



Chemical activation of calcium aluminate cement composites cured at elevated temperature

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ARTICLE INFO

Article history:

Received 23 March 2011

Received in revised form 28 April 2012

Accepted 4 August 2012

Available online 13 August 2012

Keywords:

Calcium aluminate cement

Strätlingite

Physico-mechanical properties

Microstructure

Fly ash

Silica fume

ABSTRACT

The influence of sodium sulfate, as an activator, on the hydration of calcium aluminate cement (CAC)–fly ash (FA)–silica fume (SF) composites was investigated. Different mixes of CAC with 20% pozzolans (20% FA, 20% SF and 10% FA + 10% SF) were prepared and hydrated at 38 °C for up to 28 days. The hydration products were investigated by XRD, DSC and SEM. The results showed that sodium sulfate accelerated the hydration reactions of calcium aluminate cement as well as the reactions of FA and SF with CAH_{10} and C_2AH_8 to form the strätlingite (C_2ASH_8). The later reactions prevent the strength loss by preventing the conversion of CAH_{10} and C_2AH_8 to the cubic C_3AH_6 phase. The acceleration effect of Na_2SO_4 on the reactivity of fly ash was more pronounced than on the reactivity of silica fume with respect to reaction with CAH_{10} and C_2AH_8 phases.

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1. Introduction

Calcium aluminate cement (CAC) has many industrial applications due to its advanced properties, such as rapid strength development, high temperature resistance and resistance to a wide range of chemically aggressive conditions. It is used in refractories, pipes, sewer constructions, industrial floors and dam spillways [1,2].

High early strength is one of the major advantages of calcium aluminate cement over Portland cement (PC). However, the high early strength may mislead designers to ignore the conversion reaction of calcium aluminate cement which causes strength loss in hot and humid conditions. The hydration products of CAC depend on the curing temperature [1–5]. CAH_{10} is the main hydration products at temperatures less than 20 °C. However, C_2AH_8 and AH_3 are the main hydration products above 20 °C. The high early strength of CAC cement is attributed to the formation of hexagonal hydrates (CAH_{10} and C_2AH_8) as the dominant hydration products [3]. However, CAH_{10} and C_2AH_8 are known to be metastable and convert to the more stable C_3AH_6 and AH_3 [3,6,7], according to the following equations [3]:



The conversion reactions are accelerated at high temperatures and moisture contents [8]. The conversion reactions are associated with the strength loss and the formation of microcracks in the concrete structure [8–10].

Midgley [11] stated that traces strätlingite (C_2ASH_8) were detected in aged hydrated high alumina cement. Midgley [11] suggested that strätlingite compound has better mechanical properties than hydrogarnet (C_3AH_6). Bentsen et al. [12] reported that microsilica enhances the formation of strätlingite. He also stated that the strätlingite compound crystallizes as a stable phase in the temperature range of 20–70 °C.

Mixing pozzolan with CAC was found to prevent the conversion reaction [13–18]. Majumdar and Singh [13] found that slag addition to CAC prevented the strength loss for the pastes cured under water at 40 °C. The ability of slag to prevent the conversion reactions was correlated with its hydraulic activity [19,20], which depended on the glass contents and the dissolution ability in basic solutions. Silica fume and fly ash are high reactive pozzolans [21]. Silica fume reacts in few minutes and gave large amount of silicate ions [21] in solution. ASTM C 373–88 [23] found the presence of sodium ions accelerates dissolution of silica required for strätlingite formation.

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In the present work, the possibility of using two types of pozzolanic materials (SF & FA) with calcium aluminate cement (CAC) to prepare blended cements mixes suitable for hot climate was investigated. The effect of sodium sulfate as an activator on the hydration characteristics of these blends was also investigated at elevated temperature.

2. Materials and methods

2.1. Materials

Silica fume (SF) was obtained from Ferrosilicon Co. Edfo, Egypt. Fly ash (FA) was supplied by ASH RESOURCES (PTY) LTD (Dura Pozz), India. Calcium aluminate cement (CAC) was obtained from Suez Cement Company, Suez, Egypt. The chemical compositions of the raw materials are given in Table 1.

