

Microstructural, physical, and thermal analyses of Portland cement–fly ash–calcium hydroxide blended pastes

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Abstract The effect of calcium hydroxide (CH) on the properties of Portland–fly ash cement pastes, at up to high-volume fly ash mixes has been investigated using normal consistency, setting time, compressive strength, thermal analysis and scanning electron microscope. CH as an additive material (5 and 10 wt%), lignite fly ash (FA) up to 50 wt% was used to produce Portland cement (PC)–FA–CH pastes at w/PC + FA ratio of 0.5. Water requirement for normal consistency was found to increase with increasing CH content while a decrease in initial setting time was found. Furthermore, the compressive strengths of all FA mixes with CH were found to be higher than the mixes without CH. Thermal analysis and scanning electron microscope were used to study the hydration of PC–FA–CH system. The results showed that the first phase transition detected by thermal analyses was attributed to ettringite, calcium silicate hydrate, gehlenite hydrate and was found to be higher in PC–FA–CH mixes than in pure Portland–FA cement paste resulting in an increase in compressive strength. Moreover, the hydration phases were also found to increase with increasing curing time. Overall, the results show that the additional of 5 wt% CH in Portland–FA mixes especially at high-volume FA mixes was found to accelerate FA pozzo-

lanic reaction at early ages (7 and 28 days), resulting to an increase in compressive strength.

Keywords Fly ash · Additive materials · SEM · Mechanical property · Hydration

Introduction

Fly ash (FA) is a by-product of coal-fired electric plant. It is a pozzolanic material and is used worldwide to replace cement because of its lower cost and its improvement to some properties of fresh concrete such as workability and decreased heat of hydration. Moreover, FA can also increase compressive strength and improve the durability of concrete construction such as reduced expansion from sulfate attack and penetration of chlorides to steel reinforcement of concrete structure and is used for constructions such as buildings, dams, and bridges. Not only FA gives durability benefit but also for its ecological benefit by reducing the industrial wastes from coal power plants and indirectly helps to reduce air pollution by cutting down carbon dioxide (CO₂) emission [1–5]. Two general classes of FA can be defined: siliceous FA produced by burning anthracite or bituminous coal, and calcareous produced by burning lignite or sub-bituminous coal. The glassy phases in these ashes are sometimes pozzolanic, consuming calcium hydroxide (Ca(OH)₂ also noted as CH) upon hydration. FA contributes strength to concrete by pozzolanic reaction. It is known that pozzolanic reaction is the reaction between SiO₂ and Al₂O₃ (which are the component of pozzolanic material) with CH to form a hydration product that is a cementitious binder [1–7]. But, in early age, the replacement of Portland cement (PC) with FA especially in high-volume have been found to result in a reduction in the

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strength of concrete due to the slow pozzolanic reaction between FA and CH [8–11] and a partial substitution of FA for cement can lead to concrete strengths equal to or greater than that for pure PC after 90 days [12]. This is a barrier for the application of FA concretes in precast industries due to the slow strength development for FA concrete. Therefore, it is of prime importance to improve the early strength of FA concretes to obtain full benefits of FA to precast industries [10].

Some researchers reported improved early strength of FA mixes using many techniques such as reducing a coarse FA size [13], using a superplasticizer to reduce the water to binder ratio [14] and accelerating the pozzolanic reaction by autoclave method [15]. Amer [16] and Giergiczny [17] studied FA–Ca(OH)₂ reaction (no PC) using thermal analyses to show formation of calcium silicate hydrate (C–S–H). Many researchers [4, 18–21] have already reported on accelerating FA pozzolanic reaction using activators such as Na₂SO₄, CaCl₂, CaSO₄, calcined desulphurization gypsum, and silica fume in order to improve the unsatisfactory early properties of PC paste with FA added as a cement replacement. Moreover, it is reported by Barbhuiya et al. [10] that there is another way to improve the pozzolanic reaction of FA by adding CH and improved compressive strength to FA concrete was found. Thus, the improvement in the low early strength development (before 28 days) of FA concrete is required and the effect of this combination of both FA and CH is not well understood. Hence, the interest of this paper is to investigate the effect of CH on the strength and hydration of FA pastes up to high-volume FA content. The durability of this system in concrete is out of scope of the present study and should be studied in detail in the future.

