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TRANSITION AND DECOMPOSITION TEMPERATURES OF CEMENT PHASES – A COLLECTION OF THERMAL ANALYSIS DATA

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Thermal analysis techniques provide the cement chemist with valuable tools to qualify and quantify the products formed during the hydration of cementitious materials. These techniques are commonly used alongside complimentary techniques such as X-ray diffraction and electron microscopy/energy dispersive spectroscopy to confirm the composition of phases present and identify amorphous material unidentified by other techniques. The most common thermal analysis techniques used by cement chemists are thermogravimetry, differential thermal analysis and differential scanning calorimetry. In order to provide a useful reference tool to the cement chemist, this paper provides a brief summary of the temperatures at which phase changes occur in the most common cement hydrates in the range 0-800 °C in order to aid phase identification.

Nomenclature: The data collated here will be of most interest to cement chemists, so standard cement nomenclature has been used throughout, where C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, M = MgO, $\overline{S}$ = SO_3, $\overline{C}$ = CO_2, H = H_2O.

INTRODUCTION

Ordinary Portland cement (OPC) is an inorganic binder produced by calcining a blend of siliceous and calcareous material at approximately 1450°C in a rotating kiln. The resulting clinker is ground with gypsum to form a powder which when mixed with water forms a paste that sets hard with time [1]. The main anhydrous cement phases (C_3S, β-C_2S, C_3A and C_4AF) are crystalline and as such, their detection by X-ray diffraction (XRD) is possible. Some of the products formed upon cement hydration are also crystalline, such as calcium hydroxide (CH), ettringite (also known as AF_t, $C_3A.3C\overline{S}.H_{32}$) and monosulphate (known as AF_m, $C_3A.C\overline{S}.H_{12}$), but the majority of the hydrated material is an ill-defined hydrated calcium silicate hydrate gel, usually referred to as C–S–H, which is amorphous or only partially crystalline. The hydration products of other cements are mostly crystalline; for example, in calcium sulphoaluminate cement (CSA), the main hydration product is AF_t and, depending upon the period of hydration, is likely to be present with anhydrite ($C\overline{S}$) or gypsum ($C\overline{S}H_2$). The hydration products of high alumina cement (HAC), also known as calcium aluminate cement (CAC), are a mixture of crystalline phases (CAH_{10}, C_2AH_8 and C_3AH_6) and alumina gel, but the composition depends upon hydration temperature.

Various techniques are used to detect the presence of cement hydrates, with some (such as XRD) only being able to detect crystalline material. Additionally, the overlapping of XRD reflections makes identification of individual phases difficult in a multi-phase hydrated material such as that which results from mixing OPC, or composite cements based on OPC, with water. This is why thermal analysis has been used for many years as an analysis technique complimentary to XRD in order to detect the amorphous fraction of hydrated material. The use of thermal analysis techniques continues today as a powerful tool for cement chemists [2, 3, 4], which highlights its importance to the cement community. A number of thermal analysis techniques have been used by the cement chemist with the most popular being those based on mass loss during thermal decomposition and temperature change and/or heat flow in relation to a reference during heating.

A widely available reference document listing the temperatures at which cement hydrates decompose or change state, and which can thereby be used to aid phase identification, particularly of amorphous or nano-crystalline phases, does not exist. Therefore, to aid cement scientists, this paper provides a brief compilation of temperatures between 0 and 800°C at which the principal phase changes and decompositions occur in the most common cement hydrates; the majority of the reference data originates from fundamental research performed between 20 and 30 years ago.

EXPERIMENTAL

Thermal analysis techniques

Three techniques that are commonly used to detect hydrated cementitious material are thermogravimetric analysis (TG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC). These techniques are explained in depth in standard textbooks such as those by Dunn and Sharp [5], Dodd and Tonge [6], Wendlandt [7], Daniels [8] and MacKenzie [9]. Texts specific to the application of thermal methods to cement, concrete and construction materials include those by Ramachadran [10], Bárta [11] and Ramachadran *et al*. [12].

In recent years, technological advances have allowed different techniques to be combined into one machine, with instruments offering DSC/DTA [13], TG/DTA [13], DSC/TG [14, 15] and TG/DTA/DSC [16] being available. The thermal analysis instrument can also be combined with gas analysis equipment such as mass spectrometry, gas chromatography and Fourier Transform infra-red spectroscopy to allow identification of phase decomposition in real time [14].

