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A comparative study of hydration kinetics of different cements by thermogravimetry on calcined mass basis

Jo Dweck[1] · **Maura Berger Maltez Melchert**[1] · **Frank K. Cartledge**[2] · **Rosangela Silva Leonardo**[3] · **Romildo Dias Toledo Filho**[3]

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Abstract The capture of CO_2 and SO_2 from industrial gas effluents has been done usually by lime-containing products. For this purpose, cement pastes also can be used, due mainly to their calcium hydroxide content formed during hydration. To select the best cement for this purpose, TG and DTG curves of different Portland cement pastes (types I, II, III and G), prepared with a water-to-cement ratio (*W*/*C*) equal to 0.5, were analyzed at different ages, at same operating conditions. The curves were transformed into respective cement calcined and initial mass basis, to have a common and same composition reference basis, for a correct quantitative hydration data comparison. This procedure also shows that there is an unavoidable partial drying effect of the pastes before starting their analysis, which randomly decreases the *W*/*C* ratio at which were prepared, which indicates that, when results are compared on respective paste initial mass basis, assuming that the ratio *W*/*C* has not changed, possible calculation errors may be done. Type I, II and G analyzed cements have shown similar hydration characteristics as a function of time, while the analyzed type III cement has shown a different hydration behavior, mainly due to its highest Al_2O_3 and lowest SO_3 contents, promoting the formation of hydrated calcium aluminates, by the pozzolanic action of the excess of alumina, consuming $Ca(OH)_2$, which final content at 28 days was the lowest one, among the hydrated cements.

Keywords CO_2 and SO_2 capture · TG · DTG · Basis conversion · Portland cements · Hydration

Introduction

Portland cement, the basic material for concrete, is widely used in construction in various ways. The production of cement, besides consuming large amounts of non-renewable natural resources, is responsible for releasing to the atmosphere about one ton of carbon dioxide (CO_2) per ton of produced clinker, which greatly contributes to the greenhouse effect [1]. Cement production currently represents approximately 5% of the worldwide global release of CO_2 to the atmosphere [2].

Portland cement is a hydraulic binder, which is obtained after milling Portland clinker, with gypsum and other aggregates, the contents of which are regulated by government standards. Clinker, the main component of this mixture, which is present in all types of Portland cement, is produced from the thermal processing from ambient temperature to 1450–1500 °C of a mixture of raw materials that have as its main components, calcium (CaO), silicium

✉ Jo Dweck
jodweck@yahoo.com.br

Maura Berger Maltez Melchert
maura.melchert@ig.com.br

Frank K. Cartledge
fcartledge@lsu.edu

Rosangela Silva Leonardo
rosangela@coc.ufrj.br

Romildo Dias Toledo Filho
toledo@coc.ufrj.br

[1] Thermal Analysis Laboratory, Department of Inorganic Processes, School of Chemistry, Federal University of Rio de Janeiro, Rio de Janeiro, RJ, Brazil

[2] Chemistry Department, Louisiana State University, Baton Rouge, LA, USA

[3] Sustainable Materials Laboratory, Civil Engineering Department, COPPE, Federal University of Rio de Janeiro, Rio de Janeiro, RJ, Brazil

(SiO_2), aluminum (Al_2O_3) and iron (Fe_2O_3) oxides [3]. The main mineral components of clinker are: tricalcium silicate (3CaO·SiO2), dicalcium silicate (2CaO·SiO2), tricalcium aluminate (3CaO·Al2O3) and calcium ferrite aluminate (4CaO·Al2O3·Fe2O3), which in cement chemistry nomenclature are abbreviated, respectively, as C_3S, C_2S, C_3A and C_4AF [3]. Clinker must be rapidly cooled with air immediately after being formed, to avoid the reversible transformation of C_3S back to C_2S, which occurs during slow cooling. This reverse reaction decreases the level of the final mechanical resistance of hydrated cementitious materials, which is mainly promoted by tricalcium silicate hydrates (C–S–H).

