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Short communication

The effect of fly ash fineness on heat of hydration, microstructure, flow and compressive strength of blended cement pastes

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ABSTRACT

In this paper, an experimental study on the effect of fly ash fineness on the heat of hydration, microstructure, flow and compressive strength of blended cement pastes was carried out and evaluated against control cement paste. Fly ashes with different fineness: classified fly ash, run-of-station fly ash and grounded run-of-station fly ash; with a median particle size of 17.4, 11.3 and 5.7 μm, respectively, from the same power station source in Australia were used to partially replace Portland cement at 20% and 40% by weight of cement using a fixed water-to-binder ratio of 0.40. Results of this study showed that the cumulative heat of hydration of blended cement paste decreased as fly ash content in blended cement paste was increased. For a given cement replacement level, blended cement paste containing finer fly ash released more heat of hydration when compared to coarser fly ash. Moreover, increasing the fineness of fly ash resulted in a higher consumption of calcium hydroxide at 7 and 28 days reflecting pozzolanic reactivity and, thus, a denser microstructure than blended pastes containing coarser fly ash as revealed by the X-ray diffraction (XRD), scanning electron microscopy (SEM) and compressive strength results. In addition, the incorporation of fly ash in the blended pastes led to the introduction of an additional hydration peak in the heat evolution curve possibly due to the late activation of fly ash by calcium hydroxide renewing the C_3A reaction and converting ettringite to monosulfate. The flow of the freshly blended cement pastes was also found to improve slightly with increasing fineness of the fly ash. In addition, the hardened blended cement pastes containing 20% ground run-of-station fly ash showed comparable compressive strength with the control cement pastes at both 7 and 28 days mainly due to the higher fineness of the ground run-of-station fly ash and increased reactivity compared to coarser grade fly ash.

1. Introduction

Utilization of fly ash as a supplementary cementitious material (SCM) addition for partial replacement of cement in concrete introduces several economical, technical and environmental benefits such as the conservation of natural resources and the reduction of greenhouse gas emissions [1]. Coal mineralogical composition, degree of pulverization and type of

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furnace and firing temperature used all have an impact on the physical and chemical properties of the fly ash generated [2]. Fineness, shape morphology and particle size distribution of fly ash are the most crucial factors which influence the fresh and hardened properties of concrete [3]. In addition, the hydration process in blended cement pastes containing fly ash is more complex than that of Portland cement due to the occurrence of two inter-related time-dependent processes, namely cement hydration and the pozzolanic reaction of fly ash [4]. The cement hydration process can be distinguished by five stages in heat generation in early ages as reported by several researchers [5,6] and as shown in Fig. 1: (I) the initial stage: immediately within the first few minutes of mixing cement with water, the aluminate reacts with water and sulphate, forming a gel-like material (ettringite) surrounding the cement grains. This reaction releases a significant amount of heat and is represented by the first peak of the hydration process; (II) the dormancy stage: there is a dormant stage for about two to four hours after mixing in which the aluminate reaction is controlled by the amount of ettringite gel surrounding the cement grains due to limiting the access of water to the cement grains, which controls the rate of the aluminate reaction, and thus little heat is released; (III) the acceleration stage: after super-saturation of the pore solution with calcium ions mainly from dissolving alite and belite (main cement minerals), fibre-like calcium silicate hydrate (C-S-H) gel and crystalline calcium hydroxide (C-H) start to form with significant heat evolution. The acceleration stage is represented by the second peak of hydration; (IV) the deceleration stage: interaction of C-S-H gel and crystalline C-H with remaining water and undissolved cement grains slows down the alite reaction thus reducing the heat of hydration. The amount of sulphate starts to deplete and thus the remaining aluminate reacts with ettringite to form monosulphate. The formation of monosulphate generates little heat, which may be associated with the third hydration peak; (V) the slow continued reaction stage: belite dissolves and releases calcium ions very slowly and starts to produce C-S-H and C-H after several days. However, as long as alite and belite remain in the cement system and there is enough water available in the system, the silicates will continue to hydrate [5,6].

Investigations on the influence of the utilization of fly ash on the properties of concrete have been carried out by many researchers [7,8]. However, there is limited information available in literature on the effect of fineness of fly ash, with similar chemical composition, on the heat of hydration and microstructure of the blended cement pastes. The present paper, therefore, attempts to bridge this gap in knowledge by carrying out an experimental study on the effect of fly ash fineness on the heat of hydration, microstructure and fresh and hardened properties of blended cement pastes.

2. Experimental program

2.1. Material properties

General purpose Portland cement (Type GP) conforming to AS 3972 [9] with a median particle size of 9.4 μm was used to produce the cement pastes in this study. Fly ashes of different fineness, from Eraring power station in New South Wales in Australia, were used as the source of supplementary cementitious materials (SCMs) in this study. Three types of fly ash, namely Classified fly ash (CFA), Run-of-station fly ash (RFA) and Grounded run-of-station fly ash (GRFA) classified as a grade 1, grade 2 and special grade according to AS 3582.1 test method, respectively, were evaluated for their performance in blended cement pastes. The oxide composition using X-ray fluorescence (XRF) analysis and physical properties of the GP cement (PC) and fly ashes are shown in Tables 1 and 2, respectively. The percentage passing 45 μm sieve, the Blaine fineness and specific gravity of the cementitious materials used in this study were determined according to AS 2350.9, AS 2350.8 and AS 3583.5 test methods, respectively [10–12]. Table 1 shows that there is no significant difference in the oxide composition of the three (3) types of fly ashes used in this study. Particle size distribution using laser diffraction and particle shape using scanning electron microscopy (SEM) are presented in Figs. 2 and 3, respectively. All SEM micrographs have been taken at the same magnification level (x500). From Fig. 2, it can be seen that the particle size distribution of all cementitious materials is