The choice of which technique to use requires consideration because not each technique can detect all the changes in state exhibited by all the phases within a sample. TG only detects phases that exhibit mass loss on heating whereas, DTA and DSC can identify phase composition from transformations that have occurred in the sample such as decomposition, crystallisation, sublimation, glass transitions, phase transitions and melting. It should be noted that various experimental conditions, such as sample size, heating profile, heating environment and composition of sample pan, all influence the results obtained, so experimental error should be reduced by using consistent analysis conditions. Using different techniques may also yield slightly dissimilar results.

Because the majority of thermal analyses performed on hardened cement pastes have been undertaken using TG and DTA, rather than using DSC, most of the data compiled here have been obtained using the former two techniques.

RESULTS AND DISCUSSION

Cement phases and temperatures of their decompositions or phase transitions

A brief description of the principal anhydrous and hydrated phases that will decompose in the temperature range being addressed here follows to provide a compositional explanation to the reader.

Anhydrous phases

Calcium carbonate ($C\overline{C}$, in the form of calcite, vaterite and aragonite) is one of the principal raw materials used in producing OPC and decomposes to burnt lime (C) in the cement kiln, which reacts with the siliceous material present to form the calcium silicates alite (C_3S) and belite (β-C_2S). Calcium carbonate is also added to OPC as a filler and provides advantages with workability. Additionally, one of the main hydration products of the calcium silicates in OPC powder is calcium hydroxide (CH), which, upon mixing the OPC powder with water in air, will carbonate to form calcium carbonate.

To control the setting of the calcium aluminate phase in OPC (C_3A) and avoid what is referred to as a flash set, gypsum is added to OPC clinker, usually during grinding. This sulphate phase reacts with the C_3A during hydration to form ettringite. During grinding and processing, the gypsum can decompose to hemihydrate ($C\overline{S}H_{0.5}$, also known as Plaster of Paris or bassanite) or in extreme cases to anhydrite ($C\overline{S}$). Any hemihydrate is likely to re-hydrate to re-form gypsum. The alumina-ferrite phase present in OPC (C_4AF) is a minor phase, is less reactive than the other cement phases and if identified generally remains as a relic in any hydrated cement paste.

Hydrated cement phases

The main hydration product of the anhydrous calcium silicates in OPC is C–S–H, and it is this binding phase that is responsible for the majority of the strength gain in the hardened cement paste. The other main hydration product of the calcium silicates in OPC is calcium hydroxide (CH) which is a highly crystalline phase with good cleavage and which precipitates during hydration as large platy crystals, and as such, can exhibit preferred orientation.

The two main hydrated sulphate phases formed in the hydration of OPC are AF_t and AF_m. The former is a calcium aluminate tri-sulphate phase formed during hydration from reaction between the C_3A and the calcium sulphate added during grinding of the clinker. When the supply of sulphate ions runs out, any AF_t formed reacts with any remaining C_3A to form the calcium aluminate monosulphate AF_m. Therefore, it is rare to detect any calcium sulphate phases at the same time as AF_m. AF_m contains less bound water than AF_t (12 and 32 groups respectively), and this water is held more tightly within the crystal structure which results in a higher dehydration temperature.

Cements high in aluminate phases hydrate to form products that are different to those of OPC. In all calcium aluminate cements, monocalcium aluminate (CA) is the major component which reacts with water to form a series of calcium aluminate hydrates. At low temperatures (< 18°C) these are an alumina gel and CAH_{10}, whilst at higher temperatures C_2AH_8 is also formed. C_2AH_8 and CAH_{10} are thermodynamically metastable and, at temperatures above 28°C, they “convert” easily to C_3AH_6 and $Al(OH)_3$ (gibbsite). The hydration of low purity calcium aluminate cement in the presence of other secondary cementitious materials such as blast furnace slag (BFS) forms strätlingite (C_2ASH_8). An example of this type of cement is BRECEM [17]. Alumina gel is the main hydration product formed in calcium aluminate cements, but it has also been suggested that it must be a calcium-aluminate-hydrate (C-A-H) phase [18]. The main phases formed by "conversion" in the hydration of calcium aluminate cement release water at temperatures higher than those present before conversion.

Many minor hydration products of OPC contain magnesium and form from the small quantity (approximately 2 - 4 mass. %) of magnesium oxide present in the OPC. Brucite (MH), forms from the hydration of periclase (M). The formation of magnesite (MC) will occur upon extensive mixing of the paste or during hydration in an atmosphere containing carbon dioxide. Hydrotalcite ($M_6A\bar{C}H_{12}$), is another magnesium containing phase that forms when BFS is hydrated and the decomposition characteristics haven been studied by Parker *et al.* [19].

Decomposition/Phase change temperatures

A summary of temperatures at which phase changes occur in anhydrous and hydrated cement are shown in Table 1 in general ranges of 50°C, while in Table 2 the decomposition range for the various compounds is given. It should be noted that all decompositions where there is loss of water or CO_2 are endothermic.

