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Research Article

Design and Characterization of a Semi-Adiabatic Calorimeter

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Abstract: The heat generation characteristics of cements can vary not only by type but also by manufacturer and mining location. Therefore, accurate measurement and modelling of cement hydration heat are essential to minimize the risk of cracking arising from temperature differences and deformations caused by temperature changes. This paper presents a uniquely developed calorimeter equipped with a multi-channel data acquisition system which is also suitable for the standard measurement of cement hydration heat. In this article the calibration process of the digital thermometer was described, as well as the determination of heat loss and its compensation to achieve an adiabatic environment.

Keywords: mass concrete; calorimeter; heat of hydration; cement

I. INTRODUCTION

In the early setting phase of mass concrete used in structural engineering, chemical processes occurring during cement hydration can lead to cracking in the structure. The volume change caused by cement hydration heat can significantly reduce the performance of the hardened concrete. Cements with similar properties may exhibit different thermal characteristics depending on the manufacturer and mining location, which must be considered during concrete design.

One of the most common methods used to prevent crack formation is to examine the exothermic reaction of cement during setting with a calorimeter, and then use these data as input for, e.g., finite element models [1, 2].

Chang-keun Lim et al. in [3] pointed out to the risk of cracking can be determined cost-effectively using finite element modelling. In their paper, they use adiabatic and semi-adiabatic models to investigate the cracking susceptibility of concrete.

Gibbon et al. [4] studied the heat generation process of concrete through a series of laboratory measurements using a custom-developed calorimeter. The obtained results were then applied as input for a finite element model.

The heat of hydration is a characteristic datum for every cement type, and its measurement is carried out using a calorimeter. Several research efforts have focused on identifying suitable equipment for this investigation, which is also regulated by standards. According to [5].one of the most widespread solutions is the use of the so-called coffee cup calorimeter.

J. Young in [6] highlights for the significance of the temperature compensation model for the coffee cup calorimeter and the influencing effect of the thermal properties of the insulation in a case of semi-adiabatic calorimetry. Furthermore, Radel and Navidi [7] emphasize in their work that the insulation of custom-developed "coffee cup" type calorimeters is adequate for conducting appropriate laboratory measurements. Hill and Petrucci [8] also state this, describing that styrofoam insulation causes very little heat loss.

On an international level, the ASTM C1702-17 standard [9] is a widely recognized framework for measuring the heat of hydration using isothermal conduction calorimetry, providing high-resolution data on the hydration kinetics of cementitious materials at a constant temperature. Complementing this, in Hungary, the investigation of cement hydration heat and the requirements for the equipment are described by the MSZ EN 196-8:2010 standard and MSZ EN 196-9:2010 standards [10,11]. These standard focuses on the semi-adiabatic method and defines the characteristics of the associated equipment. For examining the hydration process of cement-based systems, especially cement mortars, accurate monitoring of heat evolution is essential. Both the ASTM and the MSZ EN standards highlight the role of heat-related curves in characterizing the setting process, thus giving calorimetric investigations a key role in material qualification and development.

Justification for the Selection and Unique Development of a Semi-Adiabatic Calorimeter

Accurate temperature control of the measurement system is crucial when investigating cement hydration, as the results are significantly temperature dependent. Pang et al. [12] compared two methods for characterizing Portland cement hydration: chemical shrinkage and isothermal calorimetry. In their study pointed out that at the same temperature, both methods yield well-coordinated results; however, the ratio of heat released during hydration to chemical shrinkage is strongly influenced by temperature. At 25°C, this value is 7,500–8,000 J/mL, increasing by approximately 58 J/mL/°C with a rise in temperature. It indicates that correcting for measurement inaccuracies during data processing is particularly important. The scaling model applied by the researchers provides an opportunity for post-correction of these discrepancies, thereby increasing the comparability of different measurement systems and the reliability of the investigations. The scaling model applied by the researchers provides an opportunity for post-correction of these discrepancies, thereby increasing the comparability of different measurement systems and the reliability of the investigations.