2.2. Preparation of cement pastes

Each of the two types of pozzolans was replaced 20% of CAC to produce blended cements. In addition 10% of each pozzolan was used together to replace CAC. These mixes were designated as shown in Table 2. The water of normal consistency was determined in accordance with B.S. 4550. The water required for standard consistency is presented in Table 2. The required amount of water (with and without Na_2SO_4) was mechanically mixed with the cement blends for 3 min. The pastes were then poured and compacted into 1-in. cube moulds and their surfaces were smoothened. The moulds were placed in a humidity cabinet having about 100% RH at $23 \pm 2^\circ\text{C}$ for 24 h. After demoulding, the cubes were placed under water at 38°C until testing (1, 3, 7 and 28 days).

2.3. Methods of investigation

The compressive strength measurements were conducted on three samples of each paste at a given curing period. Apparent

porosity was determined using the standard liquid volume method adopted by the ASTM C 373-88 [23]. X-ray diffraction (XRD) analysis was performed using an automated diffractometer (Philips type: PW1840), at a step size of 0.02° , scanning rate of 2° in $2\theta/\text{min.}$, and a 2θ range from 4° to 80° . Differential scanning calorimetry runs were conducted using a Shimadzu DSC-50 thermal analyzer at a heating rate of $10^\circ\text{C}/\text{min.}$ The fracture surfaces hydrated pastes were investigated by using SEM (JOEL, Model: JSM-5600, Japan.) equipped with secondary electron detector and EDX. All samples were coated with gold.

3. Results and discussion

3.1. Physico-mechanical properties

3.1.1. Apparent porosity

Fig. 1, represents the apparent porosities of the hydrated pastes cured for 28 days at 38°C for both the neat calcium aluminate cement (shown as separate point in the graph) and the blended mixes with different amounts of Na_2SO_4 . The porosities of mixes Ma, Mb and Mc are lower than this of the neat calcium aluminate cement (Mo). Due to the relatively higher surface area of the silica fume more than the fly ash, mixes which contain silica fume (Mb and Mc) show lower porosities than those which contain only fly ash (Ma). The same increase of porosity was observed in Portland cement-SF system [24]. It is found that addition of Na_2SO_4 has a remarkable effect on the porosity. The porosity decreases with increasing Na_2SO_4 contents for each mix.

3.1.2. Compressive strength

Figs. 2 and 3 represent the results of compressive strength as a function of curing times of mixes (Mo, Ma, Mb and Mc) prepared with 2% and 3.5% wt. Na_2SO_4 respectively. The compressive strength of the neat CAC paste (Mo) increased with time up to 3 days then decreased up to 28 days. The high early compressive strength is a characteristic property of CAC cements, especially at low W/C ratio and high temperature [8,25]. The compressive strength of blended CAC mixes (Ma, Mb and Mc) with 2% and 3.5% Na_2SO_4 showed continues increase with curing time. In general, the strength values of pastes containing 3.5% Na_2SO_4 are higher than those containing 2% Na_2SO_4 .

Fig. 4 represents the compressive strength of pastes hydrated for 28 days. It clearly shows the difference between the

Table 1
Chemical compositions of CAC, FA and SF (mass%).

Oxides	CAC	FA	SF
SiO_2	5.39	62.26	95.57
Al_2O_3	48.75	26.89	0.30
Fe_2O_3	1.44	4.19	1.35
CaO	39.12	0.76	0.19
MgO	0.21	0.53	0.34
SO_3	0.15	0.31	0.11
K_2O	0.10	1.12	0.51
Na_2O	0.14	0.07	0.19
Cl	–	0.04	–
TiO_2	2.52	1.77	0.02
MnO_2	0.17	0.09	–
P_2O_5	–	0.37	0.03
L.O.I	0.53	1.42	1.10
Surface area (cm^2/g)	3650	3850	$\approx 20,000$

Table 2
Mix compositions of investigated specimens.

Mix	Compositions (mass%)			
	CAC	FA	SF	W/C ^a
Mo	100	–	–	0.25
Ma	80	20	–	0.28
Mb	80	10	10	0.295
Mc	80	–	20	0.31

^a Water/cement ratio required for standard consistency.

