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Effects of curing temperature and NaOH addition on hydration and strength development of clinker-free CKD-fly ash binders

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Abstract

Effects of curing temperature and NaOH addition on hydration and strength development of cement kiln dust (CKD)-fly ash (FA) binders were investigated. Pastes made with 50% CKD and 50% FA, having 0, 2, and 5% NaOH addition, and cured at temperatures of 24, 38, and 50 °C were evaluated. The hydration products of the binders were examined by thermogravimetric analysis (TGA) and X-ray diffraction (XRD) tests. The results indicate that the major crystalline hydration product of the CKD-FA binders is ettringite, and the ettringite is stable in the CKD-FA system at age over 100 days. Curing at elevated temperature is more effective for CKD-FA binder strength improvement than NaOH addition, the later often depressing ettringite formation in a CKD-FA system. At a proper curing temperature (38 °C), addition of a small amount of NaOH (2%) may increase CKD-FA binder strength; while at a high curing temperature (50 °C), addition of NaOH (2%) may reduce the binder strength.

Keywords: Cement kiln dust; Fly ash; Elevated curing temperature; NaOH addition; Thermal analysis; X-ray diffraction, XRD

1. Introduction

Cement kiln dust (CKD) contains partially calcined materials with some hydraulic and cementitious properties. Due to the presence of certain chemical constituents (high alkali, chloride, and sulfate content), which may cause problems in kiln operation and cement performance, CKD usually cannot be returned into the feedstock and has to be discarded.

Fly ash (FA) is a waste material produced from combustion of coal in thermal power plants. It is mainly composed of vitrified (amorphous) alumina silicate melt in addition to a small amount of crystalline minerals, such as quartz, mullite, mica, etc. Due to low chemical reactivity caused by the high degree of polymerization at which tetrahedral silicate is bridged with oxygen, most FAs, especially Class F FA, react with water very slowly at a room temperature. As a result, chemical and hydrothermal activations are often used to accelerate hydration of FA in cements (pozzolanic reaction). High-alkaline liquid phase produced by cement hydration is a known activating agent. However, even under the conditions of alkaline activation, FA hydrates relatively slowly [1].

Because of the deficiencies of these two materials, the usage of individual CKD and FA as a supplementary cementitious material (SCM) in concrete is limited. Some research has indicated that if the two materials are appropriately blended, the alkalis from CKD may activate hydration of FA and the blends may create a cementitious material in which the waste material deficiencies will be converted into benefits. Bhatty [2–4] studied binary, ternary, and quaternary mixes using ordinary Portland cement (OPC), five different CKDs, two different types of FA (Class F and C), and slag. He observed that cements containing CKD alone had reduced strength, setting time, and workability. The addition of FA into a CKD-OPC system lowered the alkali content and resulted in improved strength. Dyer et al. [5] examined ternary blends contain-

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ing two types of CKD, pulverized fuel ash (PFA), and OPC, and he found that CKD accelerated the binder hydration.

The present research is aimed at developing a clinker-free cementitious material utilizing CKD and Class F FA. It also involves optimizing the CKD-FA binder performance by varying mix proportions and activation methods, such as cogrinding processing, elevated temperature curing, and using activating admixtures. As part of the research, effects of elevated curing temperature and NaOH addition on hydration and strength development of the 50% CKD-50% FA binders have been investigated and the results are presented in this paper.

2. Experimental work

2.1. Raw materials

Chemical compositions of CKD and FA used are presented in Table 1, and they are compared with OPC (Type I cement). Particle size analyses of the materials indicated that the median particle sizes for CKD, OPC, and FA are 8, 15, and 30 μm. The CKD is finer while FA is coarser than OPC. Fig. 1 demonstrates the XRD analyses of the CKD (a) and FA (b). The major crystal phases identified in CKD include calcium oxide (CaO), calcite ($CaCO_3$), anhydrite ($CaSO_4$), and quartz (SiO_2). FA is mostly vitreous, and the presence of glassy phase is illustrated by a "hump" in the range of 15–35°. The crystal minerals identified in FA include mullite ($Al_6Si_2O_{13}$), hematite (Fe_2O_3), and SiO_2, which are fairly typical for low-Ca (Class F) ashes.

The raw CKD and FA materials were also examined under scanning electron microscope (SEM). The SEM study indicates that CKD has irregular particles mixed with many very fine spheres. These fine spheres may be formed from the materials that were entrained into a high temperature gas stream and then cooled down abruptly. In addition, a number of clusters were observed in CKD. These clusters may be due to the surface attraction of the ultrafine particles that cause agglomeration. The agglomeration phenomenon may considerably influence the water requirement and workability as well as the rate of strength development of a binder. Most FA particles occur as solid spheres of glass. The spherical shape of FA particles can improve not only the flowability of concrete but also the uniform dispersion of cementitious materials in concrete [6]. The glassy shell of an FA is the most reactive [7] and relatively easily initiates the pozzolanic reaction; whereas the crystalline phases remain inert for periods up to 1 year [8].

Table 1
Chemical composition of cementitious materials

Compound (percentage by weight)	OPC (Type I)	CKD	FA (Class F)
SiO_2	20.35	11.5	46.80
Al_2O_3	5.24	4.38	23.89
Fe_2O_3	3.58	2.04	15.77
CaO	64.29	56.0	4.74
MgO	1.13	1.34	0.91
SO_3	2.56	16.7	1.18
K_2O	0.60	5.86	1.56
Na_2O	0.11	1.02	0.79
$(Na_2O)_{eq}^{a}$	0.50	4.80	1.80
P_2O_5	0.114	0.085	0.430
Cl^-	–	0.73	–
LOI	1.10	6.00	2.36
Specific gravity	3.15	2.79	2.41

[a] $(Na_2O)_{eq} = Na_2O + 0.658\ K_2O$.

