ADIABATIC CALORIMETER FOR DETERMINING THE HYDRATION HEAT OF CEMENTS

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Some years ago, a semiadiabatic calorimeter was built at the Department of Building Materials, Technical University, Budapest. This instrument, suiting parallel measurements, was applied to determine the hydration heat of cements of different grades [1]. Nevertheless, to eliminate or at least minimize system errors, a new, adiabatic calorimeter has recently been developed, to be described in the following, together with the obtained cement hydration measurement results that will be compared to results obtained by the semiadiabatic method.

1. Principle of the adiabatic calorimeter

The adiabatic calorimeter determines the total heat released by the sample, theoretically without heat loss. Although this condition cannot be perfectly realized, heat exchange between sample and its surrounding can be minimized by various means. Partly, the reaction space has to be provided with the best thermal insulation possible, and partly, the outer surrounding has to be heated at the rate of development in the sample. Among others, KARS and SCHWIETE [2] applied this principle in their adiabatic calorimeter.

2. Built-up of the adiabatic calorimeter constructed at the Department

Scheme of the calorimeter is seen in Fig. 1.

The equipment is a double calorimeter. The sample with double insulation gets into the ultra-thermostat UT1: the mortar is cast in a brass cylinder that is put, in turn, into a food thermos lined with a PVC quilt, finally the complex gets a Nikecell cap. Hot junctions MT1 and MT2 of the control thermopile T1 and the thermometering thermocouple T2, resp., are inside the sample. Both the cold junction HT1 of the thermopile and the ohmic heater connected to the control unit immersion heater are immersed into the water bath of the ultrathermostat UT1.

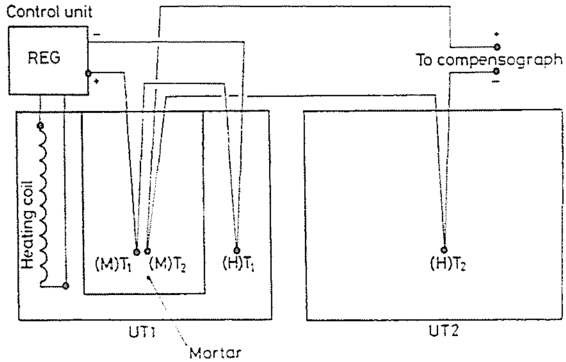

Fig. 1. Scheme of the adiabatic calorimeter. UT1, UT2: thermostats; MT1, HT1: hot and cold junctions of the thermopile of eight thermocouples; MT2, HT2: hot and cold junctions of a single thermocouple

The sample temperature is measured by thermocouple T2: its cold junction HT2 is immersed into ultrathermostat UT2 at constant temperature, while its hot junction MT2 is inside the sample. Free ends of thermocouple T2 are connected to a compensograph.

3. The thermopile T1

Thermopile T1 was made from copper and isothan wires ∅ 19 mm to produce control signals. After soldering, each of the eight cold and eight hot junctions were separately sealed with plastic, then fixed in plastic tubes of the required diameter.

4. The control unit

The control unit comprises a comparator IC with connected switches and amplifier, according to the circuit diagram in Fig. 2.

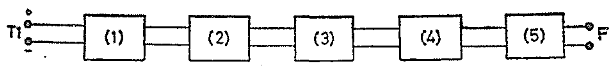

Fig. 2. Circuit diagram of the control unit. T1: Thermopile terminals; 1: Amplifier; 2: Comparator I C; 3: Transistor switch; 4: Reed-type relay; 5: Magnetic switch; 6: Ohmic heating

5. Calibration of the calorimeter

Adaptive heating of the environment may keep, or nearly, the sample's temperature. Nevertheless, to read off or calculate the total heat produced by unit mass of mortar during a time needs to know the heat quantity a given temperature on the compensograph diagram corresponds to. This equivalent has been obtained as follows:

Freshly mixed mortar was placed in a brass cylinder already containing an ohmic heater made of 2 m of constantan wire ∅ 0.8 mm. After six months when the setting could be considered as complete — at least from the aspect of hydration heat development — DC voltage was supplied to both terminals of the inner ohmic heater, producing a heat simulating the heat of hydration. In conformity with the expected temperature differences, the sample temperature was raised to max. 60 °C, instrumentally measuring the values involved (heating voltage, heating time, heating resistance).

