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THE USE OF THERMAL ANALYSIS IN THE APPROXIMATE DETERMINATION OF THE CEMENT CONTENT IN CONCRETE

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Abstract

Thermal analysis was first used to investigate the pattern of dissociation of hydrated ordinary Portland cement. Portlandite ($Ca(OH)_2$) decomposes at about 500°C. This was confirmed by kinetic calculations. Thermal analysis was then performed to establish the effect of varying the cement content on the percent mass loss associated with the decomposition of $Ca(OH)_2$ in cement mortar cured for 28 days. An increasing relation was obtained. Standard concrete cubes were then prepared with cement contents ranging from 200 to 450 kg m^{-3}. The loss in mass on heating, up to 750°C, of concrete samples cured for 28 days was then related to the cement content in concrete. The relation obtained was tested for concrete cubes of known cement content and found to be in better agreement than the results obtained by conventional chemical analysis. This method can be used for an approximate determination of the cement content in concrete.

Keywords: cement content, concrete, thermal analysis

Introduction

The determination of the cement content in concrete made with different types of cement is usually accomplished through the chemical analysis. This is a tedious and elaborate technique that has been standardized [1]. The error involved in this method should not exceed 5%.

In the present paper a method based on the thermal decomposition of concrete, made of ordinary Portland cement and silicious aggregate, is presented. The thermal behaviour of the individual cement components and cement pastes have been investigated by several authors either calorimetrically [2] or by DTA [3] or combined DTA and TG [4–6]. An endotherm was reported in the range 450–600°C. Further peaks do not appear before 800°C. Most authors have agreed that this step corresponds to the decomposition of $Ca(OH)_2$ [5, 7]. Other authors have studied the

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effect of curing time on the intensity of this DTA peak. It was found that prolonged curing has an effect to the increase its intensity [8]. This was obviously due to the increase in the extent of hydration.

In the present paper, the effect of the amount of ordinary Portland cement (in a cement–sand mortar) on the percent loss in mass associated with the aforementioned peak was investigated. This was first done by using combined TG-DTG analysis. Following the results obtained in this step, concrete cubes, prepared with the same cement, were then heated in a muffle furnace and the total loss in mass was recorded as function of their cement content.

Experimental

The raw materials used in this work were: Normal Portland cement, provided by the Helwan company (south of Cairo) with a specific surface area of 2700 $cm^2\ g^{-1}$, sand (as fine aggregate), containing 0.029% chlorides and 0.03% sulphates. Its sieve analysis showed that 50% passed 35 mesh screen (0.417 mm) and 92% passed 6 mesh screen (3.327 mm) and gravel (as coarse aggregate), containing 0.013% chlorides and 0.02% sulphates. Its sieve analysis showed that 50% of this gravel passed 9.423 mm screen, while 88% passed 13.33 mm screen.

Thermal analysis was effected in a Shimadzu TG-50 H thermal analyzer at a heating rate of 10°C min^{-1}. Samples for thermal analysis were selected from a series of mortars consisting of cement and sand in predetermined ratios, to which 35% water (on a dry cement basis) was added. These mortars were water cured for 28 days before thermal analysis. The ratio of cement used in each mortar was calculated so as to correspond to a cement content varying from 200 to 500 kg m^{-3} concrete.

Concrete cubes, on the other hand, were molded as 200 mm cubes containing sand and gravel in the volumetric ratio 1:2 and with varying cement contents (200 to 450 kg m^{-3} concrete). The water level was kept at a 35% (based on dry cement content in the mortar). The cubes were water cured for 28 days. They were then crushed to fragments of about 10–40 mm, dried overnight at 110°C and weighed, then heated in a muffle furnace at an approximate heating rate of 10°C min^{-1} to 750°C. The fragments were left to cool inside the furnace down to about 150°C, then placed inside a dessicator to cool down to room temperature to prevent slaking and carbonation of calcium oxide; they were then weighed on a digital balance.

