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Enhancement of selectivity toward ettringite during hydrothermal processes on fluidized bed combustion wastes for the manufacture of preformed building components

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Abstract

Due to its lime, alumina and calcium sulfate contents, the fluidized bed combustion (FBC) waste is worthy of consideration as a raw material for the hydrothermal synthesis of building elements based on ettringite ($6\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{SO}_3\cdot 32\text{H}_2\text{O}$). Two FBC waste samples (a fly and a bottom ash) were hydrated, alone and in mixture, at temperatures comprised between 40° and 85°C for curing times ranging from 2 hours to 7 days, and were submitted to X-ray diffraction and differential thermal–thermogravimetric analyses. It has been found that: a) the FBC fly ash could be hydrated alone, due to its satisfactory ettringite-generating ability; b) the FBC bottom ash needed to be used together with other raw materials; c) progressively better results were obtained by adding i) FBC fly ash to FBC bottom ash (to give a blend with a 60:40 mass ratio), ii) anodization mud (a by-product of anodized aluminum manufacture, acting as an additional source of alumina and calcium sulfate) in a measure of 10% by mass to the fly–bottom ash blend, iii) anodization mud (20% by mass) to the FBC bottom ash. Moreover, compressive strength measurements were carried out, according to the EN 196-1 Standard for cements, on the hydrated systems showing the two largest ettringite concentrations, and a maximum value of about 6 MPa was reached at 70°C and 16 hours of curing. The data obtained in this investigation were consistent with an industrial prefabrication of building components.

Keywords: By-product utilization; Fluidized bed combustion waste; Building industry; Prefabrication; Hydrothermal treatment; Ettringite.

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1. Introduction

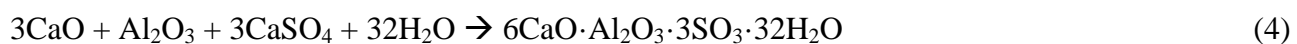
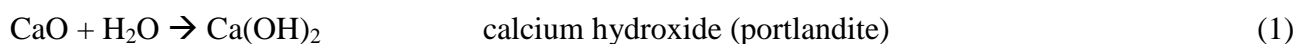
Non-traditional building elements containing ettringite ($6\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{SO}_3\cdot 32\text{H}_2\text{O}$) as a main binding phase¹ can replace cementitious products whose properties are chiefly regulated by calcium silicate hydrate,² generated by Portland cement or blast-furnace slag hydration as well as pozzolanic reaction. An accelerated curing (AC) at temperatures higher than room temperature is the basic process which controls the manufacture of precast construction materials.^{3,4} In the case of utilization of Portland cement, AC allows to obtain the industrial product within short aging times; when a blast-furnace slag or a pozzolanic material (natural pozzolan, pulverized coal fly ash and so on) is used together with a source of lime acting as, respectively, stimulating agent or basic reactant, AC enables a rapid generation of substantial amounts of calcium silicate hydrate, otherwise obtainable (to a very limited extent) only from slow room temperature-processes such as slag hydration or pozzolanic reaction. AC is generally carried out⁵ at (i) low pressure (atmospheric pressure), keeping the hydrating materials in contact with water or steam at temperatures lower than 100°C or (ii) high pressure (in autoclave), through the exposure to saturated steam at temperatures higher than 100°C . Ettringite is a compound of interest for the manufacture of prefabricated building materials, due to its binding ability, water insolubility, low density and fire resistance.⁶ It can be obtained from the hydration of sources of sulfates and oxides or hydroxides of calcium and aluminium. Similarly to the slag hydration or the pozzolanic reaction, the ettringite formation processes are rather slow at room temperature, and higher curing temperatures are required to promote the ettringite generation.⁷⁻¹⁰ AC carried out by means of the above-mentioned hydrothermal treatments can be therefore proposed for the manufacture of ettringite-based materials, except for the high pressure processes due to the considerable ettringite instability at temperatures higher than 100°C .² Moreover, it is interesting to observe that industrial wastes and by-products can be suitably used as raw materials for the manufacture of ettringite-based building elements.⁶

As far as the burning of the solid fuels is concerned, the fluidized bed combustion (FBC) technology offers several advantages from the technical and environmental point of view.¹¹⁻¹⁴ The