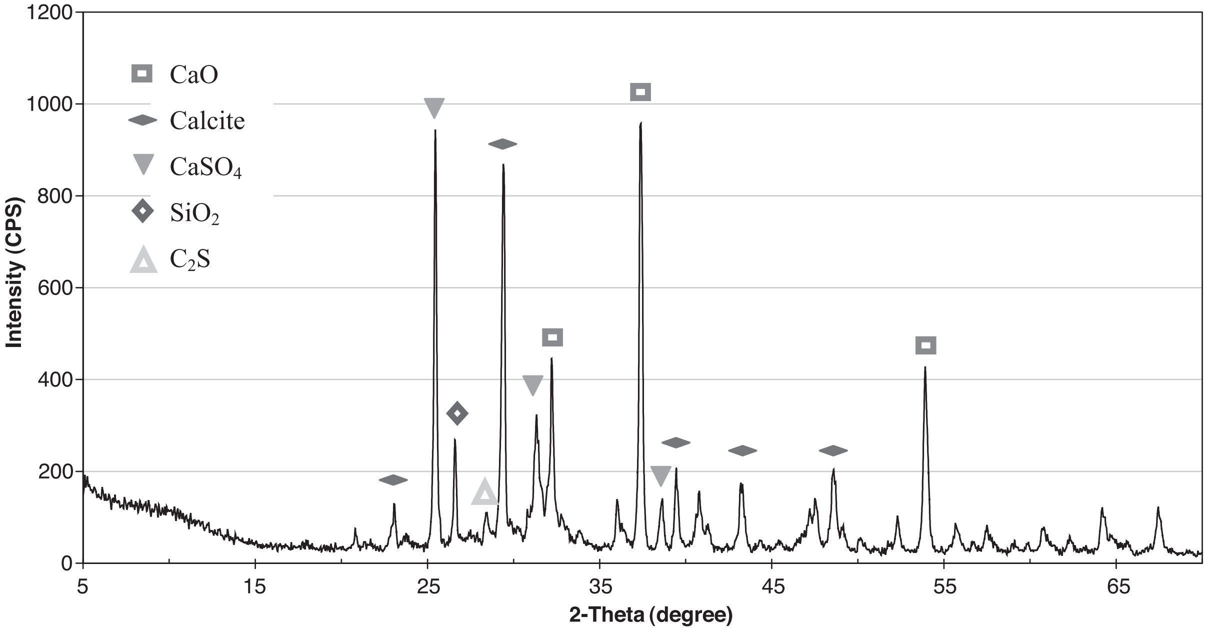
2.2. Specimens

Considering the weak cementitious properties of CKD and a low activity of Class F FA, investigators have employed different methods to activate CKD-FA blend hydration. These activation methods include the following: (1) natural activation—simply blending CKD and FA and expecting alkalis from CKD to activate FA hydration; (2) chemical (alkali) activation—adding 2% or 5% NaOH in the binder system to activate FA hydration; and (3) physical (thermal) activation—curing binder pastes at elevated temperatures 38 and 50 °C (100 and 122 °F).

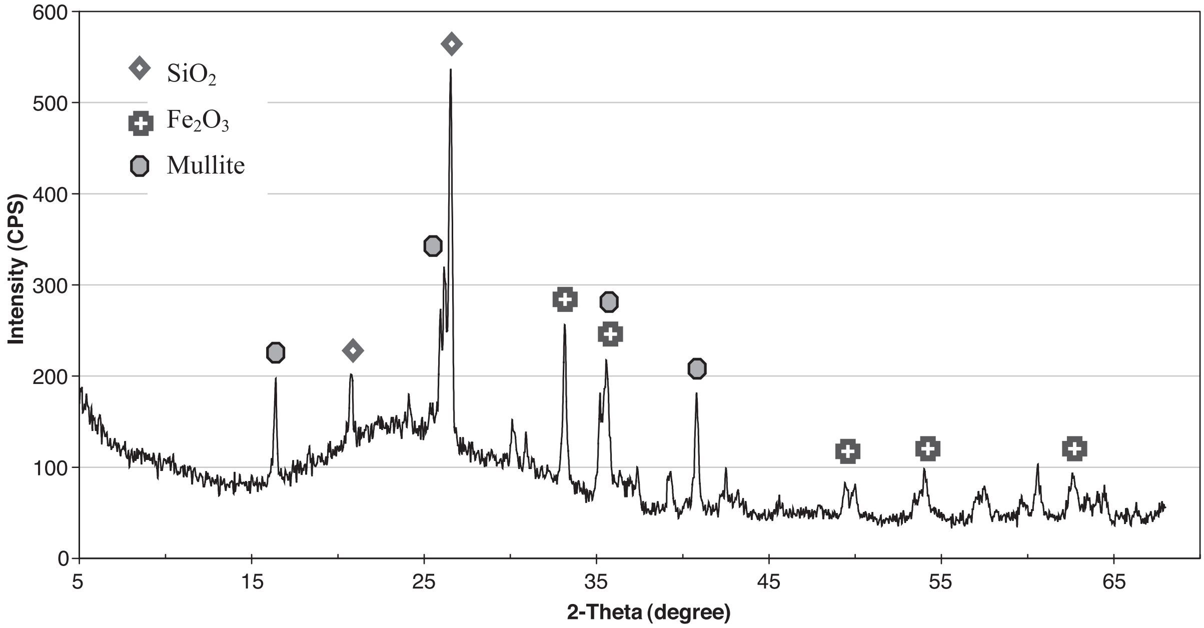
All CKD-FA paste samples were made with a CKD-to-FA ratio of 50:50. A water-to-solid ratio (w/s) of 0.52 was selected for all samples to obtain a desirable flow of the pastes. After casting in 1-in. cubic molds, the samples were cured in sealed plastic bags. The bags were semisubmerged in water in a closed container and stored in fog room at 24 °C (73 °F) or in an oven at 38 °C (100 °F) or 50 °C (122 °F). The test matrix is given in Table 2. A set of paste samples made with OPC, with water-to-cement ratio (w/c) of 0.52 and cured at 24, 38, and 50 °C, was used for reference.

2.3. Tests

Compressive strength of the pastes was determined at different curing ages. Reaction products and degree of hydration of the mixes were studied by X-ray diffraction (XRD) and thermogravimetric analysis (TGA). XRD tests were conducted with a Siemens D500 diffractometer by a powder diffraction method, with Cu Kα, voltage 50 kV and current 27 mA. TGA technique is frequently used in the study of cement hydration and the results often provide insight on features of cement hydration process and hydration products [9,10]. TGA tests were performed with a TA2950 Thermogravimetric Analyzer controlled by an IBM PC. A TA Hi-ResN software package was used to permit rapid testing while retaining high resolution. The tests were done with approximate 55-mg samples in dry



(a) CKD



(b) Fly Ash

Fig. 1. XRD powder pattern of raw materials.

nitrogen (N_2). The gas flow rate is 100 ml/min. The heating rate is 40 °C/min for all samples tested.

3. Results and discussions

3.1. Strength development

Fig. 2 demonstrates the effect of NaOH addition on binder strength development. As observed in Fig. 2a and b, when the curing temperature is 24 and 38 °C, addition of 2% NaOH increases binder strength. The binder made with 50% CKD-50% FA-2% NaOH and cured at 38 °C has the highest strength of all samples, approximate 27 MPa (4000 psi) at the age of 56 days. Addition of 5% NaOH tends to increase binder strength at an early age (before 7 days) but decreases binder strength at a later age. As previous research indicated, excessive NaOH addition may result in undesirable morphology and nonuniformity of hydration products in the pastes, thus reducing binder strength [11]. When the

Table 2
Sample list and descriptions

Mix (CKD/FA = 1:1)	NaOH addition (%)	Curing temperature (°C)
1	0	24
2	2	24
3	5	24
4	0	38
5	2	38
6	5	38
7	0	50
8	2	50

curing temperature is 50 °C (Fig. 2c), addition of even 2% NaOH into the system has caused reduction of the binder strength at both early and later ages. As discussed below, elevated curing temperature also accelerates ion dissolution and binder hydration, resulting in the nonuniform microstructure. Thus, the combination of NaOH addition and a high curing temperature (50 °C) may not be preferred for the CKD-FA binder strength development.