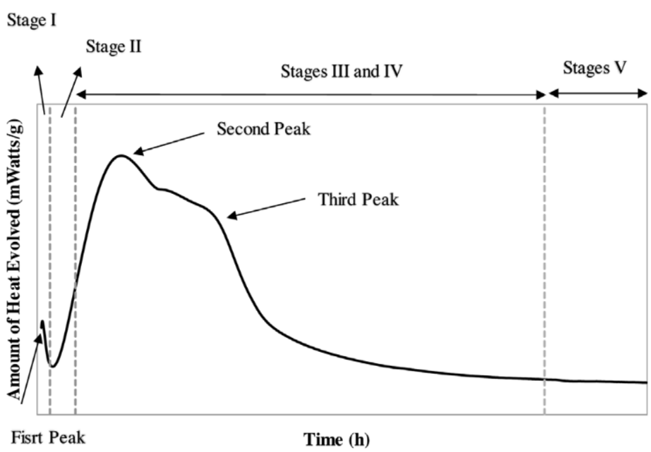


Fig. 1. Common cement heat evolution curve [5].

Table 1
Oxide composition of cement and fly ashes.

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	MgO	Na_2O	K_2O	P_2O_5	SO_3	L.O.I.
PC	19.23	5.12	2.86	64.26	0.31	1.28	0.18	0.51	0.12	2.73	4.06
CFA	59.21	28.11	3.68	2.48	1.11	0.53	0.63	1.18	0.41	0.16	1.05
RFA	60.06	26.97	3.65	3.00	1.05	0.59	0.62	1.24	0.39	0.17	1.20
GRFA	60.06	26.97	3.65	3.00	1.05	0.59	0.62	1.24	0.39	0.17	1.20

Table 2
Physical properties of cement and fly ashes.

Materials	Median particle size (μm)	Specific Gravity	Passing 45 μm sieve (%)	Blaine fineness (m^2/kg)	28-day Strength Index (%)
PC	9.4	3.09	94	396	100
GRFA	5.7	2.11	100	495	107
CFA	11.3	2.08	85	368	83
RFA	17.4	2.06	68	302	72

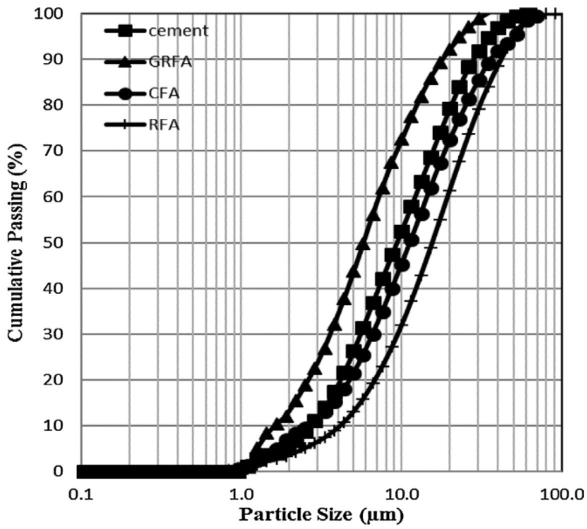


Fig. 2. Particle size distribution of cement and fly ashes.

consistent with the results of the Blaine fineness reported in Table 2. A reduction in particle size has led to increased fineness with a higher specific surface area reported. Table 2 shows that the effect of the grinding process applied to fly ash, which has resulted in reduced median particle size of the RFA below that for CFA and PC. As shown in Fig. 3, the grinding process has also altered the shape of some of the fly ash particles from spherical to angular.

2.2. Mixture proportions, mixing and curing

In both cement and blended cement pastes, the water-to-binder ratio was maintained at 0.40. In the blended cement pastes, three (3) types of fly ashes (CFA, RFA and GRFA) were used to replace GP cement at the same replacement levels of either 20% or 40% by weight of the cement. After dry mixing the cement and fly ash in a Hobart mixer, the required amount of water was added and mixing was continued to achieve a uniform consistency in the mixture. Mixture designations are based on the cement replacement level with fly ash by weight of cement, e.g. 20CFA indicates a mixture with 20% cement replacement with CFA.

Immediately after the completion of mixing, the fresh paste was assessed for its workability using the mortar flow test according to ASTM C1437 [13]. A number of 50 mm cement paste cubes were cast in steel moulds and compacted by hand using a tamping rod. For compressive strength test, the cube specimens were then sealed in plastic bags to prevent moisture loss. After 24 h, the cube specimens were demoulded and then stored in lime saturated water for curing period up to 28 days.