Many kinds of aggregates may be added to form the blended compositions of Portland cement, defining the type of cement. These mineral additions, regulated by standard governmental specifications [4–8], are from natural or industrial sources, including, for example, blast-furnace slag, pozzolanic materials (fly ash, micro-silica and calcined clay) or limestone.

The composition of blended cements will define their hydration kinetics. But independently of the type, the main reactions that occur during Portland cement hydration are:

$$3CaO \cdot Al_2O_3 + 3(CaSO_4 \cdot 2H_2O) + 30H_2O \rightarrow \underset{\text{(ettringite)}}{3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O} \qquad (R-1)$$

$$2(3CaO \cdot SiO_2) + (6+x)H_2O \rightarrow \underset{\text{(tobermorite)}}{3CaO \cdot 2SiO_2 \cdot (3+x)H_2O} + \underset{\text{(calcium hydroxide)}}{3Ca(OH)_2} \qquad (R-2)$$

$$2(2CaO \cdot SiO_2) + (4+x)H_2O \rightarrow \underset{\text{(tobermorite)}}{3CaO \cdot 2SiO_2 \cdot (3+x)H_2O} + \underset{\text{(calcium hydroxide)}}{Ca(OH)_2} \qquad (R-3)$$

$$4CaO \cdot Al_2O_3 \cdot Fe_2O_3 + 2Ca(OH)_2 + 10H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 6H_2O + 3CaO \cdot Fe_2O_3 \cdot 4H_2O \qquad (R-4)$$

During the first hydration step, called the pre-induction period, reactions R-1 and R-2 occur. Right after this period, a second dormant or induction period occurs, during which the C_3S hydration reaction (R-2) rate slows down and may extend for a few hours. During the induction period ettringite, formed by reaction R-1 during the pre-induction period, begins to coat the external surfaces of the particles, retarding their hydration process. During the next acceleration period, when R-2 reaction rate increases significantly, setting begins due to the crystallization of the calcium hydroxide and tobermorite formed from C_3S hydration.

Final set usually occurs before the end of this acceleration period, during which a maximum temperature is attained by the reacting media, due to the exothermic effect of all hydration reactions. The fourth stage, called the post-acceleration period, is characterized by a deceleration of reaction rates, right after that maximum temperature was attained, and in this period there is a continuous slow formation of hydration products by diffusion-controlled reactions, including those formed from reactions R-3 and R-4 [9].

While C_3S hydration (reaction R-2) contributes mostly to the strength development during the first 4 weeks of setting, C_2S hydration (reaction R-3), which occurs in a much lower rate, contributes to the gain in strength from 4 weeks onwards. Reaction R-4, which depends on calcium hydroxide concentration, little contributes to the strength of the hardened paste.

As the contents of C_2S and C_3S are higher than those of other oxides in a cement composition, during hydration significant amounts of calcium hydroxide (CH) and tobermorite are simultaneously formed [10]. The high CH content promotes a high pH to the solidified cementitious materials from the early hydration stages onward and turns them into CO_2 absorbent materials [11–13].

Due mainly to their calcium hydroxide (CH) content formed during hydration, the hydrated cementitious materials can also be used for SO_2 sequestration. There are other factors that act in the capture efficiency, such as sorbent porosity, effluent gases temperature, humidity, CO_2 and/or SO_2 concentrations [11].

Depending on the desired final mechanical properties of the absorbing bodies after use, exposure to CO_2/SO_2 during the early stages of hydration is desirable to maintain the best mechanical conditions, whereas exposure during the more advanced hydration stages gives a higher degree of absorption [12] because of a greater CH content. For instance, when exposing a Portland cement paste during the first 24 h of hydration to a controlled 20% CO_2 ambient, this gas may consume all the formed calcium hydroxide and converts it into calcium carbonate.

This work is part of the research activities of a project of the authors studying CO_2 and SO_2 capture by cementitious materials. The main characterization data of the four commercial cements studied in this paper were published previously [14]. The objective of the present article is to compare their hydration kinetics by thermogravimetry (TG) and derivative thermogravimetry (DTG) on cement initial and calcined mass basis [15–18], to select the most effective cement(s) for the following steps of the research.