Fig. 3 presents the effect of curing temperature on compressive strength of the CKD-FA pastes. General observation indicates that the effectiveness of activation, in terms of strength improvement, resulting from elevated curing temperature is much higher than that from NaOH addition. When a proper CKD-to-FA ratio and activation method are employed (such as a binder made with 50% CKD-50% FA-2% NaOH and cured at 38 °C), the compressive strength of the CKD-FA binder is comparable with that of OPC.

As observed in Fig. 3, when cured at 24 °C, CKD-FA pastes have 28- and 56-day strength lower than 6.9 MPa (1000 psi) and 13.8 MPa (2000 psi), respectively. While cured at an elevated temperature (38 °C), the strength of CKD-FA pastes is doubled. However, strength of OPC pastes increases only 15% when the curing temperature is increased from 24 to 38 °C. This implies that curing at elevated temperature has more significant contribution to pozzolanic reaction than that to OPC hydration. However, further increase in curing temperature from 38 to 50 °C appears not to improve (sometimes even reduce) the strength of CKD-FA pastes. This may be attributed to the effect of hydration temperature on the microstructure of binder pastes [12,13]. Because of low solubility and diffusibility, cement hydration products rapidly formed at an elevated temperature may not be able to diffuse to a significant distance from the binder grain, thus resulting in a highly nonuniform distribution of the solid phases. The dense hydration products around the cement grains would serve as diffusion barriers, slowing or even remarkably preventing further hydration. As a result, although binder hydration proceeds more rapidly with increasing temperature at early age, the ultimate strength of concrete cured at a high temperature may not increase significantly [14].

3.2. XRD analysis of the hydration products

3.2.1. Hydration process of CKD-FA binders

Fig. 4 shows the XRD patterns of CKD-FA pastes. Note that many compounds identified in hydrated CKD-FA pastes, such as $CaCO_3$ and SiO_2, actually were present in the binder raw materials. The major crystal-

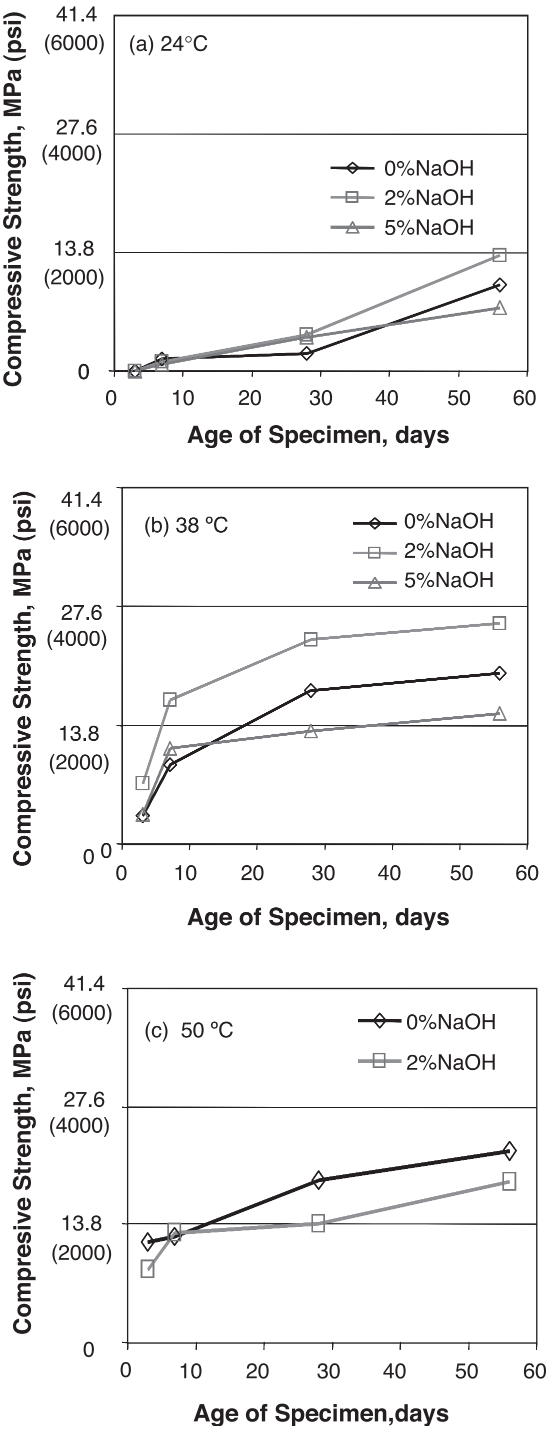


Fig. 2. Effect of NaOH addition on binder strength.