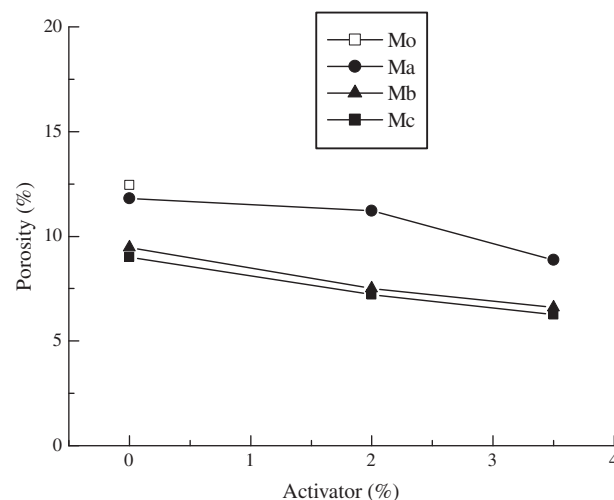


Fig. 1. Apparent porosity of hardened pastes with different amount of activator cured at 38°C for 28 days (sample Mo without activator).

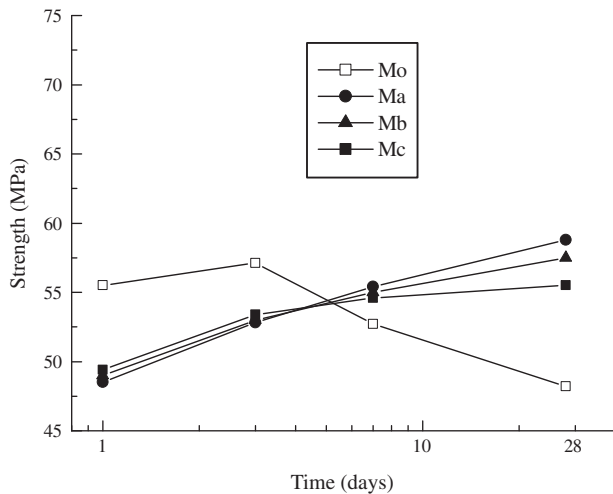


Fig. 2. Compressive strength of pastes cured up to 28 days at 38 °C (neat CAC; Mo without activator in comparison with Ma, Mb and Mc prepared with 2% activator).

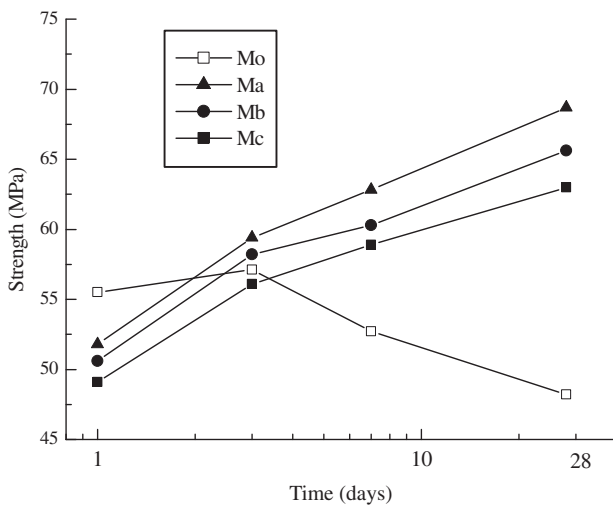


Fig. 3. Compressive strength of pastes cured up to 28 days at 38 °C (neat CAC; Mo without activator in comparison with Ma, Mb and Mc prepared with 3.5% activator).

compressive strength values of neat CAC (Mo) and those of the blended CAC mixes (Ma, Mb and Mc) prepared with and without sodium sulfate as activator.

3.2. Mineralogical composition

3.2.1. XRD analysis

XRD was carried out to investigate the hydrated phases formed in each mix cured at 38 °C for 28 days. The XRD patterns of Ma, Mb and Mc pastes in comparison with that of the neat CAC (Mo) paste, are represented in Figs. 5–7, respectively. In general, the intensities of characteristic peaks of hydrogarnet (C_3AH_6) and AH_3 diminish for the blended mixes, especially those with Na_2SO_4 . This indicates that the presence of both FA and SF prevent the conversion process of the hexagonal calcium aluminate phases to the stable cubic hydrogarnet (C_3AH_6) phase. The characteristic peaks of the main hydrated CAC phase (CAH_{10}) are slightly enhanced in the presence of silica fume and fly ash as fine siliceous materials (Ma, Mb and Mc). Many investigators [3,22,26,27] found that, fine pozzolonic materials provided nucleation sites which enhance the hydration reaction of cements.