Semi-adiabatic calorimeters represent compromise solution that – compared to fully adiabatic systems – is simpler and more cost-effective, while significantly reducing heat loss to the environment. This is particularly advantageous when examining cement-based materials, where stable environmental conditions are needed for reliable measurement of slow heat evolution [1]. The semi-adiabatic system actively tracks the sample's temperature and controls the external environment's temperature accordingly, thereby minimizing heat fluxes resulting from temperature gradients.

The hydration curve of cement mortars sensitively reflects changes in composition, chemical admixtures, or external conditions [14]. Therefore, it is essential to use a measurement system that:

- ensures minimal heat loss,
- allows for high-resolution measurement of temperature changes,
- capable of adapting to the geometric peculiarities of the samples.

Since commercially available semi-adiabatic calorimeters did not perfectly fit the specific needs for testing cement mortars (e.g., sample size, mass, shape peculiarities, handling aspects), it became necessary to develop a custom-designed, optimized calorimeter. This device allows for standard-compliant testing while providing reliable, reproducible measurement data for various cement compositions [9].

Calorimetric Methods for Measuring Cement Hydration Heat

The determination of cement hydration heat plays a crucial role in understanding material behaviour, especially when predicting early-age hardening and the risk of crack formation. The amount and time-course of heat released during hydration provide information about the kinetics of cement reactions, as well as the effects of aggregates and admixtures.

The most common method for investigating cement heat evolution is calorimetry, which is based on measuring the heat emitted or absorbed by a system. These methods can fundamentally be categorized into two groups: isothermal and adiabatic calorimetry.

Isothermal Calorimetry

Isothermal calorimeters maintain the sample at a constant temperature while continuously measuring the heat flow per unit time. This method provides high-resolution data on the initial stages of the hydration reaction and is particularly suitable for investigating the kinetic effects of admixtures [13, 15]. However, a drawback is that due to the artificial temperature control, the results do not accurately reflect the heat processes occurring in real-world agricultural or industrial environments.

Adiabatic and Semi-Adiabatic Calorimetry

The aim of the using of adiabatic calorimeters to naturally follow temperature changes due to heat generated within the system, with minimal heat loss. The temperatures of the environment and the sample are kept at nearly the same level, so the measured temperature change is directly proportional to the generated heat [14].

Semi-adiabatic measuring systems operate on a similar principle, but due to their simpler technical design where the insulation is not perfect. For this reason, the external environment electronically tracks the sample's temperature. This method is widely used, for example, for studying the hydration of cement mortars based on the MSZ EN 196-9 standard. The advantage of this method is its ability to more realistically depict the heat evolution of cements in large-volume structures, and it is also highly reproducible under laboratory conditions [12].

Coffee Cup Calorimeters

Among the simplest calorimeters are the so-called "coffee cup" type devices, which primarily serve 56educational purposes and are suitable for quick, qualitative measurements. These are typically non-adiabatic, open-system devices where heat loss is relatively high, and the sample actively exchanges heat with its surroundings. Coffee cup type calorimeters are popular in industrial and on-site cement testing due to their simplicity and low cost, especially for preliminary, rapid assessments. The CP Tech Center's development demonstrated a simple and rapid calorimetric measurement system suitable for monitoring heat evolution in concrete mixes even under field conditions [16]. A similar isoperibol system was also used by Šiler et al. when investigating the effect of various admixtures – such as zinc ions and fly ash – on cement hydration [17]. Although these devices allow for the detection of heat evolution trends and the observation of hydration process stages, their measurement accuracy is limited. Due to heat loss and temperature fluctuations, the measured data are often not entirely reliable, especially if the goal is to determine quantitative, reproducible heat amounts. Furthermore, the effect of external temperature and poor system insulation can also influence the results. Consequently, coffee cup calorimeters are recommended for qualitative rather than quantitative purposes and do not replace more precise calorimetric methods performed under laboratory conditions.

Comparison of Calorimetric Methods

Isothermal Calorimetry

Isothermal calorimeters keep the sample at a constant temperature and measure the heat flow released during the reaction. It is a very precise and sensitive method, mainly suitable for investigating reaction kinetics, especially during the first 72 hours. It is excellent for comparing fast initial reactions, various cement types, and admixtures [15].

- Advantage: Detailed, temporal resolution.
- Disadvantage: Does not accurately represent real-world conditions (e.g., heat evolution in structures).