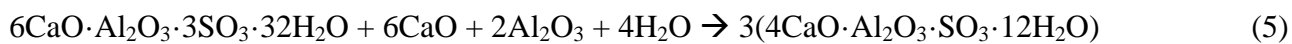
FBC solid residues are composed by coal ash and exhausted calcium-based sulfur sorbent; they are difficult to reuse in traditional application fields such as ordinary cements and concretes, due to chemical and mineralogical characteristics which are different from those of the ash coming from a pulverized coal-fired plant. In particular, FBC ash has a much lower pozzolanic activity, because of its limited glass content associated with the reduced combustion temperature.¹⁵ Furthermore, the CaSO₄ and free CaO amounts, mainly given by the exhausted sulfur sorbent, are responsible for exothermal and expansive phenomena upon hydration, even able to complicate the direct landfilling of the FBC waste,^{16,17} for which alternative uses have therefore to be found out^{18–20}.

With a view to reuse combustion ashes,^{21,22} the authors successfully investigated the formation of ettringite upon reaction of FBC waste with water, in order to utilize the hydrated residue as a highly active SO₂ sorbent.^{8–10,23,24} Furthermore, several interesting data have been reported in the scientific literature about the use of (a) FBC waste, (b) by-products originated from traditional coal-fired power plants and (c) other industrial wastes as kiln feed components for the synthesis of (i) ordinary and (ii) special (calcium sulfoaluminate-based) cements.^{25–61}

In a recent paper,¹ two FBC waste samples (a fly and a bottom ash) were explored as single raw materials for the manufacture of building components based on ettringite, in the context of a more general literature scenario concerning this topic:^{62–65} the results, especially for the bottom ash, were not fully satisfactory in terms of ettringite formation rate and side-products minimization. Previous investigation^{1,8,10,23} on ettringite-generating systems showed that the main reactions occurring during the hydration process are the following:



hexacalcium trisulfoaluminate dotriakontahydrate (ettringite)



tetracalcium monosulfoaluminate dodekahydrate (monosulfate)

The last reaction results from the ettringite instability in a CaSO_4 -depleted environment.² The sources of lime, calcium sulfate and alumina required by the ettringite formation directly arise from the raw materials; the first two can be also given by the hydration products of the reactions (1), (2) and (3). The crystalline silica contained in the FBC waste is not expected to have a role in the hydration process, due to its very poor reactivity with lime and water at the investigated curing temperatures.²

The present work is mainly concerned with the effects of the composition of mixtures containing FBC wastes on their ettringite-generating ability. Moreover, the compressive strength of two hydrating systems was evaluated as a function of curing time and temperature.

2. Materials and methods

As raw materials for this investigation, two samples of FBC ashes (a fly- and a bottom-ash, respectively labeled FA and BA) and one sample of anodization mud (AM, a by-product of the anodized aluminium manufacture) were used. The FBC samples were generated in a full-scale 790 MW_{th} circulating reactor, fired with an Italian coal, and were supplied by the ENEL Research Centre of Tutturano (Brindisi, Italy). The AM sample came from an anodized aluminium factory, operating at Tito Scalo (Potenza, Italy). All the samples were ground in a laboratory mill to pass the 90 µm-sieve before the characterization tests. The chemical composition of the raw materials is indicated in Table 1.

As analytical techniques, X-ray fluorescence (XRF) and X-ray diffraction (XRD) analyses were employed for the evaluation of the chemical and mineralogical composition of the raw materials, using, respectively, a BRUKER Explorer S4 instrument and a BRUKER D2 Phaser diffractometer (Cu α radiation, 0.02°/s scanning rate) operating between 5° and 60°2 θ . Fig. 1 illustrates the XRD patterns for the raw materials used in this investigation. The main crystalline phases of BA

and FA were anhydrite, lime, quartz, portlandite; those detected for AM were bassanite, calcite, gibbsite, dolomite.

Five Systems, numbered from 1 to 5, were investigated (Table 2). No.1 and No.2 were 100% BA and 100% FA, respectively; No.3 was a FA–BA mixture, in a mass ratio corresponding to the usual proportions of the two generated streams leaving the FBC reactor; No.4 was a binary mixture, composed by BA and AM; No.5 was a ternary mixture consisting of 90% System 3 and 10% AM.

In order to simulate the water-curing process at atmospheric pressure, all the systems were paste hydrated with a 1.0 water/solid mass ratio and cured in a thermostatic bath held at 40°, 55°, 70°, 85°C for the Systems 1 and 2, and at 55° and 70°C for the others. At the end of each aging period, ranging from 2 hours to 7 days, the hardened pastes were pulverized after grinding under acetone in an agate mortar (to stop hydration), then treated with diethyl ether (to remove water) and finally stored over silica gel–soda lime (to ensure protection against H₂O and CO₂).