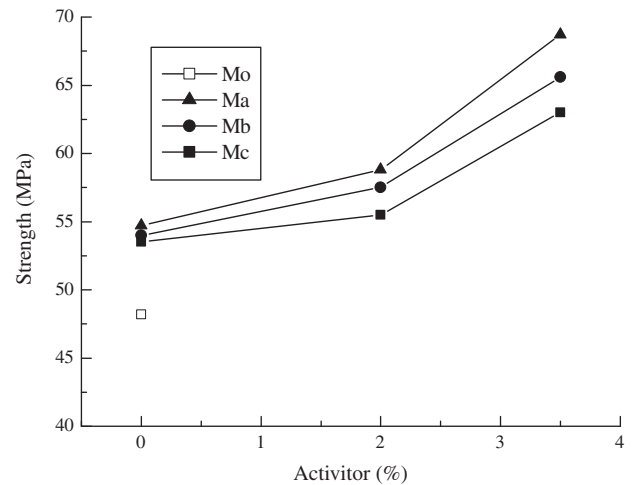


Fig. 4. Compressive strength of hardened pastes with different amount of activator cured at 38 °C for 28 days (sample Mo without activator).

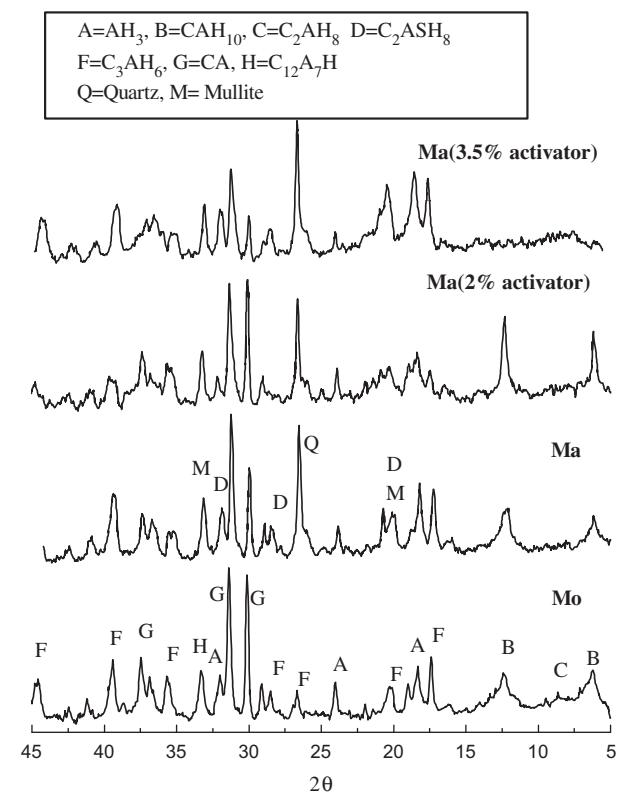


Fig. 5. X-ray diffraction patterns of Mo pastes (without activator) and Ma pastes prepared with different amounts of activator and cured for 28 days at 38 °C.

The XRD diffraction patterns of hydrated pastes with 2% Na_2SO_4 show an increase in the intensity of the characteristic peaks of CAH_{10} . Addition of 3.5% Na_2SO_4 results in disappearance of CAH_{10} characteristic peaks in mixes containing fly ash (Ma and Mb). However, the intensities of these peaks only decrease in mix containing silica fume (Mc) without FA. This is associated with a slight increase in the strätlingite (C_2ASH_8) peaks and a more diminishing in the hydrogarnet (C_3AH_6) and AH_3 peaks.

3.2.2. Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) was used to investigate the type of hydration products in calcium aluminate cement