Materials and methods

Materials

In the present study, the following Brazilian Portland cements were used: type I (CPI), type II (CPII F-32), type

Table 1 Chemical composition of Portland cement types I, II, III and G/mass%

Cement types	Compounds															
	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O	TiO_2	Na_2O	SrO	P_2O_5	MnO	ZnO	BaO	CuO	LOI
CPI	64.02	16.38	3.10	4.04	5.14	1.46	0.84	0.20	0.20	0.09	0.14	0.01	0.07	0.02	0.02	4.27
CPII F-32	64.13	15.04	3.47	3.44	4.50	1.43	0.39	0.23	0.36	0.20	0.09	0.07	0.05	0.06	n.d.	6.54
CPIII	49.07	29.79	9.68	1.07	2.53	4.84	0.52	0.58	0.27	0.10	0.05	0.13	n.d.	n.d.	0.01	1.35
G Cement	63.60	18.10	3.40	5.50	3.90	2.10	0.49	0.23	0.36	0.19	0.10	0.09	0.07	0.00	0.06	1.75

LOI LOSS of ignition (1000 °C) by thermal analysis, *n.d.* non-detected

III (CPIII). Cement Class G (Cement G) was also used. The respective chemical compositions of those unhydrated cements obtained by X-ray fluorescence are shown in Table 1.

Methods

The pastes were prepared using a water/cement mass ratio (*W*/*C*) of 0.5 and were analyzed after different hydration times (1 and 24 h; 7, 14 and 28 days).

All samples were prepared and maintained into sealed small plastic bags until analysis at room temperature, where they were cured. Right after measuring the water mass (2.5 g) in a bag, the respective cement mass (5 g) previously measured in a plastic cup was poured into the plastic bag containing the water, which was sealed. Then, the components were hand mixed by external manual shearing action for about 30 s, obtaining a homogeneous flat paste. The bags were always maintained sealed at ambient temperature, except to be opened to remove a little piece for analysis, which was put into another plastic bag also maintained sealed to avoid possible carbonation, until taking from this second bag the sample for analysis. When the piece was in solid state, it was previously hand milled into this sealed bag.

Thermal analyses were performed in simultaneous TA Instruments TG/DSC Q600 equipment. Samples of about 10 mg were used in platinum pans in each analysis. Initially, all the samples were dried during an isothermal step at 35 °C for 1 h, to eliminate the residual non-combined free water [16]. Afterward, a constant heating rate of 10 °C min^{-1} was used from 35 to 1000 °C. 100 mL min^{-1} of N_2 purge gas flow was used in all steps.

Chemical composition analysis of the cement was performed by X-ray fluorescence, in AXIOS equipment.

TG and DTG curve conversion to a same composition basis

As will be detailed in the following paragraphs, to compare the differently aged pastes of a particular cement, the TG and DTG curves, obtained by default on each sample initial mass basis, cannot be used, because the initial masses of their samples do not have the same composition [19]. Thus, for a correct comparison, the TG and DTG curves have to be converted to a same composition reference mass basis. To show how this can be done, a 28-day paste prepared with Portland cement type II (CPII F-32) will be used as an example of the conversion of original TG and DTG curves to calcined and/or initial cement mass basis.

Figure 1 shows TG and DTG curves of the unhydrated cement type II sample and that of its paste after 28 days of hydration, on respective sample initial mass basis, as were obtained by default, using the software of the equipment.

The calcined mass of any hydrated paste composed only of cement and water, at the end of its thermogravimetric analysis, will have the same chemical composition as the respective cement calcined mass, when both are analyzed in the same operating conditions [15, 16]. Thus, the difference at the calcined mass conditions of the two cases shown in Fig. 1 is that numerically indicated in the respective TG curve.

In the 28-day paste calcined mass, we have 67 parts of the initial 100 parts of the paste mass, while in the case of the unhydrated cement, its calcined mass, which has the same composition, represents 93.46 parts of the 100 parts of the initial mass of cement.