Results and discussion

Effect of cement content

Figure 1 shows the thermal TG-DTG curve obtained on heating a sample consisting of pure hydrated cement cured for 28 days. The water/cement ratio=0.35. Several losses appear on this curve. The first around 100–130°C corresponds to the evolution of physical as well as the decomposition of the nearly amorphous hydrates, mainly CSH as well as sulphoaluminate hydrates. The decomposition of these compounds continues progressively, as evidenced by the slow loss in mass between 130 and 500°C. A sharp loss then occurs corresponding to the decomposition of Portlandite ($Ca(OH)_2$). The activation energy of this decomposition step was calculated using the method suggested by Sabri *et al*. [9] that makes use of a combined TG-DTG trace at only one heating rate. The value of activation energy was found to equal 225 kJ mol^{-1} which is in good agreement with the value 240 kJ mol^{-1} reported by Nair *et al.* [10] for the decomposition of $Ca(OH)_2$.

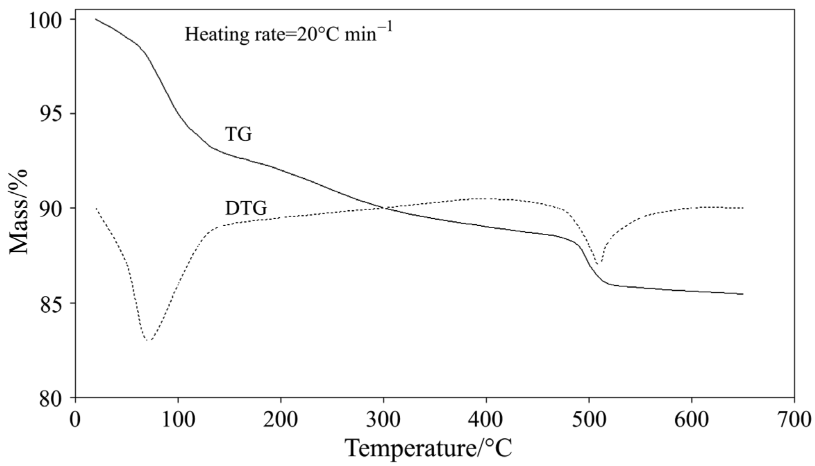

Fig. 1 Thermal analysis of a hydrated neat cement sample (*W/C*=0.35), cured for 28 days

As cement mortar samples were prepared using different amounts of cement (expressed as kg cement/cm^3 concrete), different traces were obtained. Figure 2 shows two of these traces corresponding to a minimum and a maximum cement content of 200 and 500 kg m^{-3} concrete, respectively. This figure shows that the extent of loss in mass above 500°C is higher in the latter than in the former case. The effect of varying the cement content on the percent loss in mass at 700°C is shown in Fig. 3. Two different set of points were obtained: the first curve represents the results obtained for a cement content <350 kg m^{-3} concrete, while the second curve shows the results when the cement content exceeds 350 kg m^{-3} concrete. This could be attributed to the change in the chemical composition of the hydrates formed upon increasing the cement content. Nevertheless, the percentage loss in mass is an increasing function in cement content.

This has led the authors to try to correlate the cement content of concrete cubes (water cured for 28 days) to the loss in mass as they were heated in a muffle furnace to 750°C. This relation is displayed in Fig. 4. Three cubes were tested each time.

To test the validity of this method and its accuracy, six blank concrete cubes were prepared with cement contents varying from 200 to 450 kg m^{-3} and water cured at room temperature for 28 days. The cement content of these concrete cubes was then determined by both chemical analysis and by mass loss using the relation in

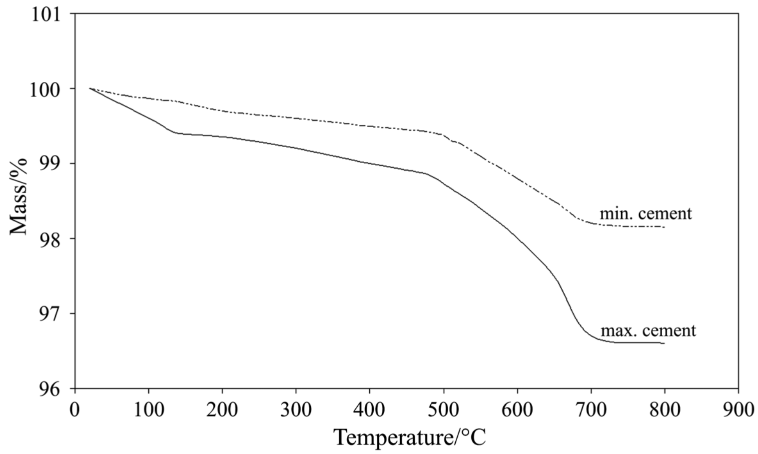

Fig. 2 TG curves for two cement mortar samples containing the equivalent of 200 kg m^{-3} (min. cement) and 500 kg m^{-3} cement (max. cement) used in concrete