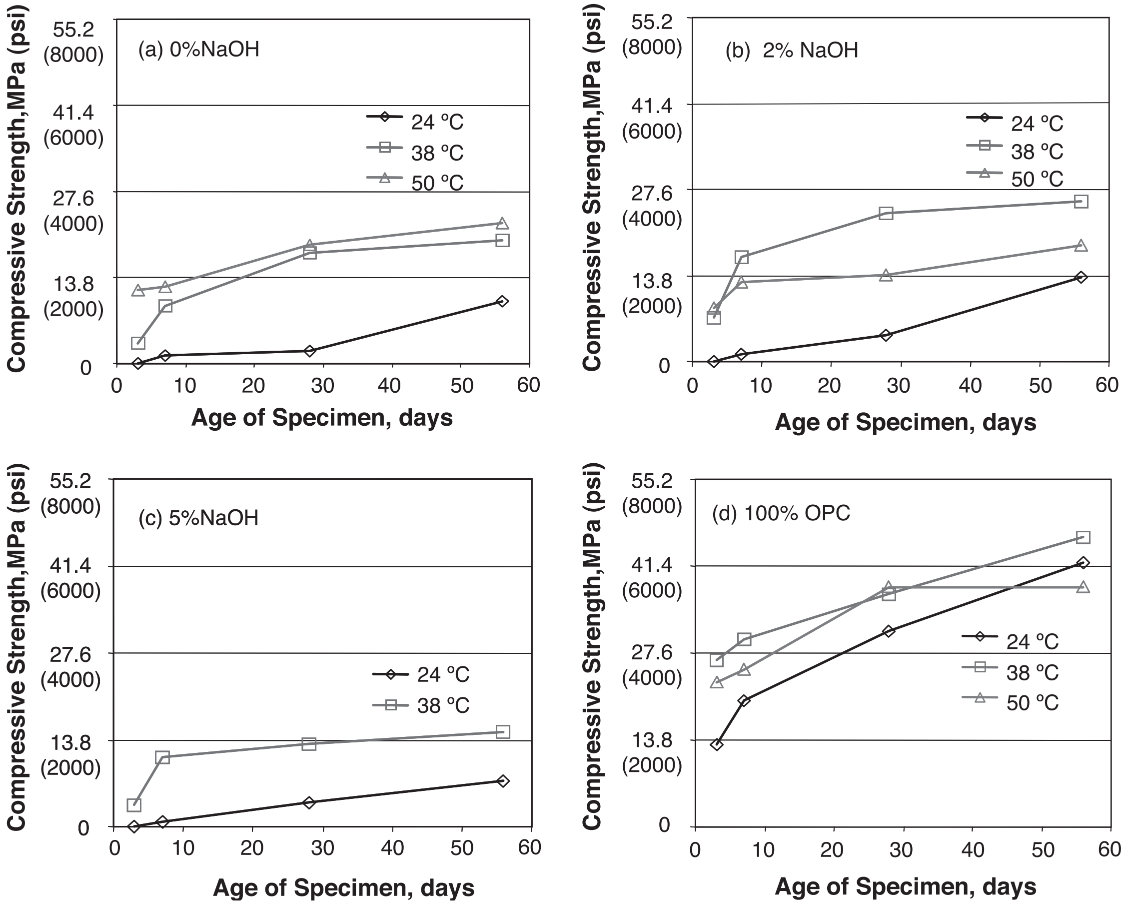


Fig. 3. Effect of curing temperature on binder strength.

line hydration product of the CKD-FA binders is ettringite.

The precipitation of ettringite contributes to stiffening (loss of consistency), setting (solidification of the paste), and early strength development of cement paste or concrete. Later, after the depletion of sulfate in the solution when the aluminate concentration goes up again due to renewing hydration of C_3A and C_4AF, ettringite becomes unstable and is gradually converted into monosulfate, $C_4A\bar{S}H_{10-12}$. While in a CKD-FA system, sulfate content is high due to composition of CKD, and aluminate content may be low due to slow hydration of the Class F FA. Therefore, the ettringite formed in a CKD-FA system is quite stable at the age of 28 days or even at 120 days. Note that although the sulfate content in a CKD-FA system is high, it may not cause abnormal binder setting problem since the alkali content is also high and the sulfate/alkali ratio of the system is low. Thus, the ettringite in a CKD-FA system contributes to both early and later binder strength development [15].

Small calcium hydroxide (CH) peaks are also observed in the CKD-FA pastes at $2\theta \approx 17^\circ$ and 34°. The CH primarily results from hydration of quick lime in CKD. Existence of this hydration product promotes a pozzolanic reaction in the CKD-FA binder system. The reactions can be expressed as follow:

$$\mathrm{CaO} + \mathrm{H_2O} \rightarrow \mathrm{Ca(OH)_2}$$

$$\mathrm{Ca(OH)_2} + \text{reactive } \mathrm{SiO_2} \rightarrow x\mathrm{CaO} - \mathrm{SiO_2} - y\mathrm{H_2O}(\mathrm{C} - \mathrm{S} - \mathrm{Hgel})$$

As presented in Fig. 4, the raw binder material (before hydration) contains crystalline phases of CaO, SiO_2, $CaSO_4$, and $CaCO_3$ from CKD and FA. After hydration at 38 °C for 7 days, the peaks of CaO phase in the XRD curve disappear and the intensity of $CaSO_4$ peaks is significantly reduced. Ettringite is clearly identified as one of the major new crystalline hydration products of the binder in addition to CH. With curing time, it is expected that more amorphous hydration products, such as C-S-H gel, will form due to pozzolanic reaction.

3.2.2. Effects of curing temperature and NaOH addition on CKD-FA binder hydration

Fig. 5 presents effects of curing temperature and NaOH addition on hydration products of CKD-FA binders, and