So, if we multiply by 93.46/67.6 all the TG curve data of the 28-day paste, the calcined mass of the new curve, which, for all its points, maintains the same mass percentage changes with respect to its initial mass as a function of temperature, will also be representing 93.46% of the initial mass of cement in the paste, as shown in Fig. 2. At the same time, this conversion transforms the TG curve of the paste also on its initial cement mass basis, because the final calcined mass, which has the same composition as the final calcined mass of the cement, represents the same numerical percentage (93.46%) of the initial mass of the unhydrated analyzed cement (100%).

Doing so, we can see that the initial mass of the paste actually represents 138.2% of the initial cement mass used to prepare the paste, as indicated in Fig. 2. The DTG curve of the paste on initial cement basis is also shown, which was obtained by multiplying the original DTG curve on paste initial mass basis by the same constant.

This example also shows the importance of treating all the results on their respective cement initial basis (or on calcined

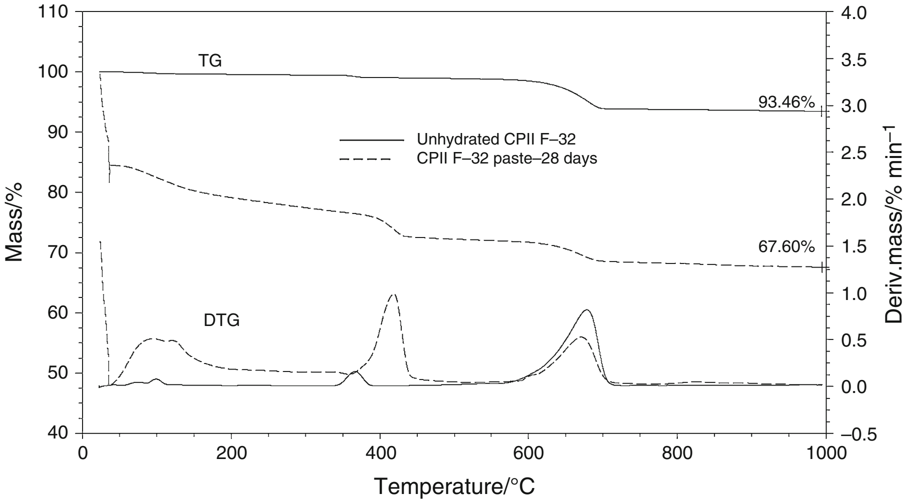

Fig. 1 TG and DTG curves of the unhydrated CPII F-32 and of its paste after 28 days of hydration, on respective initial sample mass basis

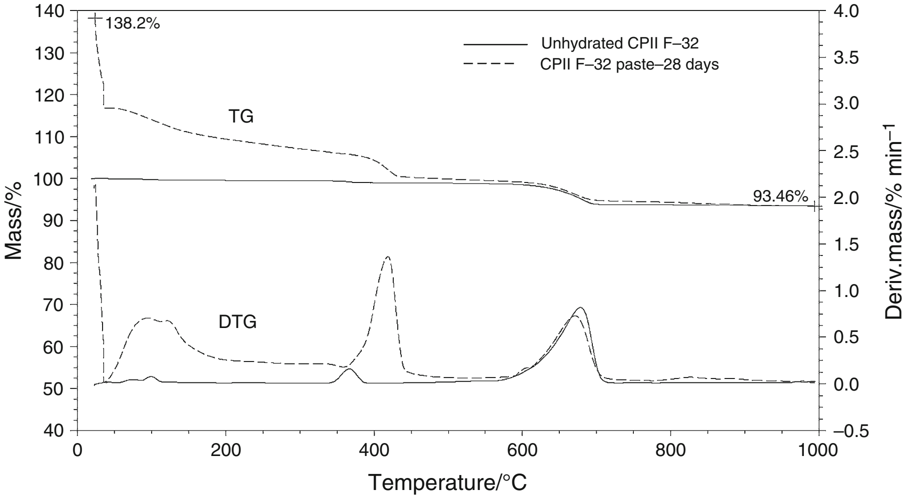

Fig. 2 TG and DTG curves of the unhydrated CPII F-32 and of its paste after 28 days of hydration, on respective cement initial mass basis

