

# Using isothermal (heat conduction) calorimetry to study the effect of mixing intensity on reaction rate of cement mortars

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## Abstract

The method and intensity by which cement pastes, mortars and concretes are mixed is known to influence the rheology of the product. In the study presented here, isothermal (heat conduction) calorimetry has been used to evaluate the influence of mixing on the rate of hydration of cement mortars. It was found that both mixing procedure and mixing time influenced the hydration rate. It is probable that this is caused by the break up of cement grain aggregates during longer and/or more aggressive mixing. It is possible that the laboratory mixing methods in use do not imitate the intensity of field mixing. Another implication of this study is that extreme mixing intensity may be a method to activate or accelerate the hydration of, e.g., injection grouts.

## 1. Introduction

The method of mixing cement products with water will influence the rheological properties of the resulting fresh products. Roy and Asaga [1] found a decrease in viscosity with increased mixing intensity. They attributed this to that cement grain aggregates were broken down when the mixing was more intense. Similar results were also obtained by Jennings and coworkers [2,3]. The concept "mixing intensity" should be seen as being a function of shear rate in the mix and not to, e.g., the energy input rate to the mixer [4].

Yang and Jennings [2] noted that (laboratory) paddle mixing does not mimic the mixing of field concrete under many conditions. This is of concern as we want our laboratory methods to give results useful under field conditions. A study of the influence of mixing on the properties of the fresh cement paste fraction of for example mortars can therefore result in better laboratory mixing techniques. Another aspect of the relation between mixing and cement paste properties is

that it may be possible to adjust the properties of mortars, grouts and concretes by the way that they are mixed, for example by activating a product by intensive mixing.

In this study heat production rate (reaction rate) was measured by isothermal (heat conduction) calorimetry on mortars that were mixed by different methods.

## 2. Materials

The measurements were made on a cement mortar made of

- 2 parts Portland-limestone cement (CEM II/A-LL 42.5 R)
- 1 part deionized water
- 3 parts fine quartz sand (0-0.4 mm)
- 1% (of the cement mass) of a melamine based plasticizer.

## 3. Methods

Mixing was made by six methods numbered 1-6. The recipe sizes used for the different mixing methods are given in brackets.

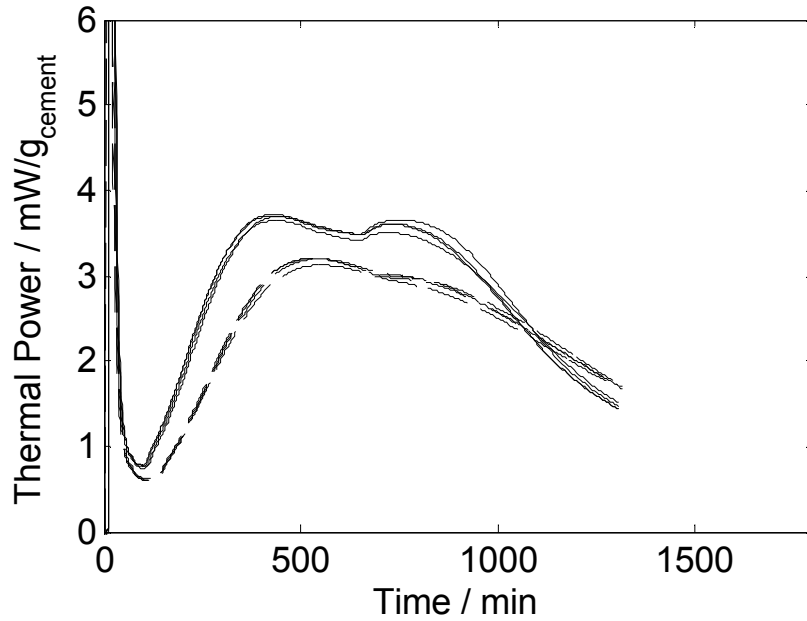
1. Hand mixing with a spoon for 30 s after all the cement is wetted (120 g).
2. Short mixing for 10 s with a test tube (vortex) mixer inside calorimetric ampoule (12 g).
3. Long mixing for 100 s with a test tube (vortex) mixer inside calorimetric ampoule (12 g).
4. Mixing 60 s with a hand-held household mixer ("cream mixer") (480 g).
5. High shear mixing for 60 s with a high speed blender (960 g).
6. Standard EN 196 mixing with a Hobart mixer (6000 g).