Adiabatic Calorimetry

In this method the sample has been completely thermally insulated from its environment, so all heat generated in the sample increases its temperature. This is particularly useful for modelling mass concrete structures [13]. However, the technical background required for adiabatic conditions is complex and costly.

- Advantage: Accurately mimics real construction industry conditions; allows for more precise measurements.
- Disadvantage: Expensive and difficult to operate.

Semi-Adiabatic Calorimetry

Semi-adiabatic methods offer a compromise: the temperature of the sample depends on the environmental temperature because the insulation technology. The using of the calorimeter heat loss compensation model which has been defined with the help of the system geometry, the material properties and the thermal characteristic are highly usable for real applications, e.g., for testing cement mortars [14].

- Advantage: Simpler, more cost-effective, accurately models real heat evolution.
- Disadvantage: Less precise for total heat quantity due to heat losses.

Coffee Cup Calorimeter

This is a quite simple, non-adiabatic calorimeter used for educational purposes, where the sample exchanges heat with the environment. For cement, it is only suitable for investigating short-term, rapid reactions, such as preliminary reactions [16, 17].

- Advantage: Simple, inexpensive.
- Disadvantage: High heat loss, inaccurate, not suitable for quantitative description of cement hydration.

Based on the requirements of the related literature, the following design criteria were established for the developed calorimeter:

- Accurate monitoring of the temperature between 20 °C and 100°C with predefined sampling time,
- Compatibility with the standard measurement defined mortar geometry,
- Sufficient insulation to maintain semi-adiabatic conditions,
- Simple design with precise measurement and customisable data acquisition system,
- Can be used for automatic long term data acquisition.

II. EXPERIMENTAL SETUP

The calorimeter designed for measuring the heat of cement hydration consists of a thermally insulated container equipped with connected temperature measurement and data acquisition units, and a vapor-proof and thermally insulating sealing element.

Design of the Thermally Insulated Container

The thermally insulated container comprises an inner mortar container for sample storage and surrounding thermal insulation layers, which serve to minimize heat loss. A crucial aspect for the mortar container is its reusability, as the fresh mortar sample solidifies by the end of the measurement.

Another primary requirement for the mortar container is to ensure water tightness, as water loss significantly influences the setting processes. Based on these expectations, we opted for a cylindrical design with a rotational body geometry.

The sample preparation followed the MSZ standard, which specifies the use of standard sand, cement, and deionized water. The quantities of the mixture required for the measurement are provided in **Table 1.**

Table 1. *Mortar mixture according to EN 196-1 standard*

Material	*Mass [g]*
cement	360±0,5
Standard sand	1080±1
Deionized water	180±0.5
Total	1620

Since the materials placed in the mixing vessel cannot be completely recovered, the amount of mortar prepared must exceed the 1575 g required for the test.

The standard sand quantity is 1500 g, with a given proportion of fractions, so the volume of the mortar container had to be adjusted to accommodate this amount. A mixture with a mass of 1575 ± 1 g is needed for the measurement; the quantities of raw materials adjusted accordingly are shown in **Table 2**.

Table 2. *Proportional recalculation of raw materials*

Material	***Quantity prescr. by standard [g]***	***Quantity relative to standard sand [g]***	***Quantity in the mixture [g]***
Cement	360±0.5	500	350
Standard sand	1080±1	1500	1050
Deionized water	180±0.5	250	175
Total	1620	2250	1575

The volumes calculated from the obtained masses and the densities of the materials are summarized in **Table 3**.

Table 3. *Density of the ingredients*

Material	***Quantity prescr. by standard [g]***	***Density [g/cm^3]***	***Volume [cm^3]***
Cement	350	1.50	230
Standard sand	1050	1.55	670
Deionized water	175	1.00	175
Total	1575	-	1075

Due to capillary effects, the volume is expected to be smaller after mixing. To avoid errors arising from calculations and rounding, the required volume was determined with a safety factor of 1.2. Thus, the necessary minimum volume for the mortar container was set to 1300 cm^3.

Theoretically, the shape of the mortar container does not have a significant influence on the measurement, but the temperature measurement must be conducted at the centre of gravity of the mortar body. Therefore, we made the height of the cylinder is similar to the diameter of its base, then determined the recommended geometry.