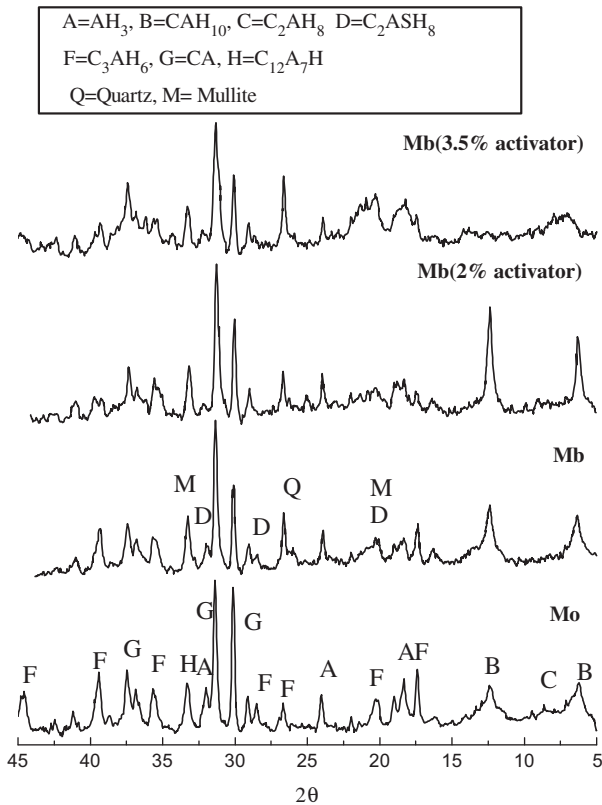


Fig. 6. X-ray diffraction patterns of Mo pastes (without activator) and Mb pastes prepared with different amounts of activator and cured for 28 days at 38 °C.

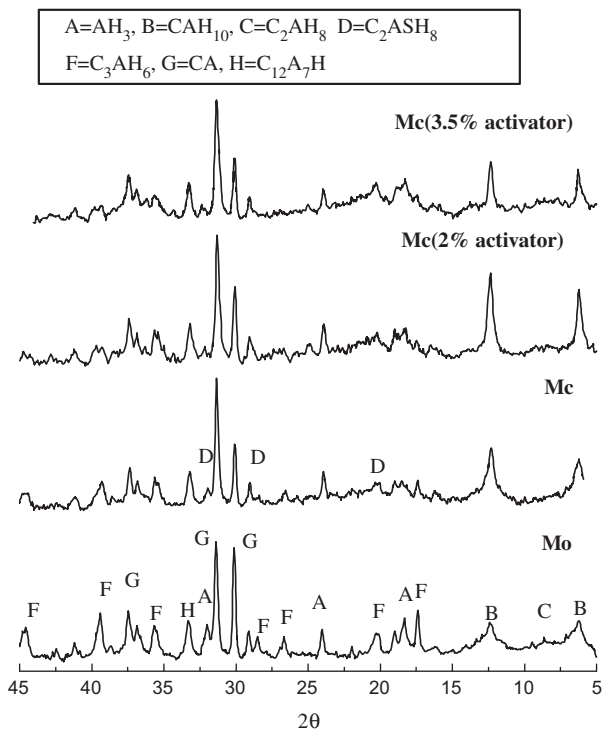


Fig. 7. X-ray diffraction patterns of Mo pastes (without activator) and Mc pastes prepared with different amounts of activator and cured for 28 days at 38 °C.

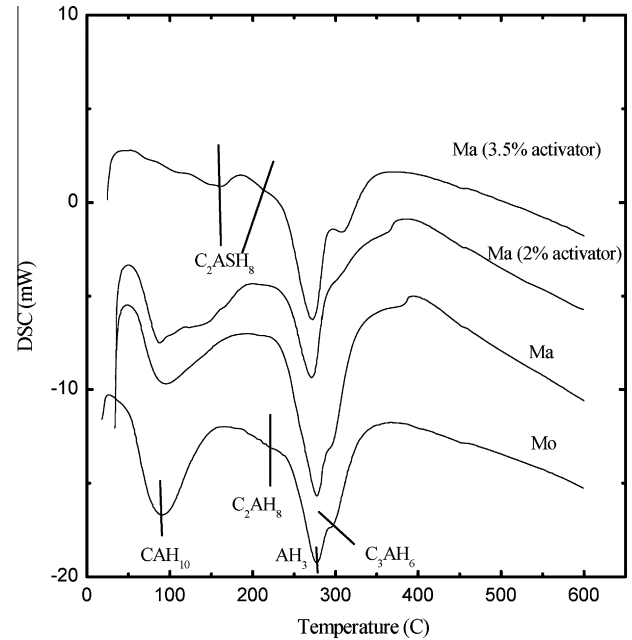


Fig. 8. DSC thermograms of Mo pastes (without activator) and Ma pastes prepared with different amounts of activator and cured for 28 days at 38 °C.