The calorimetric measurements were made at 20°C in a TAM Air isothermal (heat conduction) calorimeter (Thermometric AB, Järfälla, Sweden) with 20 ml polyethylene ampoules ("scintillation vials").

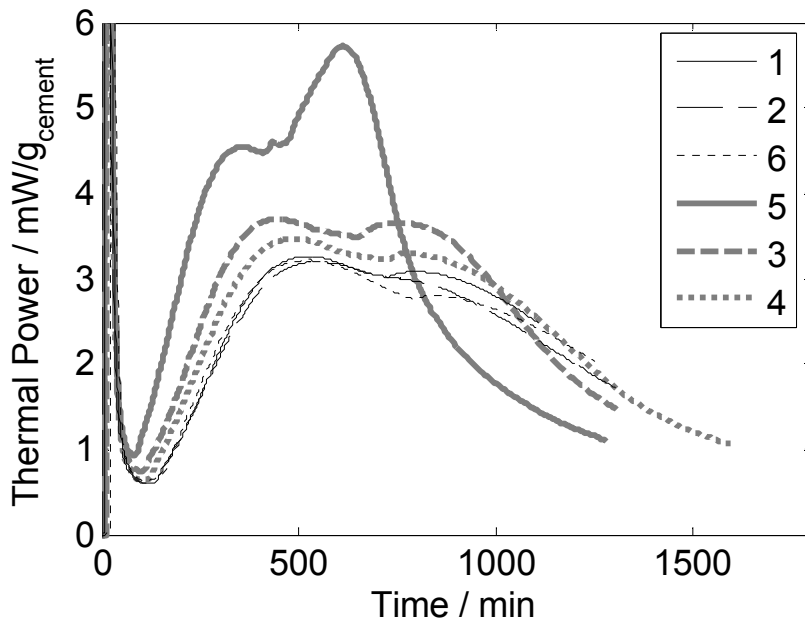
## 4. Results and discussion

Figure 1 shows the results for methods 2 and 3 (short and long vortex). It is seen that the reproducibility was good and that there is a clear difference in reaction rate between samples mixed for a short time (10 s) and those mixed for longer times (100 s). In Fig. 2, that gives one result for each mixing method, it is seen that the different methods of mixing gave quite different results. Method 5 (blender) gave the most intensive mixing resulting in a peak thermal power

(reaction rate) that was almost twice that of the other methods. It is also seen that the curve form for method 5 (blender) is different from those of the other methods.

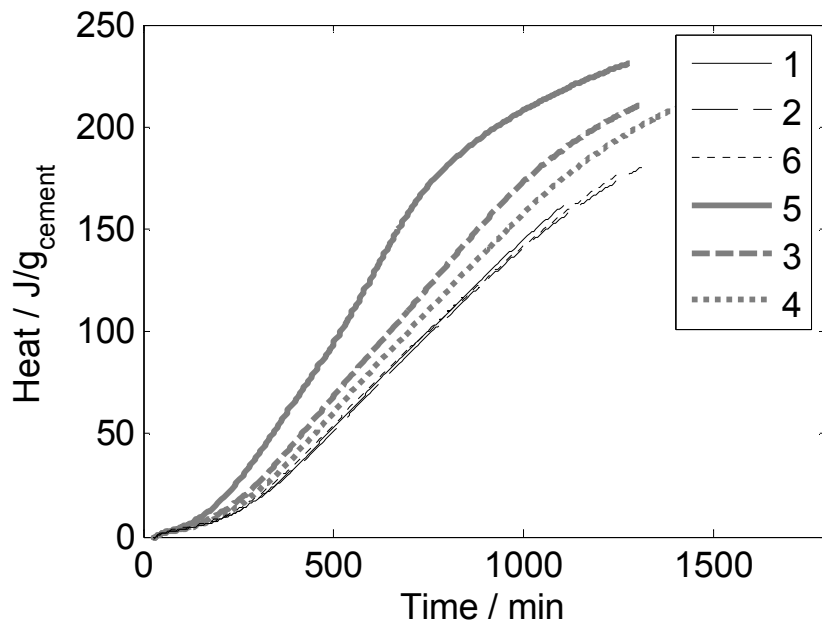


*Figure 1. Four samples mixed 10 s with a vortex mixer (dashed lines) and four samples mixed 100 s with a vortex mixer. All eight samples were measured at the same time, but were from eight individual mixes as they were mixed in the ampoules.*