Based on the results, a PVC pipe with an inner diameter of 110 mm and a height of 130 mm was chosen. Both ends of the PVC pipe were sealed with their corresponding caps.

The lower end cap was water-tightly secured with construction adhesive. The upper end cap features an 8 mm hole through which the temperature sensor can be inserted. A plastic tube – essentially a straw – fitting into the 8 mm hole facilitated the insertion of the temperature sensor into the mortar sample and allowed for its recovery after the measurement from the hardened mortar. To ensure more accurate temperature measurement, the straw was filled with deionized water to provide thermal conduction.

For the upper cap and the straw, we used a water-tight plastic sealant to prevent the mixing water from escaping the container.

The final construction of the mortar container is shown in **Fig. 1**.

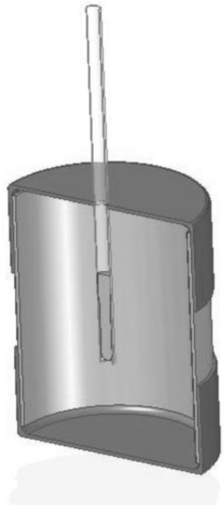

Fig 1 *Design of the mortar container*

The design of thermal insulation

When selecting the material for thermal insulation, the material's thermal conductivity was the most characteristic datum, followed by workability and economic viability as guiding principles. Foam insulation materials are primarily manufactured as expanded or extruded types. The thermal conductivity of Expanded Polystyrene (EPS) foam and Extruded Polystyrene (XPS) foam is nearly identical, at λ=0,038 W/(mK). Both materials proved suitable for designing the outer insulating container. The workability of impact-resistant expanded insulation board (EPS) is excellent. EPS boards are available in various thicknesses; for this construction the 50 mm thick EPS was used.

The geometry of the container also adopted a cylindrical design, capable of accommodating the mortar container. The outer container has an external diameter of 330 mm, and a height of 350 mm. Commercially available EPS boards have dimensions of 500×1000 mm, and the cutting plan is illustrated in **Fig. 2**.

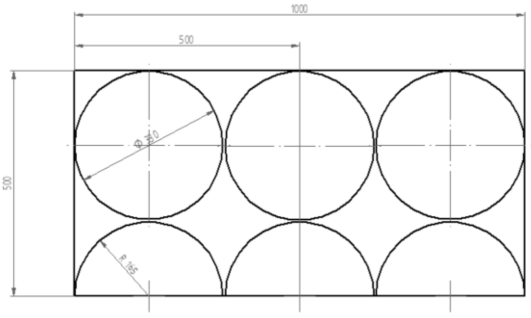

Fig 2. *EPS Board Cutting Plan*

The guiding principle for the final design of the outer thermal insulation container was to ensure the mortar container was enveloped by at least 100 mm of insulation material in every direction to achieve a semi-adiabatic system.

The resulting final construction of the calorimeter is illustrated in **Fig. 3**.

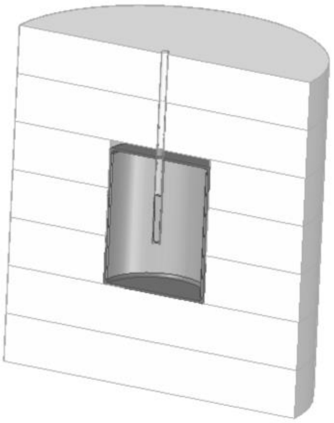

Fig 3. *The cross section of the calorimeter*

Temperature Measurement

According to the standard, temperature measurement requires a sensor with a temperature range of at least 19°C to 75°C and it must be waterproof. PT100 and PT1000 platinum resistance thermometers satisfy this requirement. However, in recent times, the use of digital temperature sensors has become increasingly widespread. These sensors are often cheaper, and multiple thermocouples can be connected to a single input, as each query sends the sensor's manufacturing ID along with a CRC code to verify the integrity of the temperature and signal transmission.

For the measurement the thermocouple was inserted from above into the centre of the sample. For the measurement, we used a DS18B20 thermocouple. Its characteristics are summarized in **Table 4**.