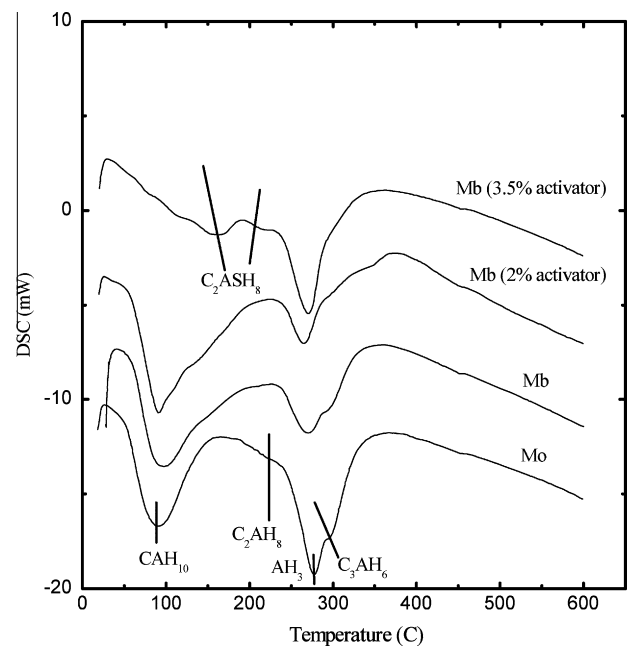
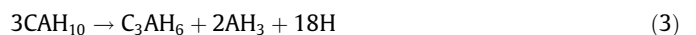


Fig. 9. DSC thermograms of Mo pastes (without activator) and Mb pastes prepared with different amounts of activator and cured for 28 days at 38 °C.

thermogram of the neat CAC (Mo) paste shows an ideal thermogram for hydrated aluminate cement [28]. It displays a group of endothermic signals characteristics of different calcium aluminate hydrates. The observed endothermic signals were: CAH₁₀ (60–130 °C); C₂AH₈ (215–230 °C); AH₃ (250–290 °C); C₃AH₆ (290–350 °C). The signal below 130 °C was assigned to dehydration of the hexagonal hydrated phase, CAH₁₀. The dehydration of C₂AH₈ occurs at 227 °C. Generally, CAH₁₀ and C₂AH₈ are unstable and transform to C₃AH₆ and AH₃ according to the following equations [28]:



composites. DSC thermograms of the hydrated pastes (Mo, Ma, Mb and Mc) cured at 38 °C for 28 days are shown in Figs. 8–10. The

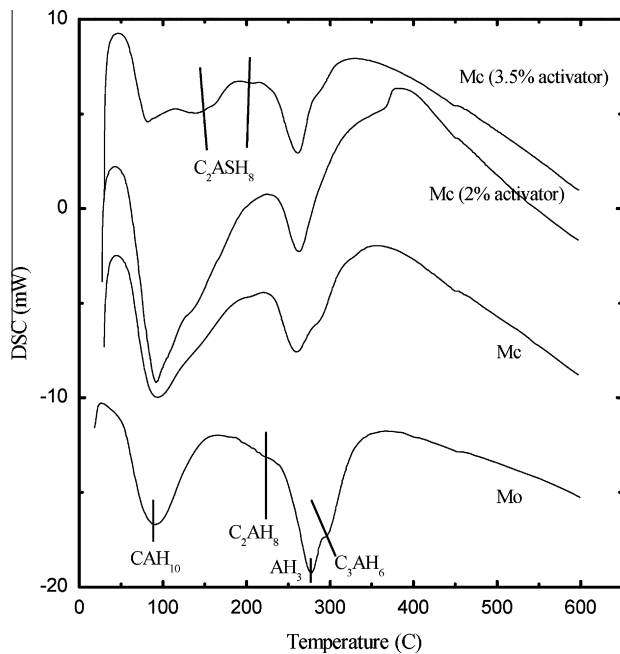
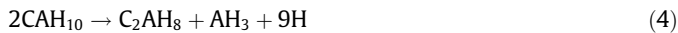
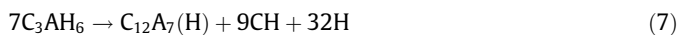
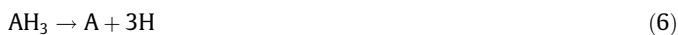


Fig. 10. DSC thermograms of Mo paste (without activator) and Mc pastes prepared with different amounts of activator and cured for 28 days at 38 °C.