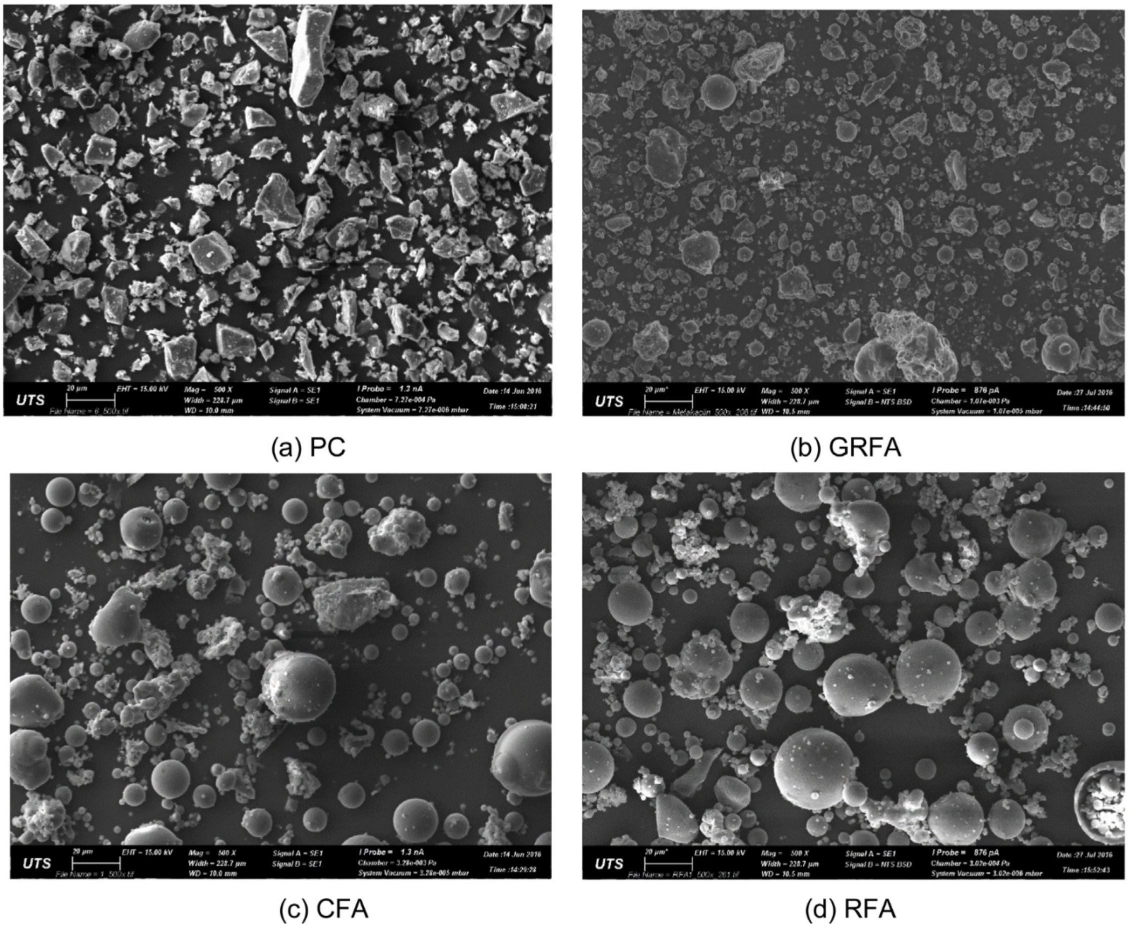


Fig. 3. SEM micrographs of cement and fly ashes (a) PC (b) GRFA (c) CFA (d) RFA (x 500).

2.3. Test details

2.3.1. Isothermal calorimetry

The heat of hydration of blended cement pastes was determined using an I-Cal 4000 isothermal calorimeter in accordance to ASTM C1679 [14]. Since the weight of the test sample was relatively small, mixing of the paste was carried out manually stirring the cement, the fly ash and the water togther with a steel rod in a plastic calorimeteric cup. The sealed cup was then placed into the calorimeter for monitoring the heat evolved for 48 h.

2.3.2. X-ray diffraction (XRD)

The blended cement paste fragments from the cube specimens were ground using a ball mill at the specified ages of 1, 7 and 28 days. One gram of powdered sample was used to measure the amount of Portlandite (P) by XRD analysis using a Bruker D8 Discover diffractometer.

2.3.3. Scanning electron microscope (SEM)

Cube specimens were fractured into small pieces at 1, 7 and 28 days. The fractured surfaces were then coated and placed under vacuum prior to imaging using a Zeiss Supra 55VP SEM.

2.3.4. Compressive strength

The compressive strength of hardened pastes was determined in accordance with ASTM C109 [15] at the ages of 1, 7 and 28 days.

3. Results and discussion

3.1. Isothermal calorimetry analysis

Figs. 4 and 5 show the amount of heat evolution of the blended cement pastes containing 20% and 40% GRFA, CFA and RFA. The blended pastes showed less heat generated up to 10 h compared to the control cement paste devoid of fly ash addition.

The first peak, which signifies the initial reaction at the surface of cement particles and represents the onset of hydration of the C_3A phase was mostly neglected, as mixing of the paste mixtures was performed outside the calorimeter. The height of the second peak was found to decrease for an increase in fly ash content in the blended pastes. A probable reason for this occurrence might be due to the dilution effect of cement with less C_3S available for increasing fly ash addition. Moreover, the slow reaction that results between alumino-silicates in fly ash and calcium hydroxide (C-H) liberated from cement due to the lower solubility index of fly ash in pore solution could be another reason for the observed trend in data [16].

The high fineness of GRFA blends also showed a higher peak intensity for the second peak compared to the CFA and RFA blends. These observations are supported by the findings of Rahhal and Talero [17] who reported higher peak intensities with an increase of fineness of fly ash particles. The peak intensity of the second peak for the GRFA20 blend was found to be 3.51 mW/g compared to 3.36 mW/g and 3.27 mW/g for the CFA20 and RFA20 blends, respectively.

Figs. 4 and 5 also show the third peak is more apparent for the blended pastes. The height of this peak was found to increase when the fly ash content was increased from 20% to 40%. Compared to the blended pastes, the third peak was also found to be absent from the control cement paste. The increase in peak height observed for an increase in fly ash content could possibly be due to the reaction of more tricalcium aluminate (C_3A) and the subsequent conversion of more ettringite to monosulfate as fly ash enhances the aforementioned reaction by providing a high number of nucleation sites for the

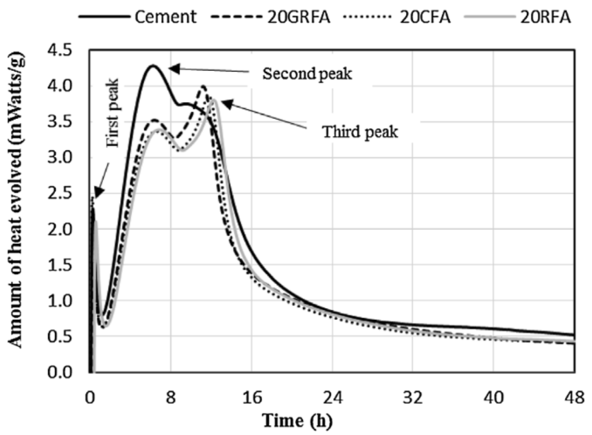


Fig. 4. Heat evolution curve for blended pastes with 20% fly ash content.