Fly ash from Mae Moh power plant in Northern Thailand, produced from lignite coals (noted as lignite FA) was used. The aim of the present study is to examine the hydration mechanism of PC–FA–Ca(OH)₂ mixes using different content of FA up to 50 wt% with and without additional CH—used as additional material especially at the early ages. Microstructural, physical, and thermal analyses of PC–FA–CH blended pastes were investigated and are reported in this paper.

Experimental techniques

Chemical compositions of PC type I, lignite FA and CH are given in Table 1. FA has been tested for pozzolanic activity index with lime as reported in term of the pozzolanic strength at 10.78 MPa according to ASTM C311-93 method [22]. Moreover, free lime content of PC and FA was measured using titration method with 0.1 M hydrochloric acid [23]. The Bogue compounds of PC were

calculated giving the compound as follows; C₃S = 54.5%, C₂S = 18.6%, C₃A = 7.2%, and C₄AF = 10.7%. The mix proportion of Portland–FA cement pastes is given in Table 2. FA was used to replace PC at 10, 20, 30, and 50 wt%. CH was then used as an additional material at 5 and 10 wt% of PC and FA binders. The normal consistency testing was carried out to determine the water requirement to achieve the same paste consistency, according to ASTM C187-98 method [24]. Setting time tests were performed according to ASTM C191-1a method [25].

For compressive strength tests, the water/PC + FA ratio of these cement pastes was 0.5. To help homogeneously, the binders (PC, FA, and CH) were first mixed in the mixer for 3 min, after that the water was added and mixed for another 2 min. The pastes were then poured into oiled molds (50 × 50 × 50 mm³) and then compacted. The specimens were surface-smoothed, and covered with plastic film. All specimens were then removed from the molds 24 h after casting. Thereafter, they were cured in water at 25 °C. Three specimens of each mix were tested for compressive strength at 7, 28, and 60 days after curing.

The microstructure of the paste specimens was studied using scanning electron microscopy (SEM; JEOL-JSM-840A). These samples were also ground for mineralogical composition analysis using X-ray diffraction (XRD; Philips PW 1729 using Ni-filtered Cu K radiation) and thermogravimetric (TG) analysis was performed by Mettler Toledo TGA/SDTA 851° heated from 30 to 1,000 °C with scanning rate of 10 °C/min in nitrogen atmosphere.

Results and discussion

The water requirement for normal consistency ASTM C187 [24] and setting time ASTM C191 [25] of PC–FA–CH mixtures are shown in Table 2. The water demand of FA mixes (w/b = 0.20–0.24) was reduced with increasing FA content which was also lower than PC paste (w/b = 0.25). These are similar to reports by other researchers [1, 2, 5, 13] and are because of the spherical particles with smooth surfaces of FA. Moreover, when CH added, the water requirement of PC–FA–CH mixtures was found to increase with increasing percentage of CH content up to 10 wt% (w/b = 0.28 for 10FA10CH mix).

For setting time test, it is observed that both initial and final setting time of all FA mixes increased with increasing FA content, compared with PC paste because of the reduction in cement content and the increment in water to PC ratio [2, 5, 13, 26]. Moreover, the setting time of Portland–FA cement pastes with CH as additive material was shorter than the corresponding mixes without CH (compared to the mixes with the same of FA content). However, for all the cases of Portland–FA cement paste

Table 1 Chemical composition of PC, FA, and CH

Materials	Oxide content/%											The pozzolanic strength/MPa
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃	LOI	Free lime	
PC	20.85	4.98	3.52	64.3	1.53	0.12	0.59	-	2.60	1.40	0.71	-
FA	39.82	21.52	13.68	15.24	2.78	1.08	2.00	0.18	2.39	1.00	2.18	10.78 ± 6.68%
CH	1.34	1.03	0.89	66.83	3.27	0.57	0.07	0.15	-	25.53	-	-