Table 1. Summary of compounds giving peaks in temperature ranges between 0 and 800°C.

Temperature (°C)	Phase composition	Cement chemist's nomenclature
50-100	$CaSO_4{\cdot}2H_2O$	$C\bar{S}H_2$
100-150	$CaSO_4{\cdot}2H_2O$	$C\bar{S}H_2$
	$CaSO_4{\cdot}½H_2O$	$C\bar{S}H_{0.5}$
	–	C–S–H ^{a}
	$Ca_3Al_2O_6{\cdot}3CaSO_4{\cdot}26H_2O$ $^{a, b}$	$C_3A{\cdot}3C\bar{S}{\cdot}H_{32}$ $^{a, b}$
	–	C–S–H
	$CaAl_2O_4{\cdot}10H_2O$ ^{b}	CAH_{10} ^{b}
	$Mg_6Al_2O_9{\cdot}CO_3{\cdot}12H_2O$	$M_6A\bar{C}H_{12}$
150-200	$CaSO_4{\cdot}2H_2O$	$C\bar{S}H_2$
	$CaSO_4{\cdot}½H_2O$	$CS\bar{S}H_{0.5}$
	$Ca_3Al_2O_6{\cdot}CaSO_4{\cdot}12H_2O$	$C_3A{\cdot}C\bar{S}{\cdot}H_{12}$
	$Ca_2Al_2O_5{\cdot}8H_2O$	C_2AH_8
	$Ca_4Al_2O_7{\cdot}13H_2O$	C_4AH_{13}
200-250	$CaSO_4{\cdot}½H_2O$	$C\bar{S}H_{0.5}$
	$Ca_2Al_2SiO_7{\cdot}8H_2O$	C_2ASH_8
	$Al(OH)_3$ ^{c}	–
	$Al(OH)_3$ $^{c, d, e}$	–
250-300	$Al(OH)_3$ ^{f}	–
300-350	$Ca_3Al_2O_6{\cdot}6H_2O$	C_3AH_6
	$Al(OH)_3$ $^{b, c, g}$	–
	$Mg_6Al_2O_9CO_3{\cdot}12H_2O$	$M_6A\bar{C}H_{12}$
350-400	$CaSO_4$ ^{h}	$C\bar{S}$ ^{h}
400-450	$Mg(OH)_2$	MH
450-500	$Ca_3Al_2O_6{\cdot}CaSO_4{\cdot}12H_2O$ ^{d}	$C_3A{\cdot}C\bar{S}{\cdot}H_{12}$ ^{d}
	$Ca_3Al_2O_6{\cdot}6H_2O$ ^{d}	C_3AH_6 ^{d}
	$Ca(OH)_2$ ^{a}	CH ^{a}
	$Mg_6Al_2O_9{\cdot}CO_3{\cdot}12H_2O$	$M_6A\bar{C}H_{12}$
500-550	$Al(OH)_3$ $^{c, d}$	–
	$Al(OH)_3$ ^{i}	–
	$CaCO_3$ ^{j}	$C\bar{C}$ ^{j}
550-800	$CaCO_3$ ^{k}	$C\bar{C}$ ^{k}
	$CaCO_3$ ^{l}	$C\bar{C}$ ^{l}
	$MgCO_3$	$M\bar{C}$

Notes: a - highest rate of loss, b - major loss, c - gibbsite, d - minor loss, e - gibbsite to boehmite transition, f - bayerite, g - gibbsite to alumina transition, h - soluble to insoluble anhydrite, i - boehmite to alumina transition, j - calcite, k - vaterite, l - aragonite.

Table 2. Summary of main decomposition and phase change temperatures between 0 and 800°C. *(Continue on next page)*

Phase		Temperature (°C)	Reference
Formula	Cement chemist's nomenclature		
$CaSO_4{\cdot}2H_2O$	$C\bar{S}H_2$	70-200	[1]
	– „ –	128	[20]
	– „ –	120	[21]
	– „ –	140	[9]
	– „ –	140 and 170	[22]
	– „ –	142	[23]
	– „ –	145 and 165	[24]
	– „ –	150-157	[25]
	– „ –	159, 184	[26]
	– „ –	Varying	[27]
$CaSO_4{\cdot}½H_2O$	$C\bar{S}H_{0.5}$	150	[9]
	– „ –	173	[25]
	– „ –	200-220	[28]
	– „ –	> 200	[1]
CaSO4 ^{a}	$C\bar{S}$ ^{a}	380	[29]
CaSO4 ^{b}	$C\bar{S}$ ^{b}	< 1000	[30]
	– „ –	1462 ^{c}	[31]

Table 2. Summary of main decomposition and phase change temperatures between 0 and 800°C.