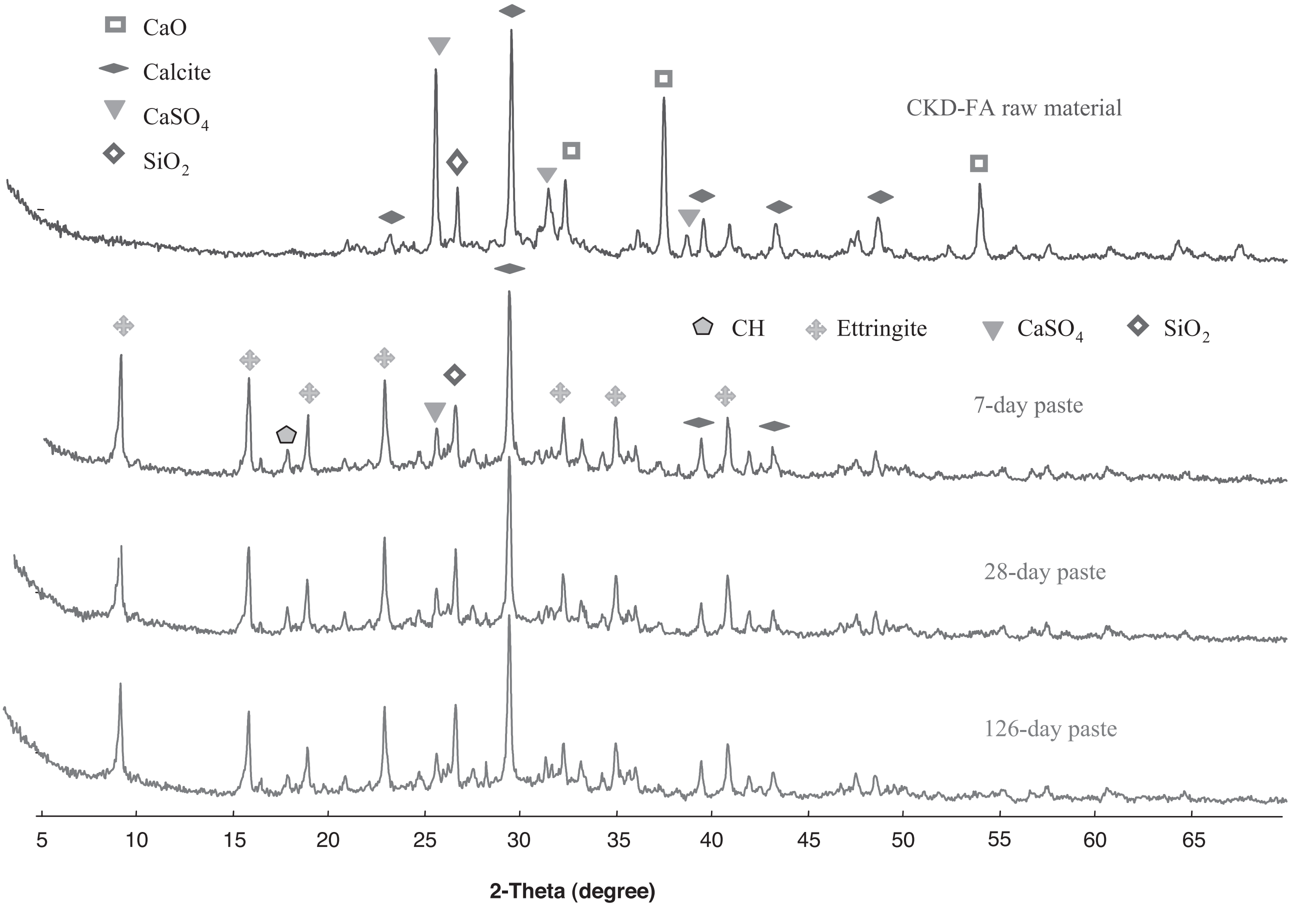


Fig. 4. XRD results of CKD-FA binder (0% NaOH) cured at 38 °C—effect of curing age.

Fig. 6 illustrates the specific effects on ettringite formation. As seen in Fig. 5, alkali (sodium/potassium) sulfate crystals are often found in the pastes having NaOH addition. As observed in Fig. 6, when curing temperature increases from 38 to 50 °C, the intensity of ettringite of a CKD-FA paste slightly increases at 7 days but decreases and becomes flatter at 28 days. The same trend is observed more clearly in OPC pastes. As discussed before, a high curing temperature may promote early hydration but prevent hydration products from diffusing away from the hydrated grain, thus impairing crystal growth at late age. When 2% NaOH was added in a CKD-FA binder, the intensity of ettringite of the paste decreased at both 7 and 28 days. NaOH addition may accelerate chemical dissolution but depress crystal (both ettringite and CH) formation during the binder hydration. Depression of ettringite formation due to NaOH addition has been observed from XRD results of many samples in the present study.

3.3. Binder hydration evaluation based on TGA results

3.3.1. Comparison of TGA results from a CKD-FA paste with an OPC paste

Fig. 7 demonstrates typical TGA curves (upper curves) and their derivative thermogravimetry (DTG) curves (lower curves) of an OPC paste and a CKD-FA paste cured at 24 °C for 28 days. As observed in the figure, at the testing temperature of approximate 45 °C, the OPC paste sample displays a sudden weight loss in its TGA curve, from 100% to 96% of its initial weight; correspondingly, a large peak appears in its DTG curve. This weight loss is primarily caused by loss of free and absorbed water in large capillary pores of the cement paste. In the testing temperature range of 50–100 °C, the sample weight drops again, from 96% to approximate 85%. The rapid weight drop of the sample at this temperature range is mainly related to ettringite decomposition. The two DTG peaks, one of which is at about 50 °C and the other at about 80 °C, are observed in this stage, indicating that two types of crystals decomposed. After the ettringite decomposition, there is a floating of the base line of the TGA and DTG curves. It is believed that the feature of continuous cascade mass loss implies the existence of large amount of C-S-H gel. Water in different bonding status in C-S-H gel structure has different bonding energy and it discharges at different temperatures, thus resulting in the continuous cascade form of the mass loss. The discharging of water causes the decrease in density and the change of thermal conductivity of the sample, therefore resulting in the

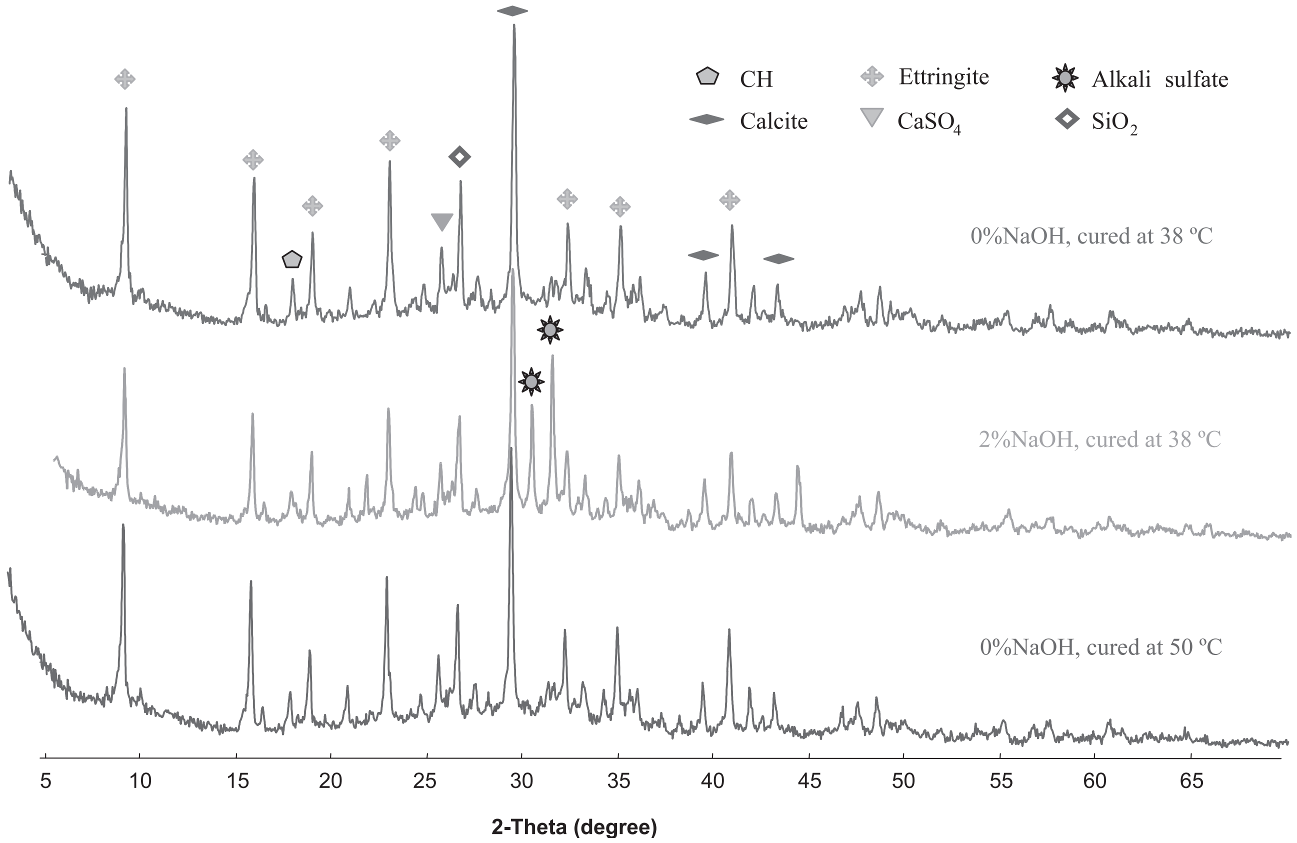


Fig. 5. XRD results of CKD-FA binder—effects of NaOH and curing temperature.

floating of the base line of the TGA curve [10]. When the temperature reaches 410–450 °C, another rapid weight loss occurs in the TGA curve and another peak appears in the DTG curve starting at temperature of 418 °C. The cement paste weight loss and DTG peak at this temperature range are related to decomposition of CH. After temperature reaches 450 °C, floating of TGA and DTG curves are seen again, indicating further C-S-H gel decomposition.