Table 2 Mix proportions, normal consistency, and setting time of PC–FA–CH pastes

Mixes	w/PC + FA	Mix proportion/wt% of PC			Water requirement for normal consistency/g, (w/b), (w/PC)	Setting time/min	
		PC	FA*	CH**		Initial	Final
PC	0.5	100	-	-	125, (0.25), (0.25)	135	190
10FA	0.5	90	10	-	119, (0.24), (0.26)	146	217
10FA5CH	0.5	90	10	5	130, (0.26), (0.26)	115	155
10FA10CH	0.5	90	10	10	143, (0.28), (0.32)	90	133
20FA	0.5	80	20	-	116, (0.23), (0.29)	151	230
20FA5CH	0.5	80	20	5	127, (0.25), (0.32)	119	178
20FA10CH	0.5	80	20	10	138, (0.27), (0.35)	96	146
30FA	0.5	70	30	-	110, (0.22), (0.31)	154	232
30FA5CH	0.5	70	30	5	123, (0.24), (0.35)	121	182
30FA10CH	0.5	70	30	10	133, (0.26), (0.38)	103	163
50FA	0.5	50	50	-	100, (0.20), (0.40)	169	240
50FA5CH	0.5	50	50	5	112, (0.22), (0.45)	124	189
50FA10CH	0.5	50	50	10	125, (0.25), (0.50)	105	170

*Cement replacement

**Additional material

with and without CH, the initial setting time were all within the standard (refer to ASTM C191).

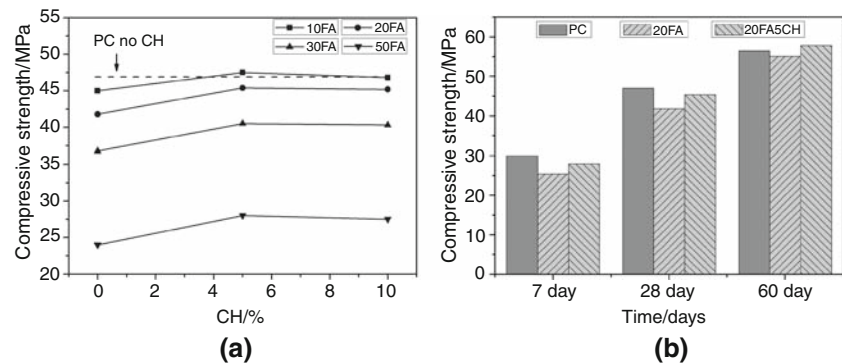
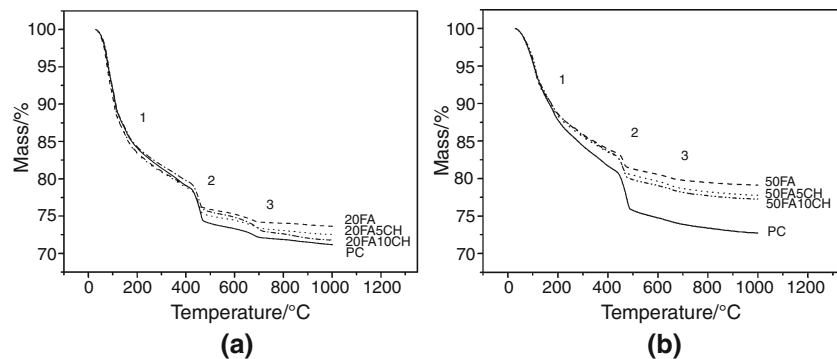
Compressive strength results of Portland–FA cement paste after curing in water for 7, 28, and 60 days are shown in Table 3 and Fig. 1a and b. All compressive strengths of Portland–FA mixtures without CH are lower than the control PC mix when compared at the same curing. However, this lower strength is generally found [8–11] for PC–FA mixes. Nevertheless, it is noticed that the compressive strength of 10FA5CH mix was found to be slightly higher than the control PC mixture at all curing times. Moreover, all Portland–FA cement pastes with CH were found to have increased strength compared to the FA mixture references (the same FA replacement without CH) as shown in Table 3. Relative strengths to PC were used as a way to determine the degree of hydration reaction and pozzolanic reaction [13]. It can be noticed that the 10FA5CH mix was found to have highest hydration and pozzolanic reaction up to 100.67, 101.06, and 103.89% for 7, 28, and 60 days, respectively. Furthermore, the results were then presented as the relative strength to the mixes without CH (compared at the same FA content and curing times) as represented in degree of pozzolanic reactivity can be seen in Table 3. It is

noticed that the pozzolanic reactivity of all mixes with CH are significantly higher than those mixes without CH where the highest strength at 7 days of 50FA10CH mix gave as much as 24% increase in pozzolanic reactivity. Furthermore, 50FA5CH mix gave the highest pozzolanic reaction with an increase up to 17% for both 28 and 60 days, agreeing with the report by Barbhuiya et al. [10]. These results confirm that the utilization of the additional CH can improve the pozzolanic property of Portland–FA blended cement.