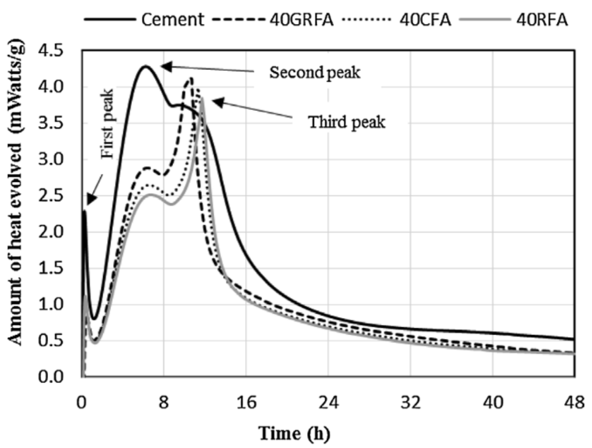


Fig. 5. Heat evolution curve for blended pastes with 40% fly ash content.

hydration products of more calcium aluminate to precipitate [6,18]. The C_3A is highly soluble and reacts rapidly with the gypsum present in the cement to form ettringite within several minutes of mixing; however, by gradually decreasing the concentration of sulphate ions in the pore solution over a period of several hours the ettringite becomes unstable and converts to monosulfate. In addition, replacing cement with fly ash decreases the gypsum content and increases the reactivity of C_3A (8.7309%, C_3A content of cement calculated from Bogue equation) in the mixture allowing the C_3A particles to undergo renewed and rapid hydration. This hydration reaction involves an exothermic process and contributes to the heat generated in the third peak [6,19]. Figs. 4 and 5 illustrate this feature and show the height of the third peak increasing with increasing fineness of the fly ash due to an increasing number of the nucleation sites available for the hydration reaction to proceed.

Fig. 6 shows the cumulative heat of hydration for cement and blended pastes over the testing period of 48 h. As expected, the cumulative heat of hydration generated for the blended pastes are lower than that for cement paste. Increasing the fly ash content in the blends also led to reducing the amount of heat evolved. The combined effect of diluting the cement and increasing the amount of fly ash available in the system has contributed to the observed trends in data [20]. Snelson et al. [16] reported that the relatively low specific surface area and the low solubility of the aluminosilicate present in the fly ash are the main contributing factors for a reduction in the cumulative heat evolved.

3.2. XRD analysis

Fig. 7 shows the XRD diffractogram patterns at the age of 7 days for cement and blended pastes with 20% fly ash content of varying fineness. The XRD pattern of pure calcium hydroxide (CH) has also been included for better understating the intensity of the peaks and comparing against the portlandite detected in the XRD patterns of the blended cement pastes. Portlandite peaks representative of CH appeared at 2-Theta of 18.07, 28.75, 34.13, 47.12, and 50.85°. At early age, fly ash does not have enough influence to decrease the amount of C-H available in the system despite the fact that the fly ashes have different particle size distributions. It is clearly seen that the intensity of the portlandite peaks are almost the same in all fly ash blends. This is possibly due to the slow pozzolanic reactivity of fly ash. There are no significant changes in the hydration process at this stage and the hydration of the cement is the dominant phase. This is in agreement with the findings of other researchers reporting the hydration reactions taking place at 7 days with the main reaction involving cement rather than fly ash [21,22].

The XRD patterns for the blended pastes containing 20% of GRFA, CFA and RFA at 28 days are also shown in Fig. 8. The results indicate that all blends have reduced portlandite available in the system with increasing fineness of fly ash augmenting this difference. A probable reason to the noted decrease in portlandite peak intensity could be due to the increased reactivity of the finer grade fly ash having a higher surface area, resulting in increased nucleation sites for pozzolanic reactivity [15]. Sybertz and Wiens [23] also found that fly ash with smaller particle size has higher amorphous content, which increases the pozzolanic reactivity due to the presence of more silica and alumina in the system and thus more C-H is consumed.

3.3. SEM observations

Figs. 9 to 11 show the SEM micrographs of the blended cement pastes with 20% GRFA, CFA and RFA content at the ages of 1, 7 and 28 days. All micrographs have been taken at the same magnification level (x3.00 K). At 1 day, most of the fly ash particles appear to be unreacted with smooth surfaces still present. Though, compared to CFA and RFA particles, the finer grade GRFA particles appear to be covered with fibrous C-S-H gel or ettringite, which suggests a faster rate of reaction, however no EDAX analysis was available to confirm that. The faster reaction of GRFA particles is possibly due to the increased

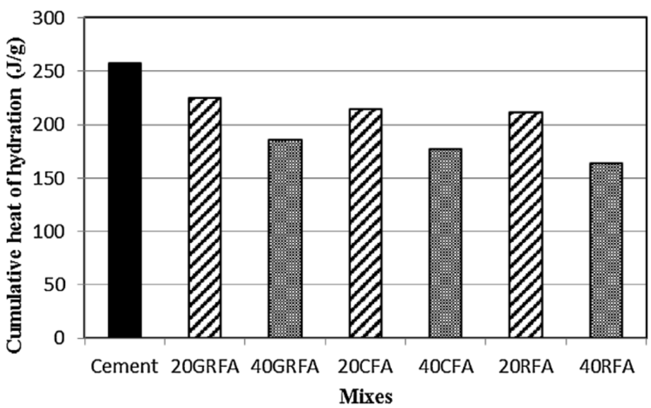


Fig. 6. Cumulative heat of hydration after 48 h for cement and blended pastes.