The XRD analysis was further utilized for the characterization of the hydrated samples, together with the simultaneous differential thermal–thermogravimetric analysis (DTA–TG) carried out through a NETZSCH Tasc 414/3 apparatus (20°–1000°C temperature range, 10°C/min heating rate). The TG analysis was also used for quantitative purposes, in order to determine the ettringite concentration in the hydrated systems; to this end, it was assumed that 24 water moles were lost by heating 1 mole of ettringite in the narrow temperature range corresponding to its strong endothermal effect.²

Compressive strength measurements on mortar were carried out for the Systems 4 and 5, according to the EN 196-1 Standard for cements. Before the test, the specimens were cured for 8, 16 and 24 hours at 55° and 70°C.

3. Results and discussion

From the examination of both the chemical (Table 1) and mineralogical (Fig. 1) composition of the raw materials it can be argued that: 1) owing to the operating conditions adopted in the FBC reactor,

the elutriated fly ash, differently from the bottom ash which was quite rich in the sorbent-derived (sulfocalcic) fraction, was relatively high in silica, alumina, iron oxide and unburned carbon (loss on ignition) in consequence of its larger coal ash concentration as well as the limited burn-off and calcium sulfation degrees associated with the short residence time; 2) for BA and FA, (i) the SO_3 amount was in practice fully involved in the CaSO_4 formation and (ii) the “free” lime content (namely the content of the lime available for the hydration process) could be calculated as a difference between the total CaO amount and the amount of calcium oxide bound in calcium sulfate: this implied CaSO_4 and free CaO contents respectively equal to 53.0% and 26.0% for BA as well as 30.9% and 26.9% for FA; 3) for BA and FA, calcium hydroxide arose from CaO hydration due to the weathering of both ashes, before their sampling; 4) basically, AM had no free lime and was a source of alumina (only in part crystalline) and sulfate (under the form of bassanite); its CaO amount was virtually combined in both calcium sulfate hemihydrate and calcium carbonate, and its high l.o.i. was ascribed to the chemically-bound H_2O and CO_2 .

For every investigated system, the maximum potential ettringite concentration (MPEC) index attainable in the paste upon hydration was evaluated by means of stoichiometric calculations and reported in Table 2. MPEC indicates the ettringite concentration in the hydrated paste as “maximum potential”, actually obtainable if (1) the hydration process were gone to completion and (2) no side-products able to subtract reactants (CaO , Al_2O_3 , CaSO_4) to the ettringite formation were generated. As expected, on the basis of the results previously¹ obtained for another couple of FBC ashes, the fly ash (System 2) had a MPEC index quite higher than that of the bottom ash (System 1), due to its larger content of limiting reactant (alumina). The System 3 was formulated in order to maximize the utilization of both FBC ashes: of course, its MPEC index was comprised between those of the Systems 1 and 2. It has been found that the AM addition, while unable to enhance to a very significant extent the MPEC index for the fly ash, promoted a large increase of that for the bottom ash (see the data in Table 2 for the Systems 4 and 5, whose composition was conceived with a view to improve the hydration behavior of the Systems 1 and 3, respectively).

The actual ettringite concentration values, measured by means of the quantitative TG analysis, are reported as a function of the curing time in Figs. 2(a), 2(b) and 2(c), for the Systems 1, 2 and 3–4–5, respectively. For each system, they were lower than the related MPEC indexes, due to the incompleteness of the ettringite formation process and, to a lesser extent, the generation of monosulfate (see later on). On the other hand, it is observed that the relative ranking predicted on the basis of the MPEC index in Table 2 (System 4>System 5>System 2>System 3>System 1) was experimentally verified by the data in Fig. 2. Namely, MPEC data are useful to understand how to design a certain mixture, in order to improve its ettringite-generating potential. On this basis, in particular, Systems 4 and 5 were designed, and they were those experimentally giving the best results in terms of ettringite generation. More in detail, the best approach to the MPEC index was shown by the System 4, followed by the Systems 5 and 2, in the order. From the examination of Figs. 2(a) and 2(b) the following observations can be drawn: a) compared to the bottom ash, the fly ash had a greater ettringite-generating ability; b) an increase of the curing temperature from 40° to 55°–70°C enhanced the ettringite formation rate; c) 85°C was not the best temperature, due to the occurrence of ettringite decomposition phenomena already observed¹ and more pronounced for the fly ash. From Fig. 2(c) it can be achieved that (i) the System 3 was better than the System 1 and (ii) the Systems 4 and 5, when compared to the Systems 1 and 3, respectively, were able to give much larger ettringite concentrations. For each of the Systems 3–4–5, an increase in temperature from 55° to 70°C generally determined ettringite concentration values (i) slightly larger at early ages (owing to the favorable influence of the hydration temperature on the ettringite formation kinetics) and (ii) slightly smaller at longer curing times (due to the above-mentioned high-temperature decomposition phenomena).