Different from the OPC paste, the TGA and DTG thermographs of the CKD-FA paste indicate that ettringite and C-S-H gel are the major binder hydration products. It is observed that the CKD-FA paste demonstrates only one large DTG peak at testing temperature of 50–80 °C, indicating that one type of crystals decomposed. No DTG peak is seen in the CKD-FA paste at the temperature of 400–450 °C, implying that little CH exists in the 28-day-old CKD-FA paste. Instead, there are two DTG peaks at temperatures of 650–700 °C and 850–950 °C, which may represent $CaCO_3$. $CaCO_3$ existed in the raw materials (shown as the DTG peak at 850–950 °C) and might also result from carbonation of the paste (shown as the DTG peak at 650–700 °C) [1]. Previous research has indicated that depending on its composition, a carbonate mineral may start loss of CO_2 at temperature of approximate 650 °C and complete $CaCO_3$ decomposition at temperature of approximate 900 °C [16].

3.3.2. Effect of curing temperature on CKD-FA binder hydration

Fig. 8 illustrates the TGA and DTG curves of CKD-FA pastes cured at different temperatures for 28 days. Note that the CKD-FA paste cured at room temperature (24 °C) displays only one large distinct peak in its DTG curve at the temperature of approximately 50–80 °C. Differently, the CKD-FA pastes cured at elevated temperatures (38 °C or 50 °C) display two DTG peaks at the testing temperature of approximate 50–80 °C. The first peak is much smaller than the second one (a similar trend was also found from the TGA tests of pastes with different CKD-to-FA ratios). The amount of weight loss within this testing temperature range is 9.116%, 9.833%, and 9.427% for the pastes cured at 24 °C, 38 °C, and 50 °C, respectively. Consistent with the XRD test results, it indicates that 38 °C is the most effective curing temperature for the ettringite formation.

Note that the paste cured at 24 °C has a very small DTG peak at 450–500 °C, indicating the presence of $Ca(OH)_2$ in the sample; while the pastes cured at elevated temperature showed no DTG peak at this tem-

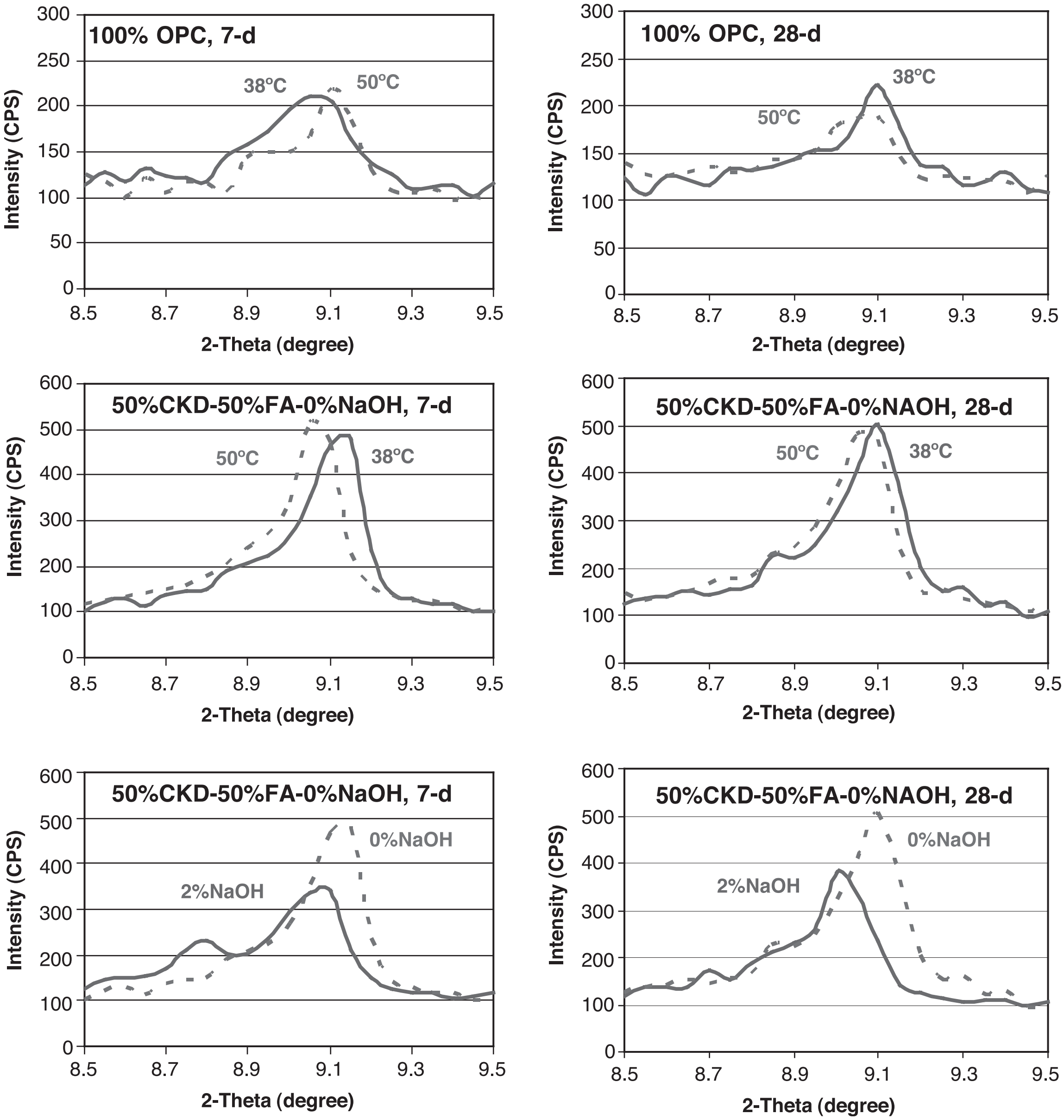


Fig. 6. XRD patterns of ettringite—effects of NaOH and curing temperature.

perature range, possibly due to the accelerated pozzolanic reaction.