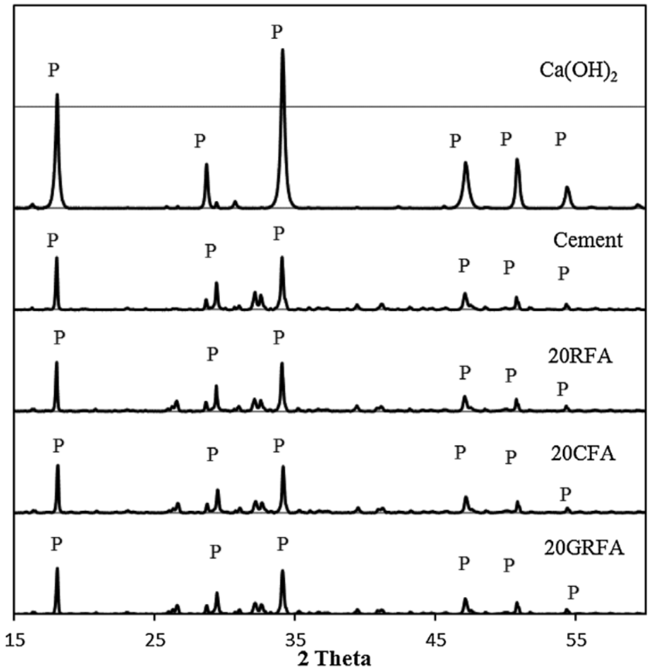


Fig. 7. XRD patterns of cement and 20% blended pastes at 7 days (P = portlandite).

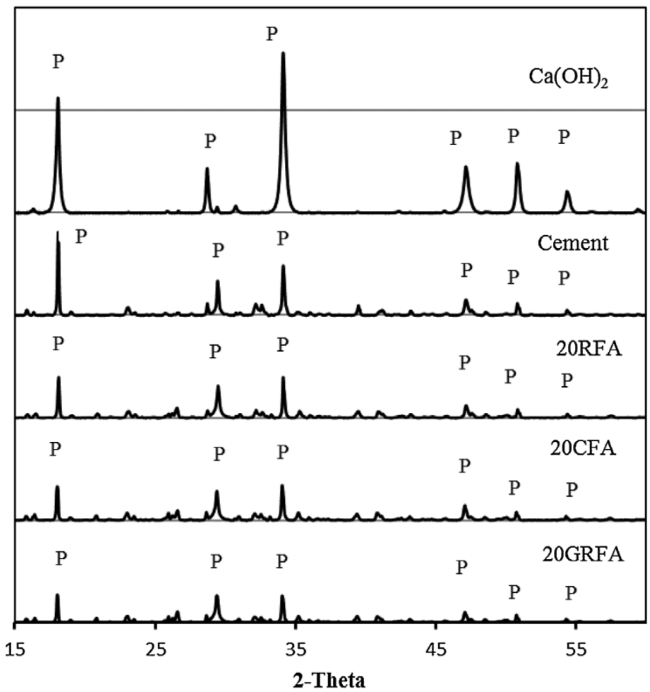
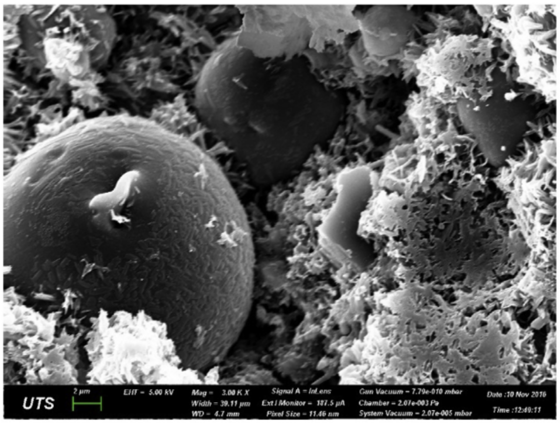
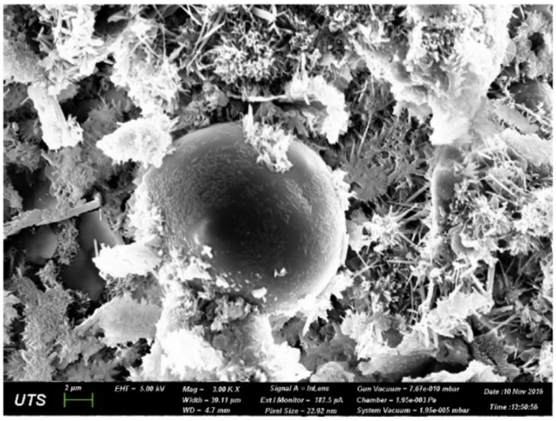


Fig. 8. XRD patterns of cement and 20% blended pastes at 28 days (P = portlandite).

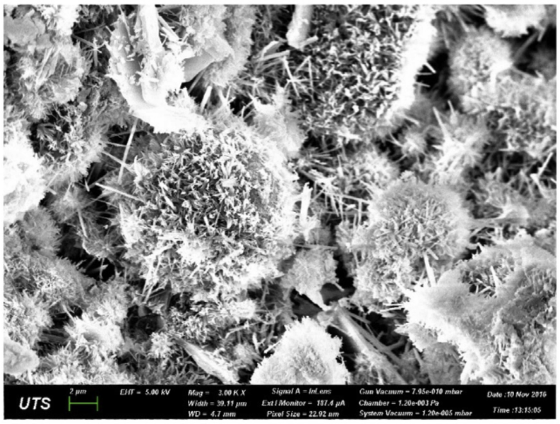
fineness of GRFA thereby increasing the surface area available for the pozzolanic reaction. Furthermore, the presence of needle-like crystals representative of ettringite are also apparent in all blends. At this age, interstices between fly ash particles and empty pores are evident in all blends due to incomplete hydration. This is in agreement with the findings of Chindaprasirt et al. [24] and Xu and Sarkar [25] who reported the same phenomena.