A further investigation was carried out on the AM-containing Systems 4 and 5, since they were those showing the largest values for the ettringite concentration in the hydrated pastes. Figs. 3 and 4 illustrate, respectively, the DTA–TG thermograms and the XRD patterns for the Systems 4 and 5 cured for 1 and 7 days at 55° and 70°C. The thermal effects (Fig. 3) of ettringite (164°±8°C) and

calcite ($762^{\circ}\pm 36^{\circ}\text{C}$) were clearly recognized.² The latter was due to calcium carbonate (i) given by AM and (ii) formed at the expense of the lime excess not involved in the ettringite generation. By means of XRD analysis (Fig. 4), other phases such as side products (monosulfate from the reaction (5)), unreacted anhydrite and inert quartz were detected. For both the hydrated systems, no significant differences were observed among the XRD patterns at the investigated curing temperatures and times.

In Figs. 5(a) and 5(b), the compressive strength data for the Systems 4 and 5 are respectively reported, as a function of the aging temperatures (55° and 70°C) and times (8, 16 and 24 hours). The maximum values were reached at 70°C and 16 hours of curing (6.2 MPa for the System 4, 6.0 MPa for the System 5) and had the same order of magnitude as those previously^{66,67} obtained for systems composed by pulverized coal ash, lime and calcium sulfate dihydrate (given by natural or chemical gypsum). The period of 16 hours is included in the range of curing times (8–24 hours) generally required by an industrial manufacture of precast construction materials based on an accelerated water-curing process.^{5,68}

4. Conclusions

When used as single raw materials submitted to hydrothermal treatments aimed at obtaining ettringite-based building elements, both FBC fly and bottom ashes confirmed to be able to generate ettringite to an extent which was larger in the case of the fly ash, due to its more proportioned contents of the oxides entering into the ettringite composition. It has been found that the best results were obtained when the hydration process was carried out at 55° – 70°C ; below 55°C and above 70°C a significant decrease of the ettringite generation was observed, due to the reduced reaction rate and the occurrence of ettringite decomposition phenomena, respectively. Moreover, the addition of (i) the fly ash to the bottom ash (to give a blend with a 60:40 mass ratio) and, particularly, (ii) anodization mud to the bottom ash and to the 60:40 blend (in a measure of 20% and 10% by mass, respectively) was able to promote a large increase of the ettringite concentration. Fair

enough compressive strength results were shown by the hydrated systems with the two highest ettringite concentration values. They were reached at curing times (8–24 hours) compatible with an industrial manufacture of preformed building elements.

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Table 1. Chemical composition of FA, BA and AM, mass %, in terms of major oxides (evaluated by means of X-ray fluorescence analysis) and loss on ignition (l.o.i.) at 950°C.

	FA	BA	AM
SiO ₂	16.0	12.0	2.9
Al ₂ O ₃	6.9	3.3	45.3
Fe ₂ O ₃	9.2	1.9	2.8
TiO ₂	0.4	0.2	–
CaO	39.6	47.8	7.7
MgO	1.8	0.8	2.7
K ₂ O	0.5	0.2	–
SO ₃	18.2	31.2	8.3
l.o.i. ^a	5.7	1.8	30.2
Total	98.3	99.2	99.9

^aDetermined according to EN 196-2 Standard for cements.

Table 2. Composition and MPEC index for the investigated systems, mass %.