Since the water bath temperature was controlled in the meantime by the control unit, it was at the desired temperature by the end of heating.

The mortar temperature started only to decrease 72 hours after the heating was off, conform to expectations, the mortar being already set.

6. The mortar

Composition of the mortar sample was the same as in earlier tests (100 g of water, 200 g of cement, 800 g of sand) [1].

7. Measurement results and evaluation

Hydration heat values of four different cements are seen in Fig. 3, indicating curves both calculated from semiadiabatic values and obtained by adiabatic measurements.

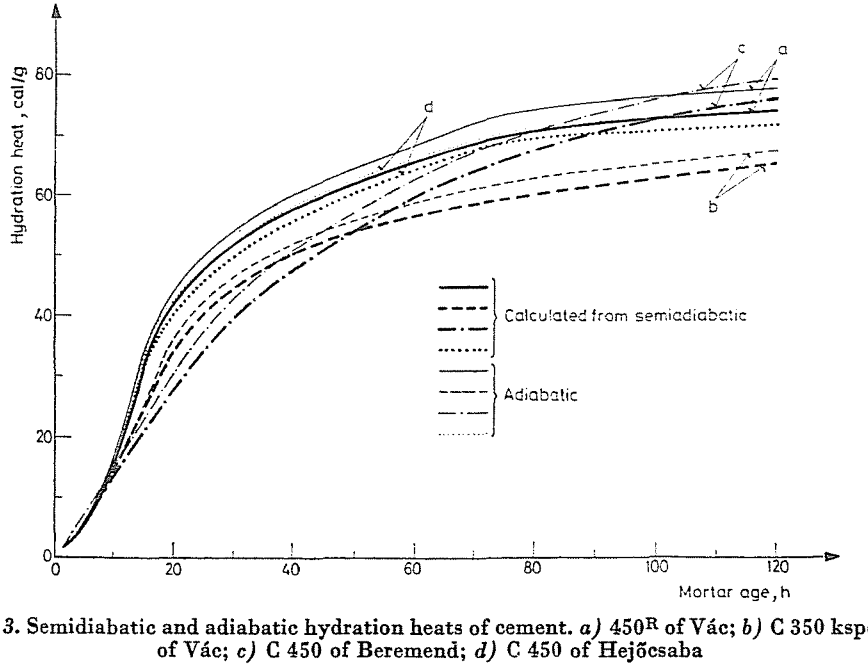

Fig. 3. **Semidiabatic and adiabatic hydration heats of cement.** ***a)*** **450**^{R} **of Vác;** ***b)*** **C 350 kspc-20 of Vác;** ***c)*** **C 450 of Beremend;** ***d)*** **C 450 of Hejőcsaba**

Curves for different cements are seen to be of rather similar shape. At the beginning of setting, adiabatic values are equal to the semiadiabatic ones. at a later stage of setting, however, about the tenth hour, the respective adiabatic values are higher by 1 to 4 cal/g.

Summary

To measure hydration heat of cements, an adiabatic calorimeter was constructed at the Department of Building Materials.

Development of hydration heat of four different cement samples has been recorded. Both the curves and observations show adiabatic hydration heat values to be always somewhat higher than the respective values calculated from semiadiabatic measurements.

References

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2. KARSCH, K. H.—SCHWIETE: Adiabatischer Kalorimeter zur Bestimmung der Hydrationswärme von Zement. Zement, Kalk, Gips, H. 4. 1963. S. 165—1969.

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* In Hungarian