(a) 20RFA



(b) 20CFA



(c) 20GRFA

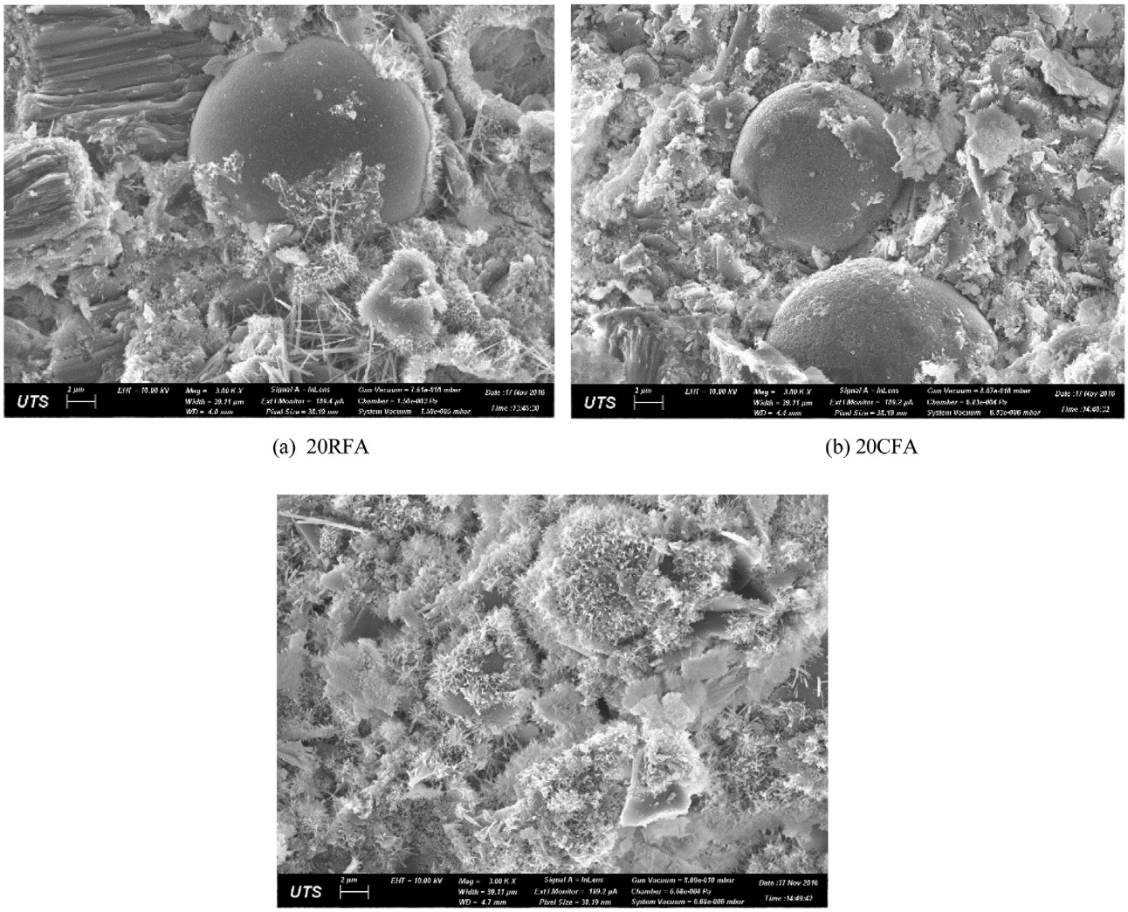
Fig. 9. SEM micrographs of blended cement pastes with 20% fly ash at 1 day (x3.00 K).

At 7 days (Fig. 10), C-S-H gel starts to fill the spaces between the fly ash particles. However, less C-S-H was found to be present in the RFA blends possibly due to less pozzolanic reactivity prevailing for the coarser grade of fly ash present in the system [26]. These observations are in agreement with the XRD results reported in Section 3.2, which showed more C-H presence in RFA blends and thus less C-S-H formation. A large number of GRFA particles were also observed to be coated on the surface with more hydration products (Fig. 10c) compared to CFA particles, which have been coated with a thinner layer of hydration products (Fig. 10b). Most of the RFA particles were found to be uncoated and unreacted (Fig. 10a).

At the age of 28 days, almost all the fly ash particles (GRFA, CFA, and RFA) were observed to be encased with hydration products. The continuous voids between fly ash particles were found to be filled contributing to a denser structure as shown in Fig. 11. The high fineness grade of GRFA (Fig. 11c) appear to be more involved in the pozzolanic reaction compared to the lower fineness grade of CFA and RFA (Fig. 11a and b). Further evidence of this level of pozzolanic reactivity is supported by XRD data in Fig. 8, which showed lower amounts of calcium hydroxide present in the GFRA systems compared to CFA and RFA systems. Moreover, a significant number of GRFA particles have also reacted and converted to C-S-H with more fibrous sheets evident (Fig. 11c). This is not the case for the coarser grade CFA and RFA particles with less C-S-H present. The finer particle size of GRFA has reacted more rapidly than the coarser particle size of CFA and RFA despite similar oxide compositions such as SiO_2, CaO and Na_2O_e. The finer fly ashes are effective in making more nucleation sites for pozzolanic reactions as well as reducing the porosity than the coarser fly ashes. However, some fly ash particles were observed to still remain smooth and unreacted in all systems, which suggests these particles have acted in part as inert material to improve the particle packing density of the cementitious matrix [26,27].