	1	2	3	4	5
FA	–	100	60	–	54
BA	100	–	40	80	36
AM	–	–	–	20	10
MPEC	34.2	61.0	51.3	86.6	75.7

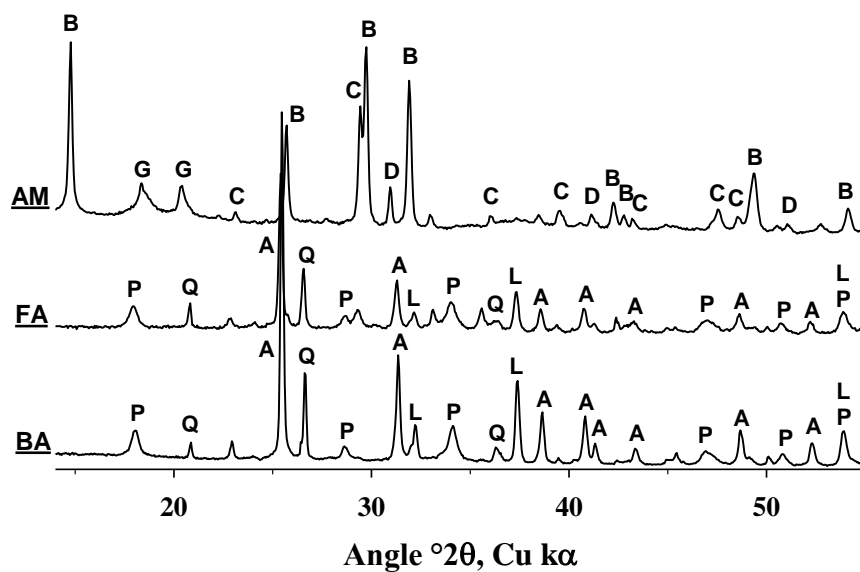


Fig. 1. XRD patterns for the raw materials. A=anhydrite (CaSO_4); B=bassanite ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$); C=calcite (CaCO_3); D=dolomite (MgCO_3); G=gibbsite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$); L=lime (CaO); P=portlandite ($\text{Ca}(\text{OH})_2$); Q=quartz (SiO_2).