Table 4. *properties of the thermocouple*

	PT100	***DS18B20***
Measuring Range	–50ºC … +250ºC	-55…125°C
Accuracy	depends to the calibration	- Between -10°C and +85°C: ± 0.5°C
Design	3-wire, stainless steel sleeve, waterproof	Waterproof stainless-steel sleeve
Size	6 x 50 mm	6 x 50 mm
Voltage range	-	3...5.5V

The chosen thermocouple sends temperature values, its factory identification number, and the CRC code of the transmitted data to the data acquisition unit in digital form. Based on **Table 4.**, its accuracy is ± 0.5°C, which, according to [7], is insufficient without calibration. Due to digital signal transmission, the thermocouple cannot be directly calibrated to a specific temperature or range of temperatures, thus requiring software-based calibration.

Fig.4. shows the electrical circuit of the Thermocouple connected to the Arduino.

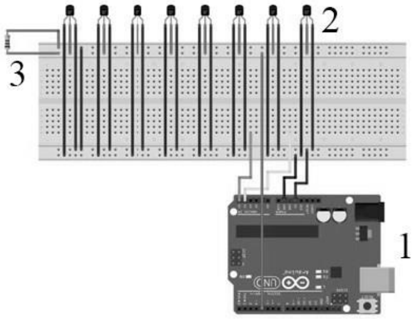

Figure 4. *Electrical circuit of the Thermocouple connected to the Arduino*

The pin num. 5 of the Arduino Uno controller (1) collects signals from the thermocouples (2). The thermocouples are connected in parallel in a so-called one-wire configuration. Data retrieval is performed based on the serial number of the thermocouple. The thermocouples receive their 5V power supply from the board. The data pin of the 8 thermocouples and the +5V supply voltage were connected in parallel with a 4.7 kOhm resistor (3), as internal resistance of the board is insufficient for this many sensors in parasitic power mode.

The implementation of tasks of the data loggers was achieved using a Matlab-Arduino interface. With the help of microprocessor and sensor support packages, the number of measurements was defined, measurement duration, temperature queries, and data logging. For 8 sensors, a query time between 3 and 3.5 seconds was required.

The thermocouples were calibrated against each other using 5000 measurement data points at room temperature, with a 10-second interval between measurements.

For determining the heat loss of the calorimeter 0,8 litre of deionized water was used. The sample holder was filled with deionized water at a temperature of approximately 80 °C. The temperature rise phase was not used for calibration; instead, data values below 55 °C were utilized.

With a constant insulation thickness, the temperature change can be described by the following differential energy balance:

$$c_p\ m\ dT = -k\ A\ (T - T_{amb})\ dt \tag{1}$$

and the resulting differential equation:

$$c_p\ m\,\frac{dT}{dt} = -k\ A\,(T - T_{amb}) \tag{2}$$

Where:

c_p: Specific heat [J/(kgK)]

T_{amb}: Ambient temperature [K]

T: Instantaneous temperature [K]

T_0: Initial temperature [K]

k: Heat transfer coefficient [W/(m^{2}K)]

A: Area [m^2]

m: Mass [kg]

t: Time [s]

When solving the equation, the temperature measured by each temperature sensor at t=0 sec was used as the initial condition.

The solution to the equation was determined in the form of (3):

$$T = (T_0 - T_{amb})e^{-\frac{k\ A}{c_p\ m}t} + T_{amb} \tag{3}$$

The used data acquisition system for measuring

One of the main criteria for selecting the microprocessor-controlled measurement data acquisition system was the ability to perform many parallel measurements, regardless of the number of inputs. Using a digital thermometer with an Arduino Uno, as opposed to traditionally used data loggers, allowed for querying up to 128 temperature sensors per digital input.

Based on the measurement methodology, two more thermocouples are needed than the number of mortar samples: one for a reference calorimetric temperature measurement (testing of hardened mortar in a calorimeter) and one for external temperature measurement.

The first solution for the data acquisition circuit used is shown in **Fig. 5**.