Thermalgravimetric analysis results of Portland–FA cement pastes with and without CH when replaced with FA at 20 and 50 wt% with different CH additional content is shown in Fig. 2a and b, to give a typical representation of reaction of PC–FA with CH (at percentage and at high volume). The TG results are represented as the quantitative mass loss of paste products, as shown in three steps similar to the phases reported by Vessalas et al. [6], Amer [16], Chaipanich and Nochaiya [21], and Roszczynialski [27]. The first step, between ≈ 30 and 420 °C, shows the mass loss of water from both humidity and molecular water (H₂O) in the hydrated phases (called dehydration) which combine several hydrated phases such as ettringite, C–S–H,

Table 3 Results of compressive strength, relative strength to PC, and relative strength to the mixes without CH for the same as FA content and curing times of all mixes

Mixes	Compressive strength/MPa			Relative strength to PC/%			Relative strength to the mixes without CH/%		
	7 days	28 days	60 days	7 days	28 days	60 days	7 days	28 days	60 days
PC	30.0	47.0	56.5	100.00	100.00	100.00	–	–	–
10FA	28.3	45.0	56.0	94.33	95.74	99.12	100.00	100.00	100.00
10FA5CH	30.2	47.5	58.7	100.67	101.06	103.89	106.71	105.56	104.82
10FA10CH	29.3	46.8	58.3	97.67	99.57	103.19	103.53	104.00	104.11
20FA	25.5	41.8	55.1	85.00	88.94	97.52	100.00	100.00	100.00
20FA5CH	28.0	45.4	57.8	93.33	96.60	102.30	109.80	108.61	104.90
20FA10CH	28.5	45.2	57.3	95.00	96.17	101.42	111.76	108.13	103.99
30FA	22.0	36.8	52.5	73.33	78.30	92.92	100.00	100.00	100.00
30FA5CH	23.5	40.5	54.0	78.33	86.17	95.58	106.82	110.05	102.86
30FA10CH	24.8	40.3	53.5	82.67	85.74	94.69	112.73	109.51	101.90
50FA	11.6	24.0	33.2	38.67	51.06	58.76	100.00	100.00	100.00
50FA5CH	13.2	28.0	38.8	44.00	59.57	68.67	113.79	116.67	116.87
50FA10CH	14.4	27.5	36.3	48.00	58.51	64.25	124.14	114.58	109.34

Fig. 1 Compressive strength of Portland–FA cement pastes with CH after curing in water **a** at 28 days and **b** at different ages**Fig. 2** TG curve results of PC paste and FA cement pastes with and without CH at 28 days; sets of **a** 20FA and **b** 50FA

and C_2ASH_8 . The second step which occurred from the dehydroxylation of water from $Ca(OH)_2$ was detected in between ≈ 420 °C and 490 °C, and the third step (≈ 490 –1,000 °C) of mass loss occurred from the decarbonation of calcium carbonate ($CaCO_3$).

As the TG technique cover all phases of ettringite, C-S-H, and C_2ASH_8 , in one single transition step, it was not possible to individually distinguish each phases separately.

The results were then plotted as a derivative thermogravimetric (DTG) to observe each phases, as shown in Fig. 3a and b. The DTG results of Portland–FA cement pastes (sets of 20FA mix and 50FA mix) after 28 days curing in water show detected phases which are ettringite, C-S-H, gehlenite hydrate (C_2ASH_8), $Ca(OH)_2$, and $CaCO_3$ detected at ≈ 76 –83 °C, ≈ 105 –110 °C, ≈ 155 –183 °C, ≈ 448 –475 °C, and ≈ 662 –701 °C, respectively, similar to the

Fig. 3 DTG diagrams of PC paste, Portland–FA cement pastes with and without CH at 28 days; sets of **a** 20FA and **b** 50FA