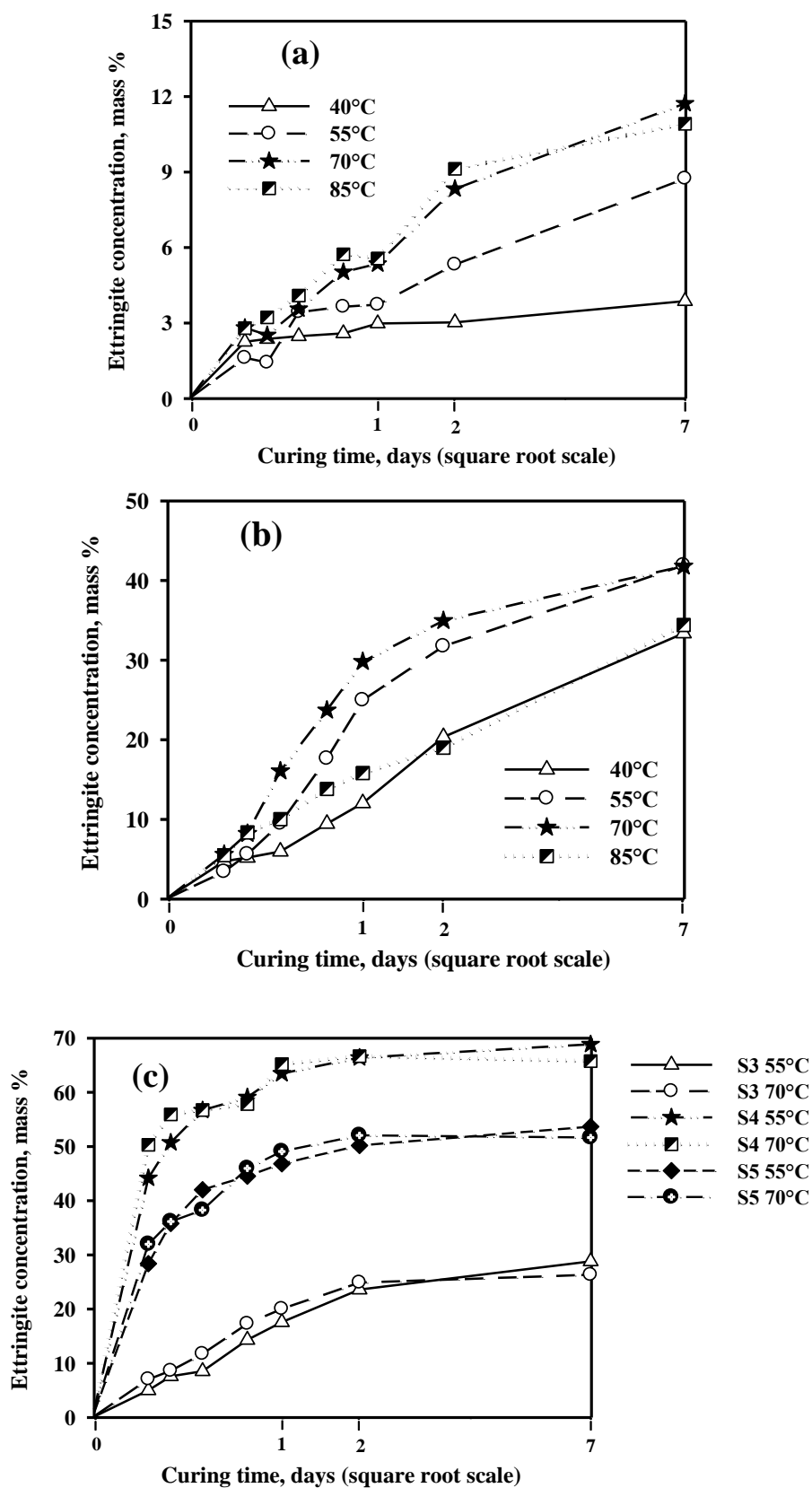


Fig. 2. Ettringite concentration vs. curing time for:
 (a) System 1, hydrated at 40°, 55°, 70° and 85°C;
 (b) System 2, hydrated at 40°, 55°, 70° and 85°C;
 (c) Systems 3, 4 and 5 (S3, S4 and S5), hydrated at 55° and 70°C.

