significantly retarded. The heat of hydration is decreased and the early reactivity of the SF is hampered. The accelerating effect of the SF is delayed.

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