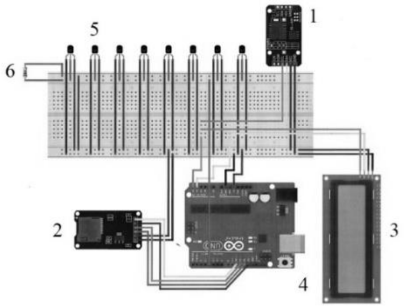

Figure 5. *The first version of measurement data logger circuit*

The system components are:

1 - DS3231+AT24C32 RTCM real-time memory module,

2 - MICROSD-M type memory card reader,

3 - Serial I2C 1602 16x2 Character LCD Module display,

4 - Arduino Uno microcontroller,

5 - DS18B20-2M type temperature sensor,

6 - 4.7 kOhm resistor.

In this setup, elapsed measurement time and corresponding temperature data were recorded to an SD card. Based on initial tests – given a planned measurement duration of 1 week – the system was not always sufficiently stable due to the hobby-grade type microcontroller. Furthermore, the data logging time (querying, opening file on SD card, logging data, closing file) caused inaccuracies in the timing module, necessitating the relocation of some microcontroller tasks to other devices.

The second and final design of the measurement data acquisition system is illustrated in **Fig. 6**. The system components are: 1 - Arduino Uno, 2 - DS18B20-2M type temperature sensor, 3 - 4.7 kOhm resistor. The sole task of the microcontroller is to read raw data from the digital thermometers and transmit it to the computer. For additional tasks Arduino panel interface for Matlab was utilized. The recorded data which can be stored in matrix form included: measurement sequence number, query timestamp, elapsed time, and temperature.

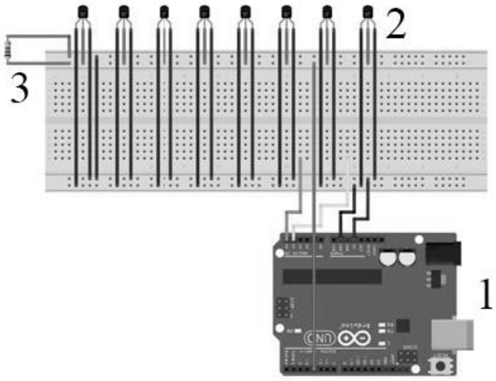

***Figure 6.** The second version of measurement data logger circuit*

With this setup, the stability of the measurements significantly increased, and out of the possible 128 sensors, data could be collected from 8 sensors every 10 seconds for 168 hours per measurement.

Temperature Compensation for Calorimeter Heat Loss

The first measured temperature does not require compensation. Every subsequent temperature value needs to be compensated using the kA product determined from Equation (4), as described in the previous subsection, to characterize the temperature and heat generation in an adiabatic environment.

The temperature loss in the semi-adiabatic system is:

$$T_{loss}(t=i) = \frac{k\ A}{c_p\ m}\,\Delta t(T_0 - T_m(t=i-1)) \qquad (4)$$

where:

c_p: Specific heat of cement mortar [J/(kg K)]

m_p: Mass of cement mortar [kg]

i: Measurement number [db]

T_0: Temperature of the first measurement [K]

T_{m} (t=i-1): Subsequent measured temperatures [K]

And the temperature related to an adiabatic system is:

$$T_k(t=i) = T_m(t=i) + \sum\nolimits_{t=1}^{i-1} T_{loss}(t) \qquad (5)$$

where:

T_{k} (t=i): Compensated (corrected) temperature [K]

Determination of Heat Generation

The heat generation of cement mortar is defined in two ways in the literature. For standard measurement procedures, the traditional form of [Joule/gram] is prevalent. However, for further work, especially as input for finite element programs, the unit of [Watt/m³] is also found in several places. This subsection describes the determination of the latter.

The heat change calculated from the compensated temperature is done using Equation (6):

$$\Delta q(i) = c_p m_p (T_i - T_{i-1}) \qquad (6)$$

The heat generation determined for the entire process is according to (7):

$$q(t) = \frac{\sum_{i=1}^{t} \Delta q_i}{t\ V} \qquad (7)$$

where:

q(t): Heat generation [W/m^3]

V: Volume of cement mortar [m^3]

t: Elapsed time [sec]

III. RESULTS

The developed calorimetric system offers the possibility to thermally characterize heat generation (or any other chemical process), not just with laboratory equipment. Compared to commercially available general-purpose data loggers, the developed system has more inputs and a lower acquisition cost. Thanks to the custom program control, sensor data can be read at arbitrary times. If necessary, the number of measurement points can be increased when detecting specific temperature changes (jumps) and reduced at other points.