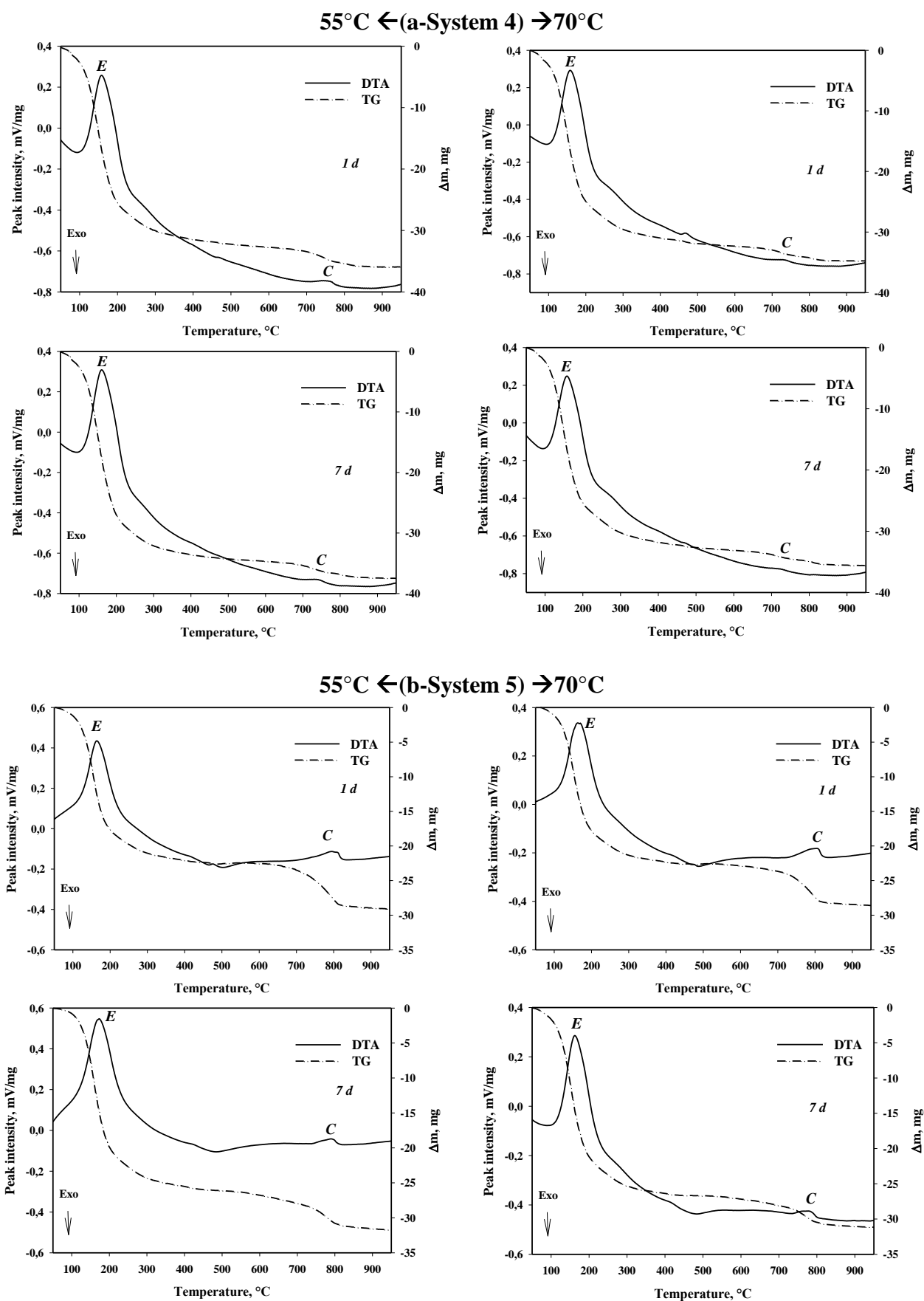


Fig. 3. DTA–TG thermograms (E=ettringite, C=calcite) for systems cured for 1 and 7 days at 55° (left) and 70°C (right):
(a) System 4;
(b) System 5.