3.3.3. *Effect of NaOH addition on CKD-FA binder hydration*

Fig. 9 shows the TGA test results of CKD-FA pastes with different amounts of NaOH additions cured at fog room (24 °C) for 28 days. As illustrated in Fig. 9, ettringite, perhaps together with other sulfate phases, in the sample with 5% NaOH decomposed at the lowest temperature (73.0 °C), and its DTG peak is the lowest. Ettringite in the sample with 2% NaOH decomposed at a medium temperature (79.4 °C). Its DTG peak has almost the same height as that in the sample without any NaOH, which decomposed at the highest temperature (82.5 °C). The figure also demonstrates that the amount of weight loss due to ettringite/sulfate phase decomposition decreases with increased NaOH addition. This implies that high alkali concentration (5% NaOH) may depress ettringite formation in the binder system, which is consistent with XRD results (a similar observation was also found in the corresponding pastes cured at 38 °C, where the amount of ettringite decreased with the amount of NaOH addition and the ettringite formed in the paste with a high NaOH content decomposes at a low temperature). Different from the binder without NaOH addition, CKD-FA binders with 2% and 5% NaOH also display no visible $Ca(OH)_2$ peak in their DTG curves, which suggests that alkali may

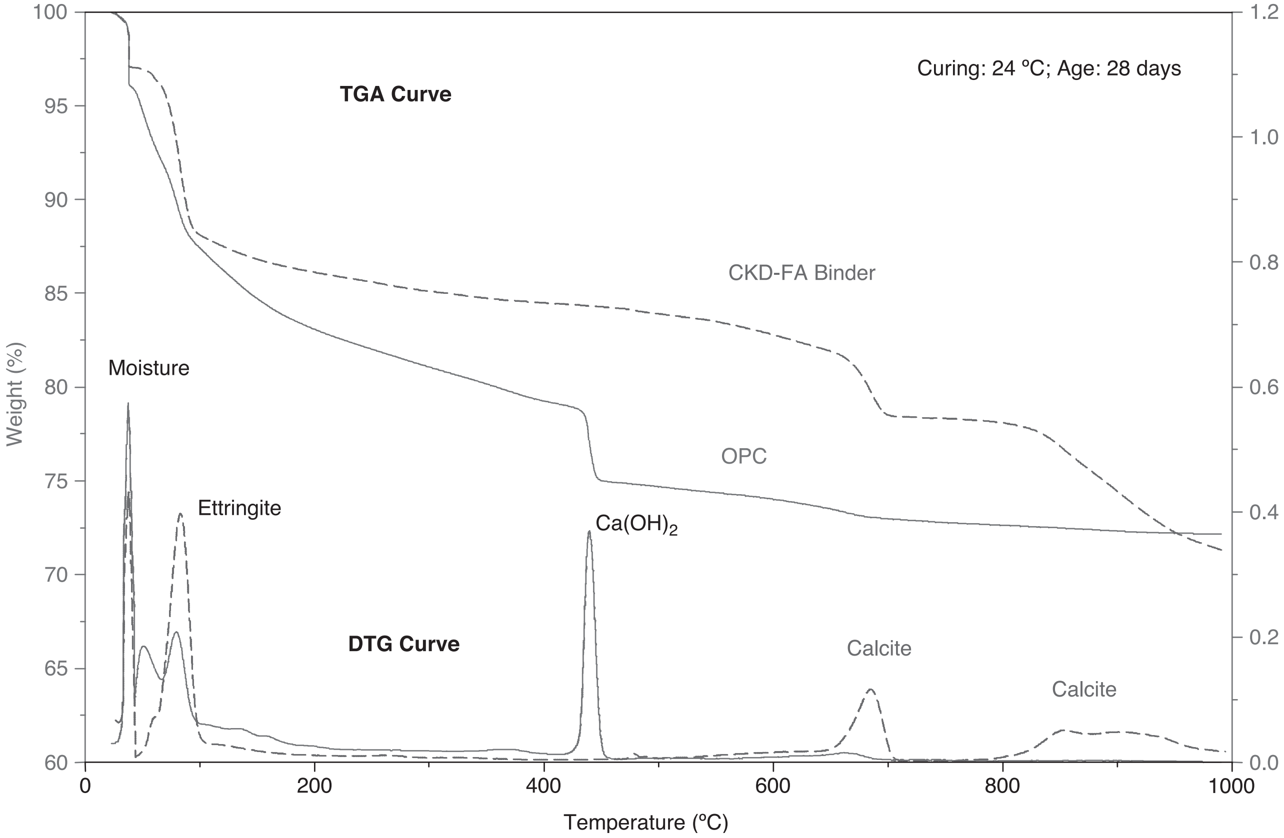


Fig. 7. TGA results—comparison of OPC and CKD-FA pastes.