Data processing can be easily performed using Matlab, even with many measurement points (hundreds of thousands) and multiple inputs.

Calibration of Temperature Sensors

The calibration was performed relative to the sensor identified as S1. The calculations were carried out at room temperature during the measurement, which was 19±0.5°C. Only the temperature differences were used during the measurement, as the raw materials for subsequent measurements underwent a 24-hour tempering process.

The results are summarized in **Table 5**. The signs of the deviations indicate temperatures smaller or larger than the reference value. The average deviation was derived from the arithmetic mean of 5000 measurement results.

Table 5. *Deviations of Thermocouples from Each Other*

ID	*Minimum*	*Maximum*	*Average*
S1	0 °C	0 °C	0 °C
S2	-0.125 °C	-0.250 °C	-0.197 °C
S3	0 °C	-0.063 °C	-0.034 °C
S4	-0.063 °C	-0.375 °C	-0.255 °C
S5	-0.188 °C	-0.313 °C	-0.236 °C
S6	-0.125 °C	-0.313 °C	-0.234 °C
S7	-0.313 °C	-0.438 °C	-0.396 °C
S8	0.063 °C	-0.063 °C	-0.023 °C

Determination of Calorimeter Heat Loss

Air-conditioned room was used to determine the heat loss of the calorimeters. Calculating heat loss is difficult due to the temperature changes in the air-conditioned room, as well as radiant heat from other surfaces related to the calorimeter's placement and from openings/windows.

Six calorimeters were used for the measurement, and two thermocouples measured the external temperature (one of these served as a backup for subsequent measurements, increasing the number of evaluable measurements in case of thermocouple failure).

The end of the measurement was determined by the temperature inside the first calorimeter dropping below 21°C. During the measurement, 14000 measurement points were recorded (approximately 39 hours of measurement). For function approximation, points from 1-5000 and 9001-14000 of the measurement were used. The first interval of points was needed for approximating the initial slope change of the exponential function, while the last 5000 points were necessary due to the function's asymptote at infinity. **Fig. 7** illustrates the signal from one in-calorimeter sensor and one ambient temperature sensor from the data series.

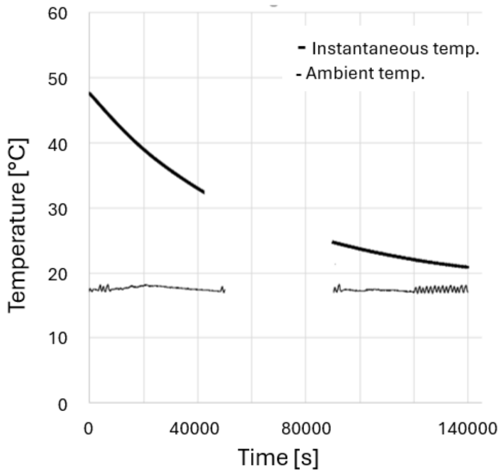

Figure 7. *Temperature-Time Diagram Shape Used for Processing*

In the relationship defined by Equation (4), the value of the kA product was determined from the recorded temperature-time data. For function approximation, the nonlinear least squares method with the TRM algorithm was used. The maximum number of iterations for the approximation was 400. The results of the regression calculation are summarized in **Table 6** (sensors S3 and S7 measured the room temperature).

From the values in **Table 6**, it is evident that the coefficient of determination (R^2) for the function approximation is greater than 0.99. Further calculations regarding the heat transfer coefficient can be performed from the kA product. However, for subsequent calculations, this specific value is not needed separately, so the products presented in **Table 6.** will be used in the calculations.

Table 6. *kA Values*

ID	*k A [W/K]*	*R^2*
S1	0.05990	0.9997
S2	0.06105	0.9998
S4	0.06141	0.9997
S5	0.06119	0.9997
S6	0.05977	0.9997
S8	0.06032	0.9997

Determination of Temperature Change for CEM III Type Cement Mortar

Preparations for testing the CEM III type mortar were carried out according to [1]. During the measurement, data were recorded every minute. The total duration of the measurement could only be estimated at the beginning. Thus, the objective was set that the end of the measurement would be determined by the sample temperature remaining unchanged for 1 hour. The test therefore lasted 73.5 hours.