3.4. Flow and compressive strength results

The effect of replacing cement with 20% and 40% GRFA, CFA and RFA on the flow of the blended pastes are shown in Fig. 12. The flow varied between 105% and 140% for all the pastes evaluated in this study. It can be seen that GRFA and CFA marginally improved the flow of the blended pastes at both 20% and 40% fly ash contents. This could be due to improving the ball bearing



(a) 20RFA

(b) 20CFA

(c) 20GRFA

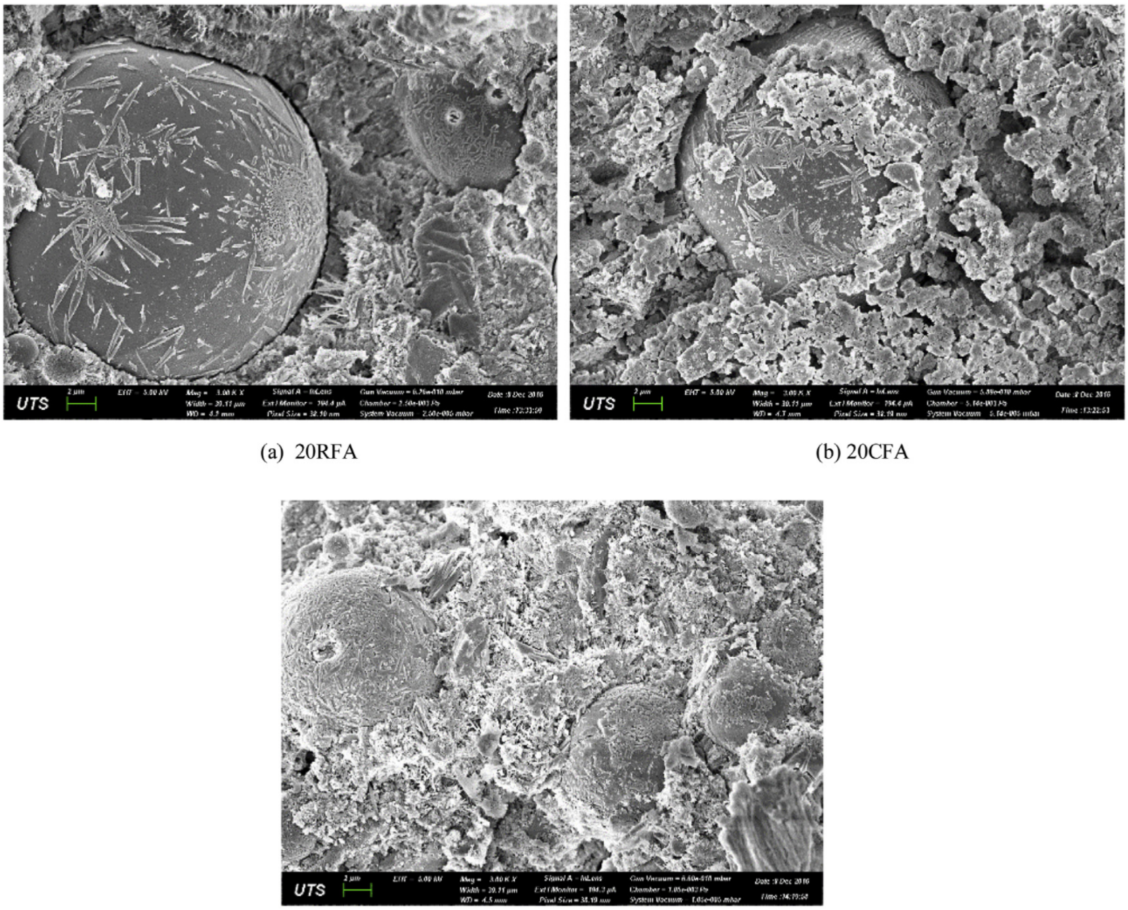
Fig. 10. SEM micrographs of blended cement pastes with 20% fly ash at 7 days (x3.00 K).

effect of blended cement pastes resulting from the spherical shape of the fly ash particles with the effect being more apparent with increasing fly ash content. In addition, the reduction in the water absorption capacity of the fly ash particles due to their inherent glassy and smooth surface texture compared to cement particles could be another reason for improving the flow. GRFA also showed the improvement in workability due to the increased amount of finer sized fly ash particles available per unit mass, which increased the lubricant effect in comparison to coarser sized particles (CFA and RFA) despite having more angular particles as shown in SEM images.

Ferraris et al. [28] reported that lower particle density of fly ash compared to that for cement makes more paste content for a fixed binder content by weight and thus improves the flow, especially in mixtures containing the finer grade fly ash. Mora et al. [29] stated a similar relationship with the flow of mortars improving for decreasing particle size of fly ash.

Figs. 13 and 14 show the development of compressive strength with age for blended pastes containing 20% and 40% CFA, RFA and GRFA. Fig. 15 shows the relative strength of blended pastes at 7 and 28 days compared to cement paste devoid of fly ash addition. The results show that increasing the fly ash content from 20% to 40% contributes to decreasing the compressive strength at both 7 and 28 days. However, the strength may well continue to increase beyond 28 days for the mixes containing fly ash due to the slow pozzolanic reaction between the fly ash and calcium hydroxide generated from cement hydration [30,31]. The relative strength of the blended pastes was found to increase with the use of fly ash of increased fineness. The noted increase in relative strength for increased fineness could be possibly due to the consumption of more C-H and the formation of more C-S-H gel in the system due to the pozzolanic reaction. These observed trends are in agreement with the XRD and SEM results reported in Sections 3.2 and 3.3, respectively, as well as the findings of other researchers [30–33]. A similar relationship is observed for the CFA blends having gained more strength compared to the RFA blends at both 7 and 28 days.