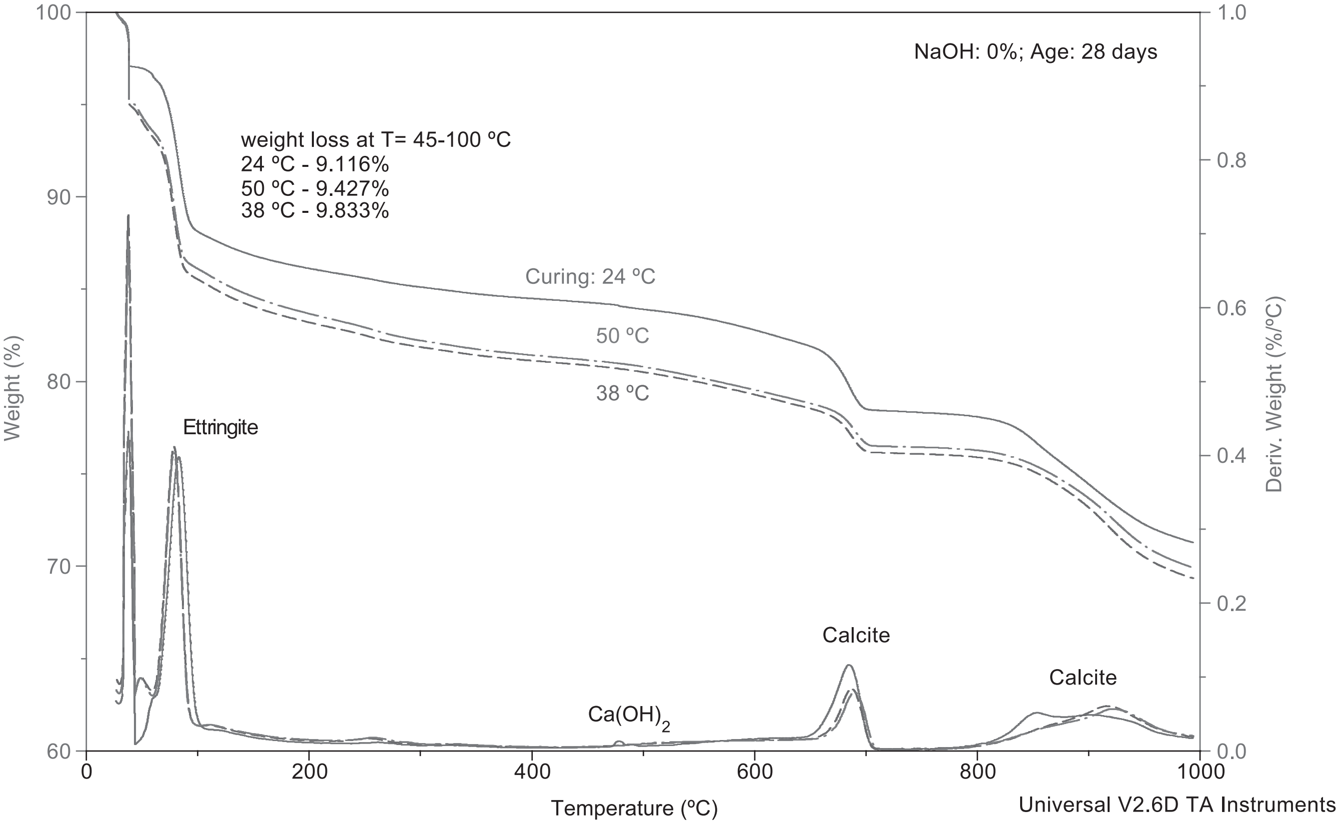


Fig. 8. TGA results—effect of curing temperature on CKD-FA binder hydration.

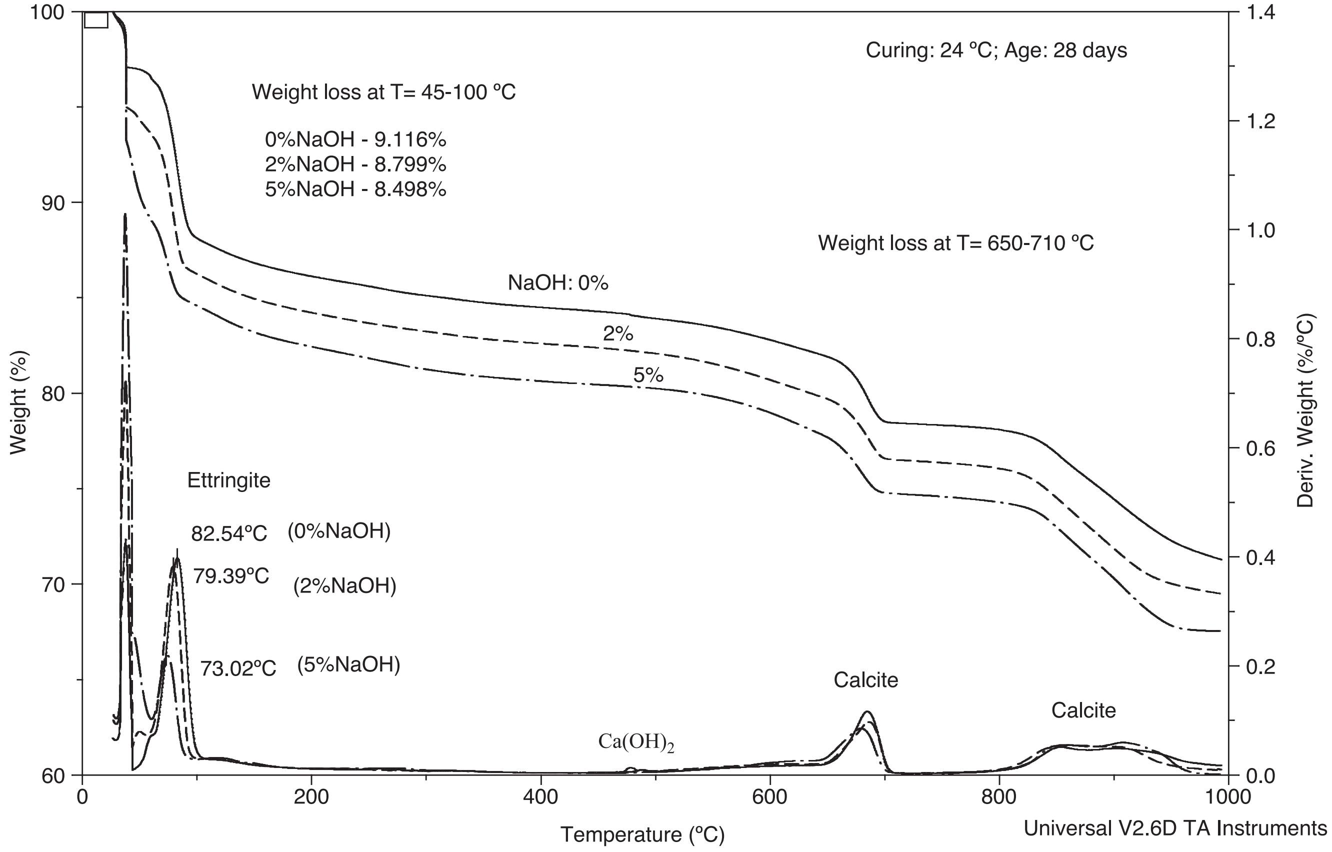


Fig. 9. TGA results—effect of NaOH addition on CKD-FA binder hydration.

depress various types of crystal formation in cementitious systems.

4. Conclusions

The following conclusions can be drawn based on the present study:

1. The major crystalline hydration product of a CKD-FA binder is ettringite. The ettringite formed in a CKD-FA system appeared stable at the age over 100 days;
2. Curing at elevated temperature had more significant influence on CKD-FA binder strength development than on OPC strength development and appeared more effective for CKD-FA binder strength improvement than NaOH addition;
3. Ettringite formation in a CKD-FA binder was slightly reduced, and the crystal formation was impaired at later age when curing temperature increased from 38 °C to 50 °C;
4. NaOH addition might depress ettringite formation in a CKD-FA binder at both early and later ages. With an increased amount of NaOH addition, the ettringite formed in CKD-FA pastes decomposed at a reduced temperature.
5. Addition of 2% NaOH increased the strength of CKD-FA binder at both early and later ages; while addition of 5% NaOH increased the strength only at early age when pastes of the binders were cured at 24 °C or 38 °C. Addition of 2% NaOH will reduce the strength of CKD-FA binder at both early and later ages when the paste of the binder was cured at 50 °C; and
6. Binder made with 50% CKD-50% FA-2% NaOH and cured at 38 °C had satisfactory strength, approximate 27 MPa (4000 psi) at the age of 56 days.

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