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# Influence of limestone filler on cement hydration and chemical shrinkage at early age

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## 1. Introduction

Self-compacting concrete (SCC) is a fluid concrete cast without vibration, whose one of benefits is to enhance of working conditions on site. This kind of concrete is achieved by modifying usual mixture proportion of ordinary (or vibrated) concrete. In addition to superplasticizer, high powders contents are needed to ensure both great flowability and good segregation resistance. In order to limit cost, powders are not only composed of cement but also of materials like limestone filler or fly ash. These constituents could be supposed non or low reactive at early age. However, literature review reveals that the hydration process of C<sub>3</sub>A and C<sub>3</sub>S, main components of clinker, is affected by the presence of limestone filler : hydration kinetic is accelerated, more heat is generated and hydrate composition is changed [1-3].

The present study aims at verifying the influence of a limestone filler on the hydration of different cements (CEM I and CEM II types), materials widely used in industrial SCC composition. The effect of limestone filler is investigated on cement pastes through thermogravimetric analysis, chemical shrinkage measurements and Vicat needle tests.

## 2. Experimental

### 2.1. Materials and composition of the cement pastes

The studied limestone filler is composed of 98,4% CaCO<sub>3</sub> and has a specific surface (Blaine) of 397 m<sup>2</sup>/kg. Different amounts of filler were mixed with two kinds of cement. The first one is a CEM II 42.5 R composed of 92% clinker and 6% CaCO<sub>3</sub>. The second cement is a CEM I 52.5 composed of 97% clinker and 3% CaCO<sub>3</sub>. In the cement pastes, the limestone filler was substituted for cement by mass and the water on cement ratio was kept constant (W/C=0.4). Table 1 summarizes the composition of the cement pastes and the associated filler/cement ratios (F/C).

Table 1 – Mass proportions of the cement pastes

	C : CEM II 42.5				C : CEM I 52.5		
Cement (C)	1	0.8	0.6	0.5	1	0.8	0.6
Limestone filler (F)	0	0.2	0.4	0.5	0	0.2	0.4
Water (W)	0.4	0.32	0.24	0.2	0.4	0.32	0.24
F/C	0	0.25	0.67	1	0	0.25	0.67
W/C	0.4	0.4	0.4	0.4	0.4	0.4	0.4

### 2.2. Experimental procedures

Cement hydration can be analyzed at micro-scale by determining composition and content of hydrates [4]. In this work, the hydration process was studied by thermogravimetric analysis (TGA). The hydration degree at time t is supposed to be correlated to  $W_n(t)/W_n(\infty)$ , ratio of the non-evaporable water mass at t and the non-evaporable water mass corresponding to the full hydration of cement.  $W_n(t)$  is defined as the mass loss, recorded during TGA test between 145 and 1050°C, minus the mass loss due to CO<sub>2</sub> releases produced by the calcite decomposition between 600 and 800°C.  $W_n(\infty)$  is estimated from the mineralogical composition of cement and the initial anhydrous cement mass of the sample. TGA was also used to measure the portlandite Ca(OH)<sub>2</sub> content in cement pastes.

Cement hydration can also be studied by measuring its effects at macro-scale [4]. In particular, hydration leads to a global contraction of the cement paste, called chemical shrinkage (or Le Chatelier

contraction), because hydrates are denser than reactives. Thereby, influence of limestone on hydration was investigated through measurements of chemical shrinkage. These measurements are carried out using a gravimetric method. After mixing, a paste sample is introduced into a flask, which is hung on a balance and immersed in a desaturated water bath. The amount of supply water penetrating into the flask is determined from the variation of the recorded mass  $\Delta M_s(t)$ . The chemical shrinkage, expressed in  $\text{mm}^3/\text{g}$  of anhydrous cement, is calculated as :  $\Delta V(t) = M_s(t)/(M_c \cdot \rho_w)$ , where  $M_c$  is the initial anhydrous cement mass and  $\rho_w$  is the water density. Another macroscopic effect of hydration is the setting : as the hydration degree increases, a solid structure develops inside the material. Setting of the studied pastes was determined using Vicat needle tests.

### 3- Results and discussion

For a given age below 1 day, the hydration degree from TGA increases with the limestone filler content of the paste, whatever the cement type is (Fig. 1-a and 1-b). The portlandite content follows the same tendency. This proves clearly the accelerating effect of limestone filler on cement hydration at early age.  $\text{CaCO}_3$  enhances the nucleation rate of cement components.

At macroscopic scale, limestone filler accelerates both chemical shrinkage kinetics and setting. In addition, amplitude of chemical shrinkage increases with F/C ratio. Thus, it seems that  $\text{CaCO}_3$  modifies not only hydration rate but hydrates composition.

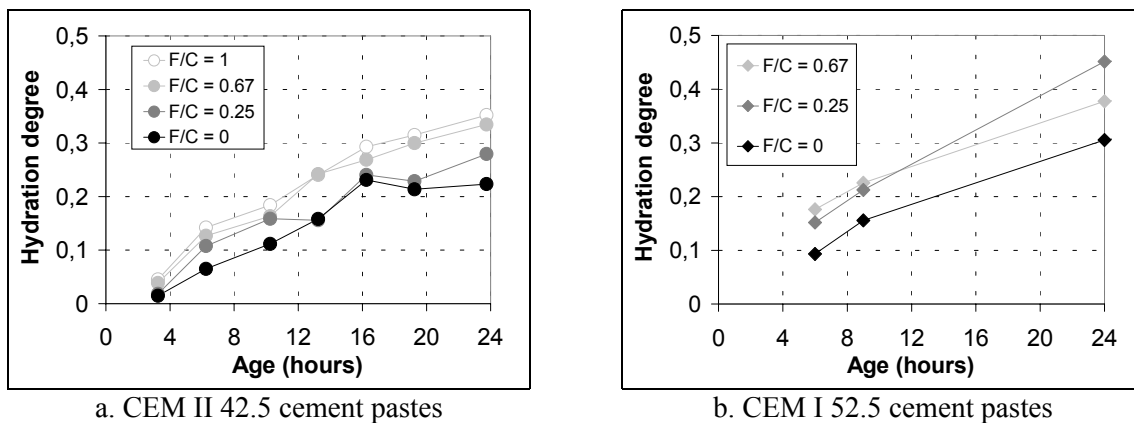


Fig. 1. Evolution of hydration degree for different filler on cement ratios F/C.

### 4- Conclusion

The wide use of limestone in SCC composition makes necessary all experimental data on its effects on material behavior. In this work, the accelerating effect of limestone on cement hydration at early age was shown at micro and macro-scale.

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