mass basis), and not on the basis of the initial mass of each sample, which may lose some free water during handling. As the paste was prepared with a *W*/*C* ratio equal to 0.5, the initial paste mass should be 150% of the initial mass of cement. But, as shown in Fig. 2, it is only 138.2%, indicating that the paste sample has lost some of its non-combined water due to some volatilization during handling and more importantly to the partial drying effect of the purge gas before initiating the analysis. Thus, the effective *W*/*C* mass ratio at the beginning of the analysis of the paste was 0.382 instead of 0.5.

Results and discussion

Effective initial *W*/*C* mass ratio at the beginning of the analysis

The conversion to the calcined and/or initial mass basis, besides allowing a correct comparison by using the same composition basis, avoids other calculation errors introduced by assuming that the initial analysis mass of the paste maintains the same *W*/*C* ratio with which it was prepared. The unavoidable drying effect of the purge gas occurs from the time that the sample is inserted in the equipment, up to the beginning of the analysis, when the operating control software registers the initial mass of the sample, as being the initial point of the TG curve and equal to 100% of the sample mass. In the present work, all TG/DTG curve comparisons among differently aged pastes were done using their respective cement initial mass. The unavoidable partial drying effect occurs in all hydrated cementitious materials analyses when there is free non-combined water, as shown in Table 2.

Considering (a) the method used to calculate Table 2 data described in the paragraph right before "Results and discussion" item; (b) the mean experimental measured error of our TG curves obtained during the last 5 years in our equipment, after calibration, has been of about 0.1% of the sample mass, within a temperature range of 1000 °C (for 10 mg initial sample mass as usually used); (c) the

Table 2 Initial effective *W*/*C* mass ratios on respective initial cement mass basis at the beginning of the performed thermogravimetric analyses

Time Cement	Hydration			
	24/h	7/days	14/days	28/days
CPI	0.415	0.423	0.407	0.396
CPII F-32	0.490	0.380	0.431	0.382
CPIII	0.468	0.386	0.390	0.422
G Cement	0.395	0.431	0.380	0.384

worst case among Table 2 data, which was an effective initial *W*/*C* mass ratio of 0.380, which comes from a TG curve measurement of 138% at the beginning of the respective thermogravimetric analysis on respective initial cement mass basis, the estimated experimental error we may have on the measured *W*/*C* values is of about $0.1 \times 100/38 = 0.26\%$.

To avoid any misunderstanding, it is important to note that during curing, as it was done in sealed bags, the *W*/*C* mass ratio was maintained equal to 0.5. The changes referred in Table 2 are due to the unavoidable losing only of some of the non-combined water present in the sample, which occurs during the transportation of the sample from the bag-sealed condition onto the base of the open crucible, putting the crucible on the holder and then submitting the sample to the drying action of the purge flowing gas before starting the analysis.

Thus, treating the thermogravimetric data on the initial cement mass basis, as described in this paper, allows correct quantitative comparisons, as done to study the different cements hydration evolution, because this basis of calculation does not depend on any change on initial *W*/*C* ratio, caused by the unavoidable random free water loss, which occurs in all cases, as shown before.

However, if the comparisons are done with TG data on each initial sample mass basis, considering that the initial masses of the pastes have a same *W*/*C* ratio, which is not actually what occurs, they will lead to erroneous quantitative conclusions, because they will be done and based on different initial mass compositions.

Cement hydration analysis

As a typical example of what was done with each cement type, Fig. 3 presents the TG and DTG curves of the unhydrated CPI and the CPI paste at different times of hydration, on the respective initial cement mass basis. Different combined water mass losses, presented in next figures, were calculated from TG curve mass losses, between respective transformation temperature limits indicated by respective DTG peaks. They represent the effective values of respective formed combined water, which were obtained subtracting from the total measured respective combined water from the TG curve of a paste, and the combined water released at the same temperature range and measured from the TG curve of the original unhydrated cement. The temperature ranges at which the free water and the different hydrated phases combined water were released are indicated as follows.