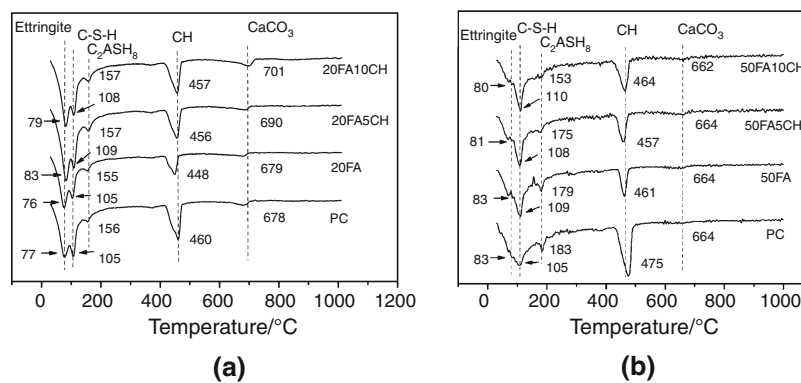


Table 4 Calculated mass loss of the phases of Portland–FA cement pastes

Mix	Mass loss from TGA curves/%			Mass loss to overall mass loss/%		
	Ettringite + C-S-H + C ₂ ASH ₈	CH	CaCO ₃	Ettringite + C-S-H + C ₂ ASH ₈	CH	CaCO ₃
PC-7 days	19.77	4.59	2.30	74.16	17.22	8.63
PC-28 days	21.10	4.80	1.98	75.68	17.22	7.10
PC-60 days	20.25	4.61	1.91	75.64	17.22	7.13
20FA-7 days	20.96	2.83	1.88	81.65	11.02	7.32
20FA-28 days	21.08	2.77	1.94	81.74	10.74	7.52
20FA-60 days	22.05	2.56	1.35	84.94	9.86	5.20
20FA5CH-7 days	20.05	3.47	2.14	78.14	13.52	8.34
20FA5CH-28 days	21.31	3.47	1.89	79.90	13.01	7.09
20FA5CH-60 days	22.63	3.39	1.82	81.29	12.18	6.54

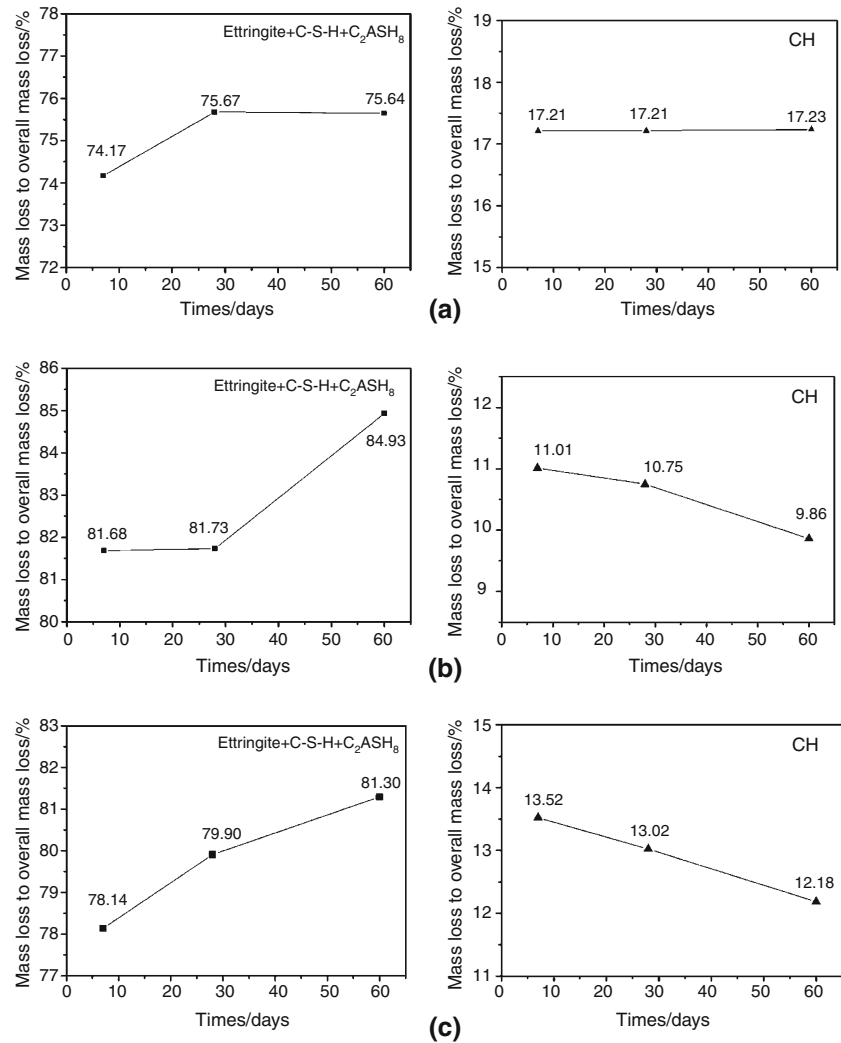
phases reported by Bai et al. [28] and Chaipanich and Nochaiya [21]. Although, the phases detected of the additional 5 and 10 wt% CH mixes were found to be similar to the mix without CH, it is noticed that there is an increase in C-S-H and C₂ASH₈ phases with increasing in CH at 5 and 10 wt%, respectively (compared to the same FA content). This is because of the increase in the pozzolanic reaction with increased CH and thereby FA can react with CH resulting to C-S-H and C₂ASH₈ phases [16]. Moreover, the CH phase was found to increase with increasing CH content in FA mixes, it could be explained due to the fact that the addition of CH might have resulted in quantities of CH greater than that required for the FA particles to react [10].