The two distinctive endothermal peaks exist between 250 and 330 °C are associated with the decomposition of AH_3 and C_3AH_6 respectively [29,30]. This could be explained by the decomposition of AH_3 and C_3AH_6 produced during cement hydration as well as those formed during the previous decomposition reactions of CAH_{10} and C_2AH_8 [31]. Ukrainczyk et al. [32] found that, C_3AH_6 losses 29.6% of its mass, in two stages; first 22.0% and then 7.2%.



It is interesting to remark that the AH_3 dehydroxylation signals for different mixes appear at slightly different temperatures. This can be explained by the presence of three different AH_3 polymorphs, namely, bayerite at 266°, nordstrandite at 270° and gibbsite at 278 °C, respectively [33].

The broad peak characteristic for CAH_{10} detected in the thermograms of the neat CAC decreases with the presence of both FA and SF. This peak disappears in case of pastes containing FA (Ma and Mb) with 3.5% sodium sulfate, which confirms the results of XRD. However, CAH_{10} peak stills detected in the corresponding mix with only SF (Fig. 10). Peaks characteristic for AH_3 and C_3AH_6 also decrease with the presence of both FA and SF and/or the increase of the amount of activator.

Figs. 8–10, show small broad shoulders at about 164 and 210 °C characteristic for the strätlingite (C_2ASH_8) [34] in mixes containing pozzolans (SF & FA). These peaks increase with activator additions. Kuzel [34] and Matschei et al. [35] found that, strätlingite showed three endothermic peaks; a minor peak at ~120 °C and main peaks at ~165 °C and 220 °C.



3.2.3. Scanning electron microscopy (SEM)

The fracture surfaces of cement pastes hydrated for 28 days were studied using SEM/EDX. The microstructure hydrated neat

CAC pastes prepared without activator and those of Ma prepared without activator and with 3.5% activator are shown in Fig. 11.

The micrograph of neat CAC shown in Fig. 11A indicates the presence of well crystalline cubic and granular crystals of C_3AH_6 as well as AH_3 gel. The micrograph of Ma (without activator) shown in Fig. 11B indicates the presence of the thin flaky-plate like morphology of the strätlingite hydrate (C_2ASH_8) with little amounts of amorphous AH_3 gel. The spherically shaped particles of FA could be also detected. Fig. 11C of Ma (with 3.5% activator), shows a massive structure of well crystalline strätlingite (C_2ASH_8) as sheets of thin flaky structure like which are stacked as parallel layers; which reflects a relatively more closed compact texture. The spherically shaped particles of FA could not be detected. The microstructure showed the formation of dense masses of hydration products having micro and narrow pores. The plates of C_2ASH_8 are deposited within the pore system, which can accommodate larger amounts of hydration products leading to relatively