From TG curves, the mass loss up to the end of the isothermal step at 35 °C is due to the release of free water. The continuously lost mass between 35 and 200 °C refers to the combined water released from dehydration of tobermorite and ettringite phases, which increases with hydration time. It may be noted that during the first hours of hydration, the dehydration of the gypsum phase occurs simultaneously from 100 to 150 °C, when it is present [10]. It can be seen that for 24 h of CPI cement hydration, the gypsum was totally consumed, as occurred for the other cement pastes at the same age.

Between 200 and 350 °C, thermal decomposition from the other hydrated phases occurs, releasing respective water. From 350 to 500 °C occurs the dehydroxylation of calcium hydroxide releasing water the content of which increases significantly up to 7 days of hydration and is one of the main products of cement hydration. From 500 to 750 °C, the decomposition of calcium carbonate occurs, which was aggregated to the clinker during cement manufacture.

Figure 4 presents the water mass losses, between 35 and 200 °C, due to the dehydration of tobermorite and ettringite phases formed in the pastes of each kind of cement, as a function of time. It can be seen that CPII F-32 is the cement which mostly forms these phases, while CPIII forms the lowest content, releasing the lowest combined water from these phases, very probably because, as it has the lowest SO3 content, it forms the lowest ettringite content. After 7 days of hydration, for the other 3 cements the attained content remains practically stable, while for CPIII, it slightly increases up to the 14th day to decrease slowly afterward.

It must be noted that for CPII F-32 cement, at the early hydration periods, when tobermorite gel is initially being formed, it has more than 10 mols of combined water per mol. As hydration proceeds, the gel liberates the water to form other hydrated products, tending its combined water content to a constant value of 4 mols per mol of tobermorite, after 7 days [20]. From Fig. 4 results, it seems that cements I and G also do not change the combined water content of their tobermorite phases after the 7th day of hydration, whereas in case of cement type III, its tobermorite phase combined water has probably a different behavior, because other simultaneous hydration reaction(s) may be occurring, as will be further discussed.

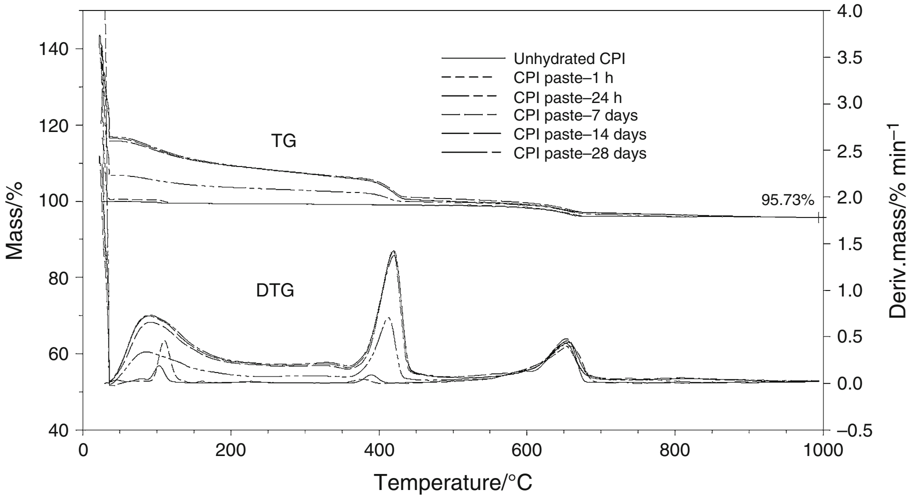

Fig. 3 TG and DTG curves of the unhydrated CPI and of its paste after 24 h and 7, 14 and 28 days of hydration, on respective cement initial mass basis