In the semi-adiabatic system, the temperature change is indicated by the dashed line in **Fig. 8**. The continuous line in the diagram shows the result of applying the temperature compensation model.

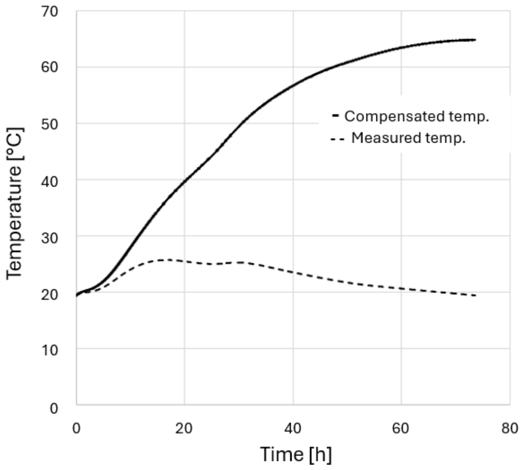

Figure 8. *Temperature-Time Diagram for CEM III*

Determination of Heat Generation

The heat generation according to Equations (6) and (7) is illustrated in **Fig. 9**. Based on the measurement, the interdependent chemical processes can be observed, as well as the fact that the maximum heat generation for CEM III type cement occurs between 25-35 hours, which is supported by the literature.

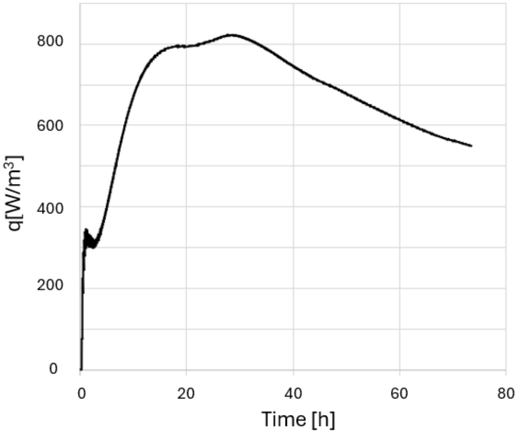

Figure 9. *Heat Generation-Time Diagram for CEM III*

IV. CONCLUSIONS

For the purpose of this study, a semi-adiabatic calorimeter was designed and manufactured in compliance with the MSZ EN 196-9 standard requirements and specifically adapted to mortar geometry. To ensure precise investigation of the heat of hydration, digital thermometer compensation was performed on the developed device. Subsequently, the thermal properties of the insulated mortar container were characterized through hot water measurements, utilizing a differential equation-based approach.

During the mortar experiments, the system demonstrated long-term data acquisition stability over a period of approximately 73.5 hours. The mortar temperature was successfully obtained as a monotonically varying function. By integrating this function with the physical characteristics of the mortar, the time-dependent heat generation of a 1 m^3 CEM III sample was determined.

A key contribution of this study is the application of the kA product, which successfully established a link between the semi-adiabatic model and the theoretical, adiabatic (heat-loss-free) case. This methodology allowed for the derivation of the concrete mortar temperature as a monotonically varying function. These results provide a robust foundation for further crack investigations and the analysis of tensile stresses during the hardening process, using a finite element (FE) model with a non-constant heat source intensity.

Despite the successful development and calibration of the calorimeter, certain limitations should be acknowledged. In future work, following a multi-point calibration of the thermocouples, the impact of the error function must be integrated into the compensation model. Although the mathematical model was verified using water-based measurements, no direct performance comparison was conducted between the developed device and a commercially available semi-adiabatic calorimeter during this phase of the research.

Future studies will focus on cross-validating the results with industry-standard equipment to further verify the absolute accuracy of the obtained heat generation curves.

AUTHOR CONTRIBUTIONS

E. Domonyi: Conceptualization, implementation of the measurement system.

M. Réger: Development of the compensation model, writing, review and editing.

A. Zachár: Supervision, review and editing.

DISCLOSURE STATEMENT

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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