Moreover, the mass loss from TG curves and calculated mass loss to overall mass loss [21] at different aging times (if taken all mass loss as 100%) of PC (control), 20FA, and 20FA5CH mixes are shown in Table 4 and the mass loss at 28 days are plotted in Fig. 4a–c. The first phase (ettringite + C-S-H + C₂ASH₈ phases) covered the mass loss between room temperature to 420 °C and the second phase (CH phase) between 420 and 480 °C. The hydration products of all mixes can be represented by the mass loss detected; there was an increase in the first phases and a decrease in the second phase with increasing curing time

(Table 4). For PC mix, the increment of hydration products (first phase) from 7 days to 28 days, the mass loss can be observed to be 74.16 and 75.68%, although at 60 days the mass loss was found to be similar to the loss at 28 days. Therefore, from these calculations, it is seen that most of the hydration reaction of PC took place during the early ages (before 28 days). The second phase (CH phase) of PC mix can be seen to have similar mass loss at all different curing time. Moreover, it can be noticed that the second phase of PC mix were higher than that of FA mixes both with and without CH. This is because the pozzolanic reaction between FA and CH would attribute to lower CH phase.

For the case of 20FA mix, the lower loss detected in hydration products at early ages is shown in Fig. 4b. It was found that the mass loss at 7 and 28 days (81.65 and 81.74%, respectively) was observed to be slightly increased and a greater increase was detected at 60 days (84.93%) which is different to that of the control mix. This can be explained by the pozzolanic reaction of FA was found to react with CH phase especially after 28 days curing [12] so that the mass loss of CH can be seen to reduce with increasing in curing time (11.02, 10.74, and 9.86% for 7, 28, and 60 days, respectively). Furthermore, it is noticed

Fig. 4 Mass loss to overall mass loss at 28 days of ettringite + C-S-H + C_2ASH_8 (phase 1) and CH (phase 2) of **a** PC mix, **b** 20FA mix, and **c** 20FA5CH mix



that the first phase of 20FA5CH mix (Fig. 4c) was found to be relatively high and higher mass loss was found with increasing in curing time (78.14% for 7 days, 79.90% for 28 days, and up to 81.29% for 60 days) which is indirectly proportional to the reduction in the CH phase, where the mass loss of CH was detected at 13.52, 13.01, and 12.18% for 7, 28, and 60 days, respectively. Therefore, it is noticed that, although, the total first phase mass loss of these two mixes are similar, but the increment in the mass loss of first phase from 7 to 28 days of 20FA5CH mix was higher than the 20FA mix. This is attributed to the additional CH which can react with FA to give better pozzolanic reaction and was found to occur before 28 days, thereby led to increase in compressive strength at the early ages.

The effect of curing time on mass loss of 20FA5CH mix is reported using the TG curves as shown in Fig. 5. Three similar ranges were detected at the temperature range as above for these phases. The TG results at different time of 20FA5CH mix was evidently found to show an increase in all mass loss with increasing curing time