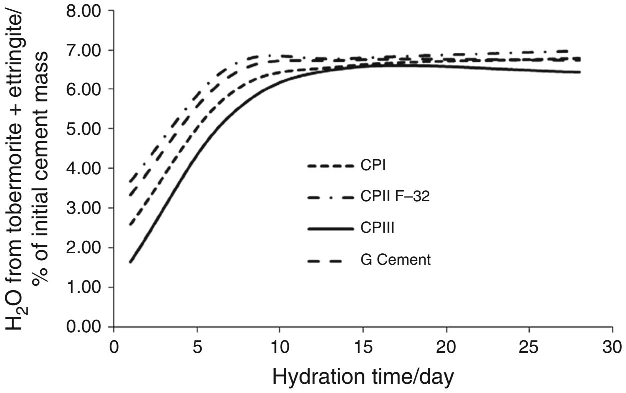

Fig. 4 Combined water released from tobermorite and ettringite phases of cement pastes at 24 h of hydration, on respective cement initial mass basis

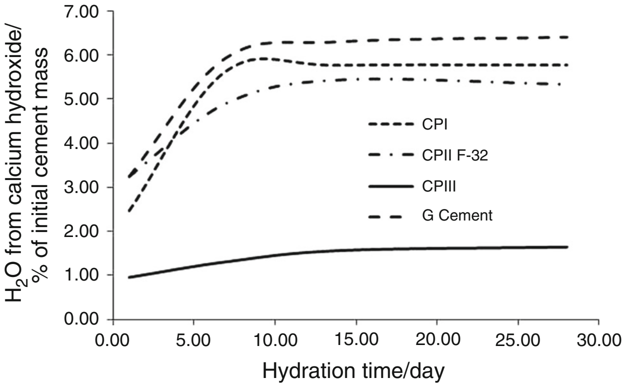

Fig. 5 Combined water released from calcium hydroxide from the hydrated cement pastes, on respective cement initial mass basis

Figure 5 shows the combined water released from calcium hydroxide dehydroxylation as a function of hydration time. It can be noticed that CPIII paste shows the lowest values in comparison with the other cement pastes, which is mostly due to its lower CH content, which seems to maintain a certain constancy after 14 days of hydration.

As indicated in Fig. 5, CPIII paste presents, in all ages, a much lower level of calcium hydroxide, when compared to the other cement pastes.

From XRF results in Table 1, it can be seen that its original Al_2O_3 content is more than twice higher than that of the other cements, while its SO_3 content is the lowest one. In this conditions, as there is no enough SO_3 (in the form of calcium sulfate or one of its hydrated salts) to form ettringite by reaction (R-1) during the first 24 h of hydration, consuming all present Al_2O_3 content, the non-reacted Al_2O_3, which is an amphoteric oxide, may form, by its pozzolanic property, calcium aluminate since the early stages of hydration, reacting with $Ca(OH)_2$, as this hydroxide is formed by tricalcium calcium silicates hydration in the same period.

This fact very probably explains CPIII lower content of calcium hydroxide in all ages, as measured in respective pastes and shown in Fig. 5. Consequently, the hydration of the formed calcium aluminate occurs, causing to this kind of cement type, its well-known high early strength properties promoted by the formed hydrated calcium aluminate phase.

To confirm the above hypothesis, it is important to note that Gruyaert et al. [21] in their recent study by calorimetry and thermogravimetry have also reported the enhancement of the hydration reactions and respective increase in total generated energy, when increasing the partial substitution of ordinary Portland cement (OPC) by blast-furnace slag, which is actually how Portland type III cements also called slag cements are manufactured.

Figure 6 presents the water mass losses from other hydrated phases as a function of hydration time. It is interesting to note that although during the first 7 days of hydration type III cement was the one which was losing the

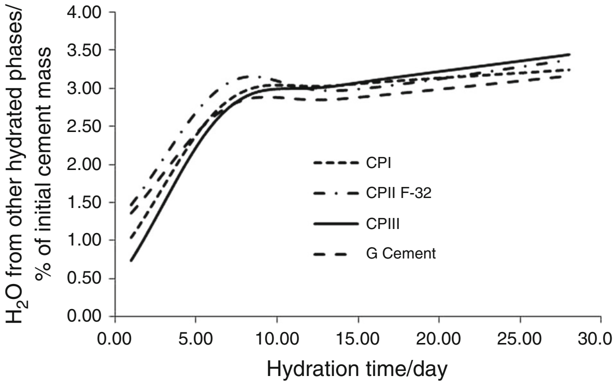

Fig. 6 Combined water released from other hydrated phases from cement pastes, as a function of hydration time, on respective cement initial mass basis

lowest combined water from other phases, this situation is inverted during the second week of hydration, being cement type III the one to have the highest content of combined water at the end of the 28 days of hydration.