Fig. 15 shows that at the ages of 7 and 28 days, the compressive strength of the blended pastes containing 20% GRFA is comparable to the control cement paste devoid of fly ash addition. At the age of 28 days, the blended paste with 20% GRFA achieved about 96% of the strength of that of the control paste. However, the relative strength for the blended CFA and RFA



(a) 20RFA (b) 20CFA

(c) 20GRFA

Fig. 11. SEM micrographs of blended cement pastes with 20% fly ash at 28 days (x3.00 K).

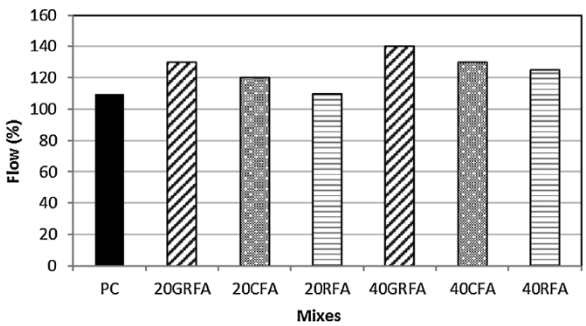


Fig. 12. Flow of the pastes containing fly ashes with different fineness and cement replacement levels.

pastes at the same cement replacement level was about 83% and 72%, respectively. At the same age of 28 days, an increase in fly ash content from 20% to 40% caused the relative strength of the GRFA, CFA and RFA pastes to decrease to 71%, 59% and 51%, respectively.

Although the oxide compositions of all fly ashes investigated in this study were reported to be the same, it is quite possible that a more disordered structure exists in the GFRA compared to the CFA and RFA. Iyer and Scott [34] have reported that a higher level of reactivity of fly ash prevails for a more disordered structure of fly ash arising from such factors as changes in temperature and duration of combustion and quenching during the manufacturing process of the fly ash.

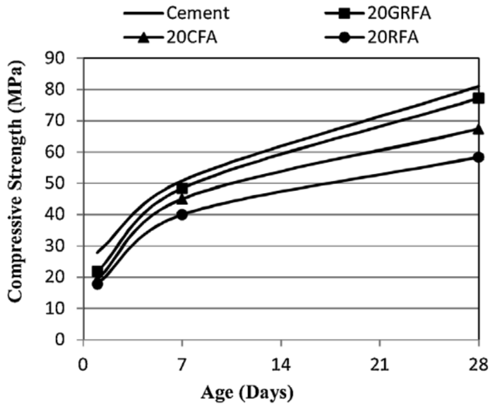


Fig. 13. Compressive strength of blended cement pastes containing 20% fly ash content of different fineness.

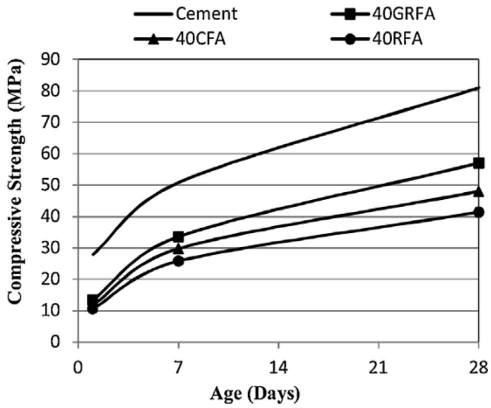


Fig. 14. Compressive strength of blended cement pastes containing 40% fly ash content of different fineness.

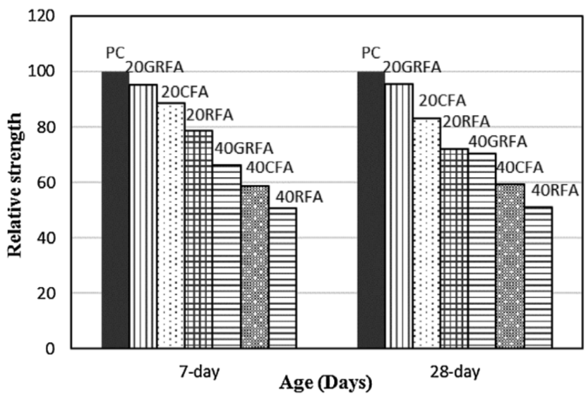


Fig. 15. Relative compressive strength of blended cement pastes containing varying fly ash content of different fineness.

4. Conclusions

From the isothermal calorimetry, XRD, SEM, flow and compressive strength tests performed on the blended cement pastes and two (2) cement replacement levels of fly ashes of different fineness, the following conclusions can be drawn.

1 Partially replacing cement with fly ash, of different fineness, decreased the cumulative heat evolution; the reduction in heat evolved increased with an increase in fly ash content. However, the finer grade fly ash generated more heat of hydration compared to the coarser grade fly ash.
2 The blended paste with ground run-of-station fly ash exhibited increased hydration products on the fly ash particle surfaces, especially at 28 days according to the SEM results.
3 The XRD results indicated that the fineness of fly ash did not have any significant influence on the consumption of the calcium hydroxide at the ages of 1 and 7 days; however, the consumption of calcium hydroxide increased at 28 days by increasing the fineness of the fly ash.
4 Increasing the fineness of fly ash marginally influenced the flow of the blended cement pastes.
5 The blended cement paste containing finer grade fly ash demonstrated higher compressive strength than the coarser grade fly ash at both replacement levels of cement (20% and 40%). The rapid pozzolanic reaction observed for finer grade fly ash by consuming more C-H and producing more secondary C-S-H gel in the system makes the microstructure denser compared to the mixture containing coarser grade fly ash, and thus increases the compressive strength as supported by XRD and SEM results. In addition, the blended paste containing 20% ground run-of-station fly ash showed comparable results to the control cement paste devoid of fly ash addition at both 7 and 28 days.

Conflict of interest

None.

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