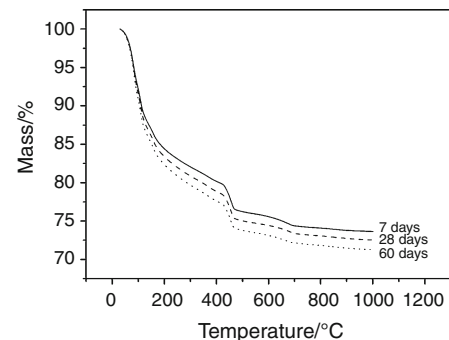


Fig. 5 TG curve results of 20FA5CH mix compared at 7, 28, and 60 days

(60 > 28 > 7 days). This is a consequent of an increase in hydration products with time. A direct reduction of CH phase with time can also be observed. Furthermore, comparisons of DTG and DTA curves of PC-FA-CH paste when replaced PC with FA at 20 wt% and using added CH at 5 wt% plotted at different times are shown in Fig. 6a and

Fig. 6 a DTG and b DTA diagrams of 20FA5CH mix at 7, 28, and 60 days

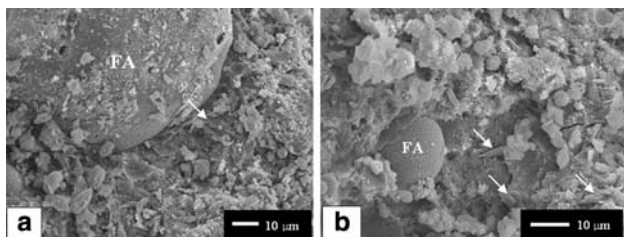
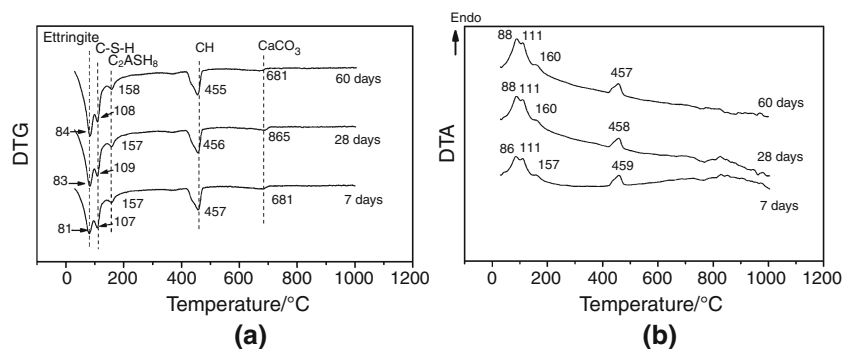


Fig. 7 SEM image of blend cement paste after 28 days cured at magnitude. **a** $\times 1000$ and **b** $\times 1500$

b, respectively. DTA curves show endothermic peaks, as ettringite, C-S-H, C_2ASH_8 , and $Ca(OH)_2$ phases in the temperature range of $\approx 86\text{--}88$ °C, ≈ 111 °C, $\approx 157\text{--}160$ °C, and $\approx 457\text{--}459$ °C, respectively, similar to those reported by Giergiczny [20] and Ray et al. [29]. Although, the CH detected was found to be similar at all aging time, it is noticed that there is an increase in the ettringite, C-S-H, and C_2ASH_8 phases when there is an increase in curing time. This enhancement in ettringite, C-S-H, and C_2ASH_8 phase would therefore agree with the compressive strength results where the compressive strength of these mixes also increased with curing time.

The microstructure of blended PC-FA-CH pastes at 28 days was analyzed using SEM as shown in Fig. 7a and b. It was found that some parts of FA was shown to partly react with CH to form of C-S-H and that remaining CH (shown by white arrows) can be observed close to the FA particle. Generally, the microstructure at the area chosen was seen to be fairly dense due to pozzolanic reaction between CH and FA that formed C-S-H phase which filled the voids between particles of FA and cement grain or between cement grains [30].

Conclusions

The following conclusions are drawn from results of this investigation showing the effect of additional CH on Portland-FA cement pastes properties. Water requirement for normal consistency was found to increase and time of setting

was found to decrease when CH increases. Moreover, the compressive strength was found to increase when CH was added and optimum strength was obtained when CH was used at 5 wt%. Thermal analyses by TG, DTG, and DTA all confirmed better pozzolanic reaction at 28 days where hydration phases attributed to ettringite, C-S-H, C_2ASH_8 was found to increase with increasing CH content, and also with increasing curing time. A fairly dense matrix of PC-FA-CH system was observed under SEM. Therefore, from this study, it appears that the additional of 5 wt% CH can be used with Portland-FA mixes especially at high-volume FA mixes to accelerate FA pozzolanic reaction at early ages (7 and 28 days).

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