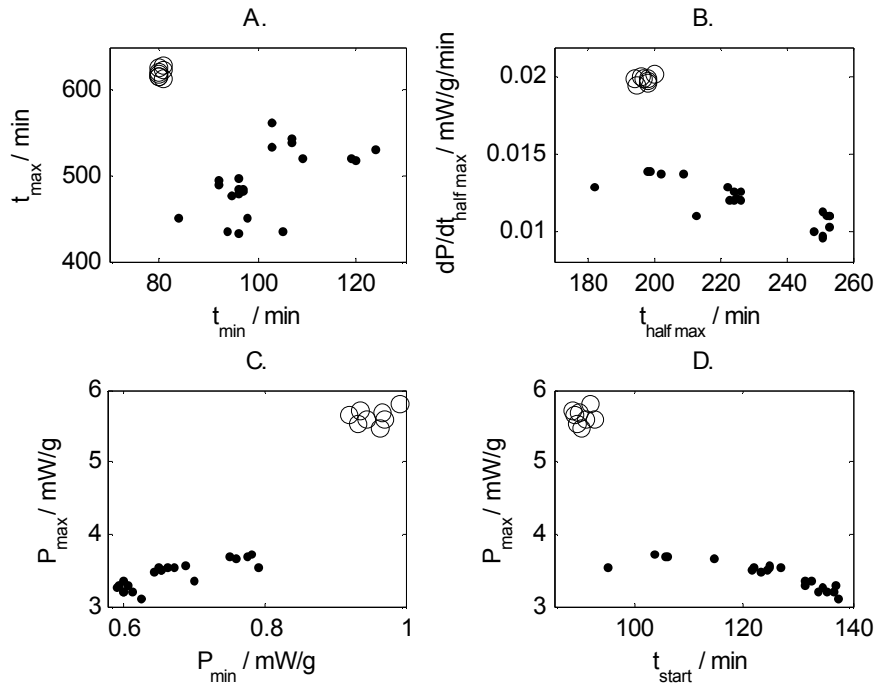


*Figure 2. One thermal power curve from each of the six mixing methods. Numbers given in the legend are the mixing methods.*

Figure 3 gives the integrals of the results shown in Fig. 2. Method 5 (blender) gave the highest heat during the 24 h measurements. This is an indication that method 5 also may have had a more rapid strength development, especially if the high reaction rate was caused by the breakdown of particle aggregates, as this may also give a more homogeneous hardened paste with less defects. Figure 2 and 3 indicates that mixing with method 5 gave quite different results compared to the other methods. I have therefore in Fig. 4 compared different parameters calculated from the thermal power curves to see if there is a qualitative difference between the curves.



*Figure 3. The heat produced by the samples. Calculated as integrals of the curves in Fig. 2 starting at 40 min. As the samples were charged into the calorimeter at different times, the present measurements did not give any reliable data for the early reactions. Therefore the integration started at 40 min during the dormant period with low thermal powers.*



*Figure 4. Relations between parameters calculated from the thermal power curves. The samples made with method 5 (blender) are shown as circles, all other results are shown as dots. A. Time to the main peak as a function of time of the lowest thermal power (during the dormant period). B. Rate of increase of thermal power at half the main peak thermal power as a function of the time to this point. C. Peak thermal power as a function of the lowest thermal power. D. Peak thermal power as a function of the end of the dormant period (start of the main reaction).*

It is seen in Fig. 4 that method 5 mixing gave results that does not follow the trends seen for the other results. It therefore seems that this very intense mixing method may have changed not only the reaction rate, but that also the course of the reactions (the chemistry) may have changed.

The above results are interesting both for the optimization of laboratory mixing methods and for the reaction rates of cement based products in the field. A continuation of this study should concentrate on comparing the intensity of laboratory mixing with that of field mixing with different equipment.

### **Acknowledgment**

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## References

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