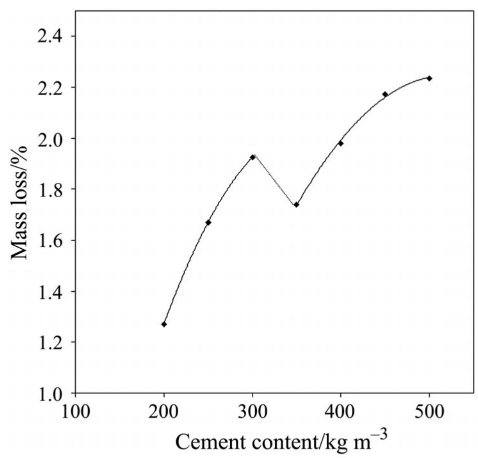

Fig. 3 Effect of cement content (equivalent to that used in concrete) on the percent mass loss of mortars at 700°C

Fig. 4. An empirical equation was deduced that relates the loss in mass at 750°C to the cement content of concrete x, in kg m^{-3} concrete:

$$\%\ \text{loss} = -10^{-5}x^2 + 0.0293x - 2.853$$

The results obtained by both methods (chemical analysis and loss in mass) were plotted on the same graph and a 45° line has been drawn to compare the results with the blank values. Figure 5 shows that the results obtained by both methods are comparable although those obtained by chemical analysis are more deviated from the 45° line than those obtained by thermal decomposition. The maximum deviation recorded was 15% in case of chemical analysis and 6% in case of thermal analysis.

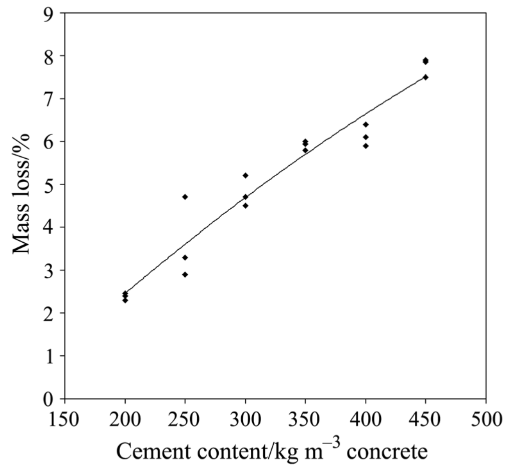

Fig. 4 Effect of the cement content on the percent loss in mass of cured concrete cubes crushed and heated up to 750°C

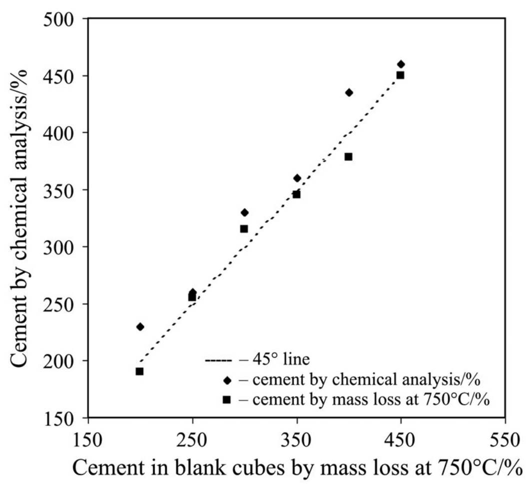

Fig. 5 Comparison between the percent cement calculated by chemical analysis and by mass loss at 750°C for blank standard cubes

Conclusions

An approximate method for the estimation of the cement content in concrete cubes cured for 28 days was elaborated, based on thermal analysis. This was based on the percent loss in mass at 750°C as evidenced by TG traces. A parabolic relation between cement content of concrete cubes and the percent loss in mass was obtained. This relation was used to estimate the cement content of six blank cubes and the results were compared with those of standard chemical analysis. The results were comparable although the proposed thermal method yielded better accuracy.

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