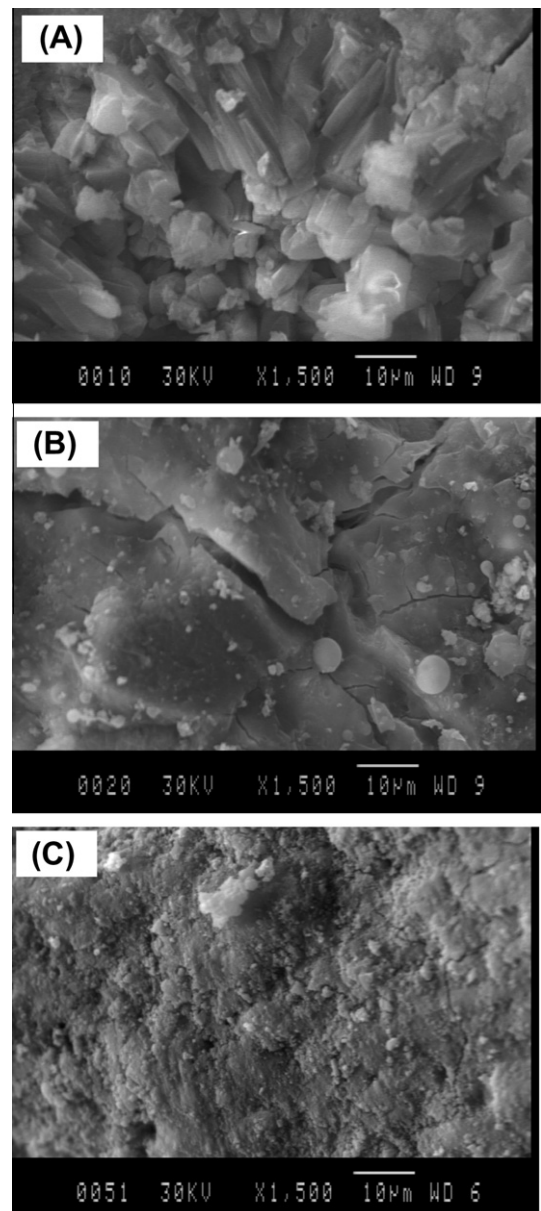


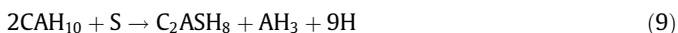
Fig. 11. SEM micrographs of pastes hydrated for 28 days at 38 °C, (A) Mo (neat CAC, without activator), (B) Ma (without activator) and (C) Ma (with 3.5% activator).

higher compressive strength values of these CAC/FA pastes as compared with those of the neat CAC paste (Mo).

4. Discussion

Calcium aluminate cement reacts with water to form two main hydration products namely CAH_{10} and C_2AH_8 as well as a little amount of AH_3 . CAH_{10} and C_2AH_8 compounds are metastable, which over long periods of time or at elevated temperatures transform to the hydrogarnet phase (C_3AH_6). This reaction is known as the conversion reaction. The hardened neat CAC paste cured at 38 °C shows the highest porosity. This due to the conversion reactions of the two main hydrated hexagonal phases CAH_{10} and C_2AH_8 to the cubic hydrogarnet phase (C_3AH_6) [5,36]. Ding et al. [37] explained the increase in porosity due to the differences in relative volumes of the phases. The conversion of CAH_{10} to C_3AH_6 results in a volume decreases to about 50% whereas that of C_2AH_8 to the cubic phase results in a decrease of about 65% of the original volume of the reactants [37]. Thus, the major impact of the conversion process is the increase in porosity and the decrease in mechanical strength.

In a composite system containing calcium aluminate cement (CAC) and pozzolanic materials (FA & SF), the main hydrated phases (CAH_{10} & C_2AH_8) can incorporate silica to form strätlingite compound (C_2ASH_8) in accordance to the following equations [37]:



This inhibits the conversion reactions. Addition of Na_2SO_4 as an activator to CAC–FA and CAC–SF composite cements increases the effectiveness of both FA and SF in inhibiting the conversion reactions. This explains the decrease in porosity and the increase in strength of the cement pastes containing silica fume and/or fly ash and sodium sulfate as an activator. Mixes containing fly ash (Ma & Mb) show lower porosity values than those of mixes only containing SF. This proved that the fly ash is more reactive than silica fume with respect to reaction with CAH_{10} & C_2AH_8 to form strätlingite. The rate of C_2ASH_8 formation is controlled by the rate of silicate ions dissolution from siliceous materials which can be increased using chemical activators such as Na_2SO_4 used in the present study. Na_2SO_4 has a great influence on the reaction of both FA and SF with the two main hydrated phases of CAC pastes (CAH_{10} and C_2AH_8) to form the more stable strätlingite (C_2ASH_8) phase. Mixes containing FA have the highest strength values which indicate that the FA is more reactive to react with the hydrated CAC phases in the presence of sodium sulfate.

5. Conclusion

Hydration of CAC in hot and humid conditions results in a reduction in compressive strength due to the conversion reaction, in which the CAH_{10} and C_2AH_8 compounds are converted into the more stable cubic C_3AH_6 compound. The conversion reactions were usually associated with increase in porosity and strength loss in neat CAC paste. The addition of FA and SF to the CAC inhibits these conversion reactions. Addition of Na_2SO_4 as an activator increases the inhibition efficiency of both FA and SF on the conversion reactions. The use of sodium sulfate accelerated the hydration of CAC as well as the reaction of FA and SF with CAH_{10} and C_2AH_8 phases. Sodium sulfate accelerates the dissolution of silica required for the formation of strätlingite (C_2ASH_8). Investigation of the results of mechanical properties, XRD and DSC indicated that the FA was more reactive to form the strätlingite compound than SF especially with 3.5% Na_2SO_4 as activator.

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