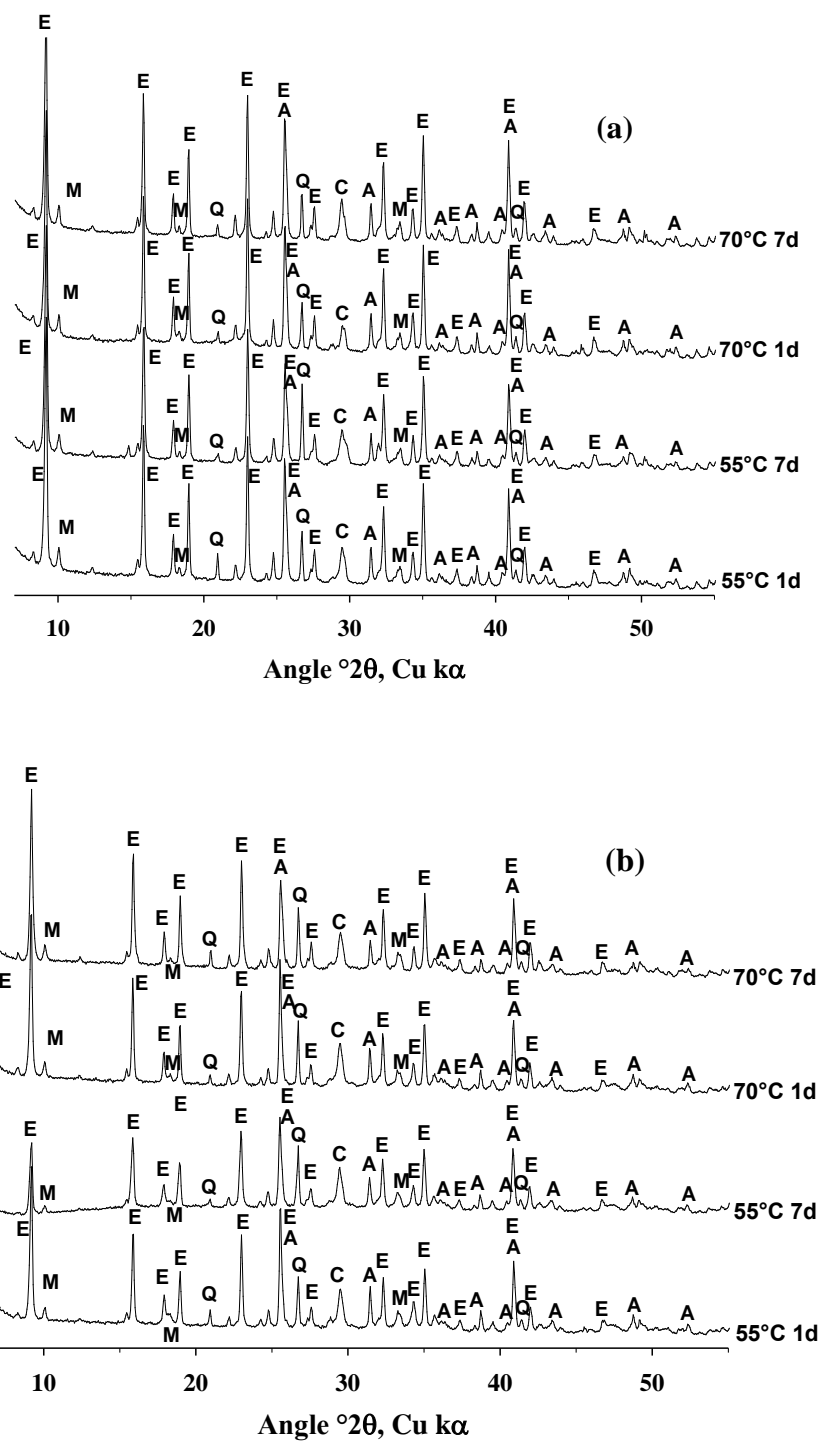


Fig. 4. XRD patterns (A=anhydrite, C=calcite, E=ettringite, M=monosulfate, Q=quartz) for systems cured for 1 and 7 days at 55° and 70°C:

- (a) System 4;
- (b) System 5.

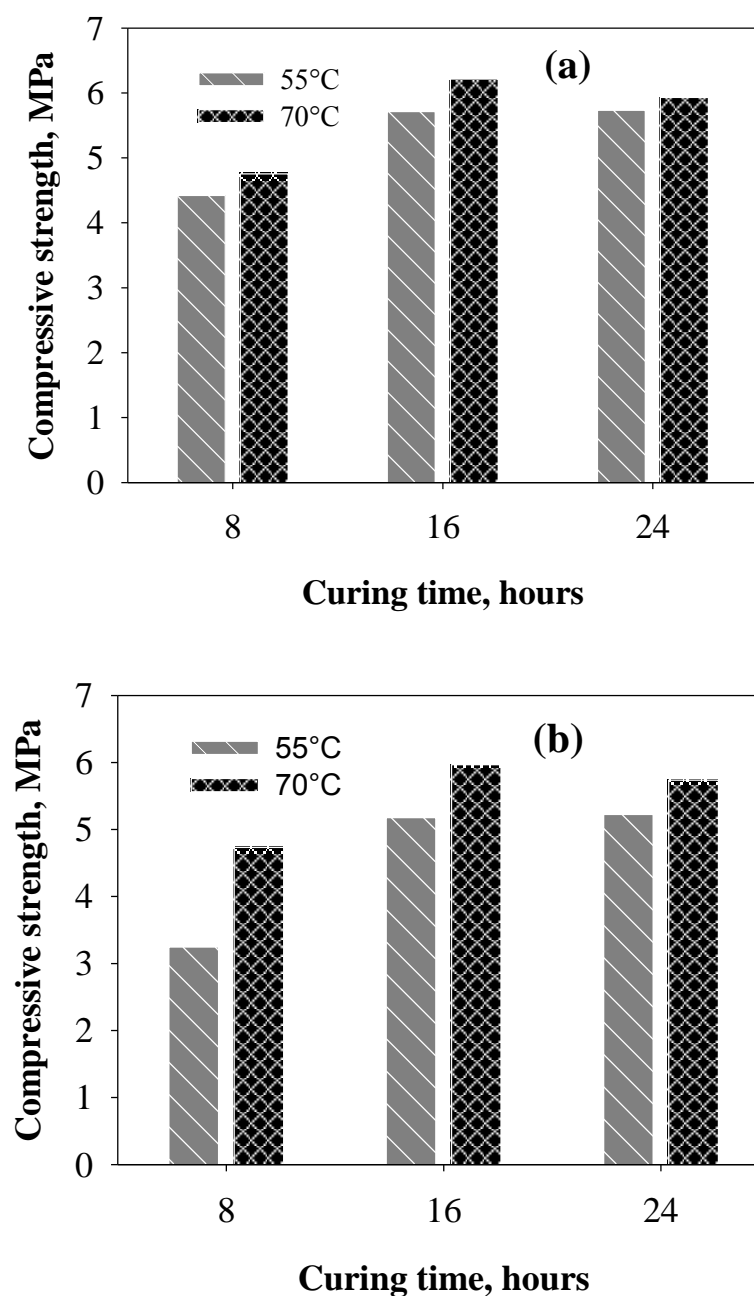


Fig. 5. Compressive strength data for systems cured for 8, 16 and 24 hours at 55° and 70°C:
(a) System 4;
(b) System 5.

The advance is concerned with reactivity improvements of hydrating systems, based on fluidized bed combustion wastes, toward ettringite.