Phase		Temperature (°C)	Reference
Formula	Cement chemist's nomenclature		
–	C–S–H	50-600 ^{d}	[1]
	– „ –	100-125 ^{e}	[1]
	– „ –	111	[25]
	– „ –	115-125	[24]
	– „ –	130-140	[32]
	– „ –	< 200	[10]
$Ca_3Al_2O_6\cdot 3CaSO_4\cdot 26H_20$	$C_3A\cdot 3C\overline{S}\cdot H_{32}$	50-800 ^{d}	[1]
	– „ –	90-120 $^{e,\,f}$	[1]
	– „ –	125	[33]
	– „ –	125-132	[25]
	– „ –	~ 130	[22]
	– „ –	135-140	[24]
$Ca_3Al_2O_6\cdot CaSO_4\cdot 12H_2O$	$C_3A\cdot C\overline{S}\cdot H_{12}$	75-800 ^{d}	[1]
	– „ –	185-200	[24]
	– „ –	200 & 290 ^{f}, 440 & 480 ^{g}	[1]
–	C–A–H	90-120	[34]
	– „ –	100-120	[35]
$CaAl_2O_4\cdot 10H_2O$	CAH_{10}	100-160	[34]
	– „ –	130-150 ^{f}, 290 ^{g}	[1]
	– „ –	140	[35]
$Ca_2Al_2O_5\cdot 8H_2O$	C_2AH_8	140-200	[34]
	– „ –	170-180	[35]
$Ca_4Al_2O_7\cdot 13H_2O$	C_4AH_{13}	180-200	[34]
	– „ –	195-200	[32]
$Ca_2Al_2SiO_7\cdot 8H_2O$	C_2ASH_8	192	[34]
	– „ –	200	[36]
	– „ –	200-230	[37]
$Ca_3Al_2O_6\cdot 6H_2O$	C_3AH_6	200-250	[1]
	– „ –	250-310 ^{f}, 450-550 ^{g}	[38]
	– „ –	315-330	[32]
	– „ –	320	[35]
	– „ –	~ 330	[34]
$Al(OH)_3$ h	–	~ 230	[34]
	– „ –	220-230 $^{g,\,i}$, 310-325 $^{f,\,j}$, 495-525 $^{g,\,k}$	[39, 40]
	– „ –	~ 320	[40]
$Al(OH)_3$ ^{l}	–	280-300	[39]
$Mg(OH)_2$	MH	350	[20]
		~ 400	[41]
		415-430	[32]
$Ca(OH)_2$	CH	400-600 ^{d}	[1]
		450	[41]
		470 ^{b}, 450 ^{m}	[42]
		480 ^{e}	[1]
		492-528	[25]
		< 500	[22]
		500-600	[12]
		515-590	[32]
		530-550	[24]
$CaCO_3$	$C\overline{C}$	500-600 ^{n}	[1]
		500-700 ^{o}	[43]
		530-760 ^{p}	[44]
		> 800 ^{n}	[12]
		< 800 ^{n}	[22]
$CaCO_3$	$C\overline{C}$	650-660 ^{n}	[41]
		720-740 ^{n}	[42]
$MgCO_3$	MC	617	[41]
$Mg_6Al_2O_9\cdot CO_3\cdot 12H_2O$	$M_6A\overline{C}H_{12}$	50-230	[19]
		260-370	[19]
		375-650	[19]

Notes: a - Soluble to insoluble anhydrite, b - in air, c - melting temperature, d - Total loss, e - highest rate of loss, f - major loss, g - minor loss, h - gibbsite, i - gibbsite to boehmite transition, j - gibbsite to alumina transition, k - boehmite to alumina transition, l - bayerite, m - in argon, n - calcite, o - vaterite, p - aragonite.

CONCLUSSION

This paper provides a compilation of temperatures associated with decompositions and phase changes in the main cement hydrates within the temperature range 0 to 800°C. The use of thermal analysis techniques in identifying cement hydration products provides a powerful qualitative and quantitative tool. However, various experimental conditions, such as sample size, heating profile, heating environment and composition of sample pan, all influence the results obtained, so experimental error should be reduced by using consistent analysis conditions. Using different techniques may also yield slightly dissimilar results. Additionally, not all techniques will detect all phase transitions, for example TG only detects phase transitions with an associated mass loss and will not detect structural phase transitions or changes in symmetry in crystalline materials. Therefore, the choice of technique and experimental parameters must be considered carefully.

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