This behavior indicates that very probably, the hydration of the excess of Al_2O_3 phase with respect to the lowest content of SO_3 forms the calcium aluminate, and then, the corresponding hydrated phase(s) seems to occur continuously but in a lower rate after 14 days of hydration, which explains the increasing behavior of the other phase combined water from the 14th to the 28th day of hydration, shown in Fig. 6.

It is important to note that calcium aluminate hydrates may decompose from 180 to 390 °C [22], confirming the previous occurrence, as well as indicating that in the case of cement III hydration, the increase in combined water loss during the first 14 days attributed so far to tobermorite and ettringite dehydration between 35 and 200 °C can also be due to the dehydration of a kind of calcium aluminate hydrate, which begins to decompose at 180 °C.

Figure 7 shows the total combined water released from cement pastes as a function of hydration time, on respective cement initial mass basis, which was obtained by the sum of the partial combined water losses shown in Figs. 4, 5 and 6. Due mainly to the very low content of formed calcium hydroxide, the lowest values are shown in case of cement type III, whereas for the other three cements, the highest total combined water values occur in case of cement type I.

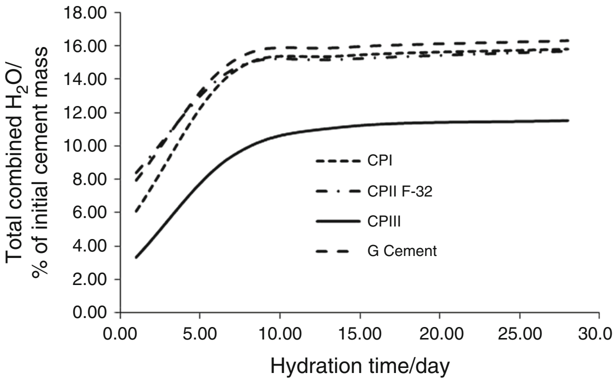

Fig. 7 Total combined water content released from cement pastes as a function of hydration time, on respective cement initial mass basis

Conclusions

- To have a correct quantitative comparison between a type of unhydrated cement phases and those of respective hydrated cement pastes, respective TG and DTG curves must be plotted on a same composition basis, which can be either, on respective cement calcined or initial mass basis.
- This procedure avoids having incorrect comparisons among differently aged pastes, when they are done on the paste sample initial mass basis, assuming that the initial paste samples have the same water/cement mass ratio as that used to prepare them. As proved in the present paper, due to the random and unavoidable free water evaporation during sample handling and by the purge gas partial drying action, before beginning the analysis, the initial *W*/*C* ratio of the initial paste mass is always lower than the ratio used to prepare it.
- The higher hydration kinetic differences among the different cements, which can be evaluated from the main formed phases combined water contents as a function of hydration time, occur during the first 7 days of hydration.
- In a general way, type I, II and G analyzed cements have shown similar hydration characteristics with respect to their similar main phase formation rates during the first 7 days, maintaining afterward respective hydration characteristics up to the 28-day analyzed period, although type I formed the highest calcium hydroxide content at the end of that period.
- The analyzed type III cement has shown a significantly different hydration behavior when compared to the other cements, mainly due to its highest Al_2O_3 and lowest SO_3 contents, promoting the formation of hydrated calcium aluminates, by the pozzolanic action of the excess of alumina, consuming $Ca(OH)_2$, which final content, at 28 days of hydration, was the lowest one among the hydrated cements.

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