Quality control with automated isothermal calorimetry

The integration of automated isothermal calorimetry with intelligent control software enables real-time process control in cement manufacturing by combining rapid reactivity measurements with AI-driven predictive analytics. The approach marks a significant advance in data-driven quality control, providing cement plants with a robust tool for optimising resource efficiency and product performance.

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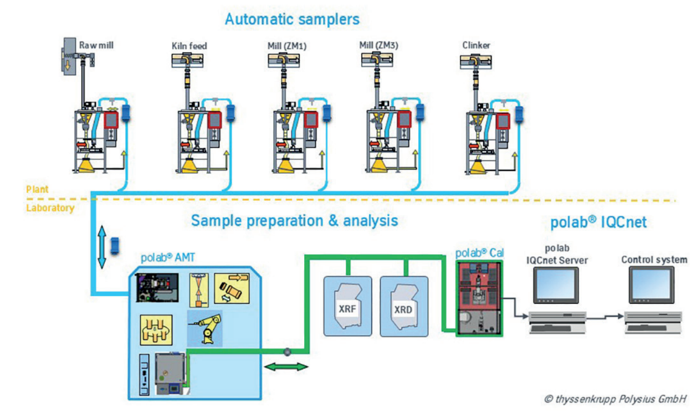
One of the key challenges in cement production is reducing clinker content while increasing the use of composite materials, which requires precise control and optimisation of both clinker and cement reactivity. Currently, cement plants do not directly quantify clinker and cement reactivity, relying instead on slow and error-prone methods such as compressive strength testing. Delayed feedback and limited data availability lead to material waste from out-of-specification production and prevent the integration of results into real-time process control. To address this, it is recommended that cement laboratories transition from retrospective analysis to proactive decision-making, enabling real-time process monitoring instead of relying on delayed reactivity data from the physical laboratory.

This transition in cement production can be supported by AI-driven software systems that combine predictive analytics, economic modelling and real-time regulation with fast reactivity analysis. Automated isothermal calorimetry offers a fast and precise method to overcome delayed responses by providing reactivity data for both cement and clinker. Integrating calorimetric data into intelligent control systems enables high-frequency analysis, dynamic process adjustments and consistent performance. This improves process stability and supports sustainability by reducing clinker content and optimising resource efficiency. As digitalisation progresses, the integration of automated calorimetry and intelligent software solutions marks a strategic move towards efficient, data-driven cement manufacturing.

Figure 1: simplified polab system configuration installed at customer site, featuring automatic samplers, polabAMT system and polabCal integrated into automated laboratory set-up with IQCnet software system



Isothermal calorimetry

Isothermal calorimetry monitors heat evolution and reaction kinetics during the hydration of cement. When water is added to a cement or clinker sample, exothermic hydration reactions are initiated, where the isothermal calorimeter continuously measures both the rate and total heat released over time. The amount of heat release directly reflects cement reactivity, which is closely linked to its performance characteristics. The method provides continuous, quantitative monitoring of hydration, in particular during the first 24h when the most intense reactions take place.

Cement hydration typically progresses through certain phases, initiated by the reaction of the main clinker phases: a strong initial peak caused by rapid dissolution of mainly C_3A, followed by a dormant period with minimal heat evolution, an acceleration phase dominated by the main C_3S hydration and a deceleration phase characterised by declining heat release. In some cement types, a secondary peak may occur due to sulphate depletion.[1]

Isothermal calorimetry is widely used to study, for instance, the effects of sulphate content, chemical admixtures and particle size distribution.[2] In industrial practice, it primarily serves to verify the compliance of low-heat cements and to assess sulphate balance. However, the method faces limitations in capturing early hydration reactions (< 1h), mainly due to thermal disturbances during sample insertion and labour-intensive sample preparation.[1]

An automated system with a temperature-controlled environment and robotic sample handling significantly improves reproducibility and enables real-time analysis of early hydration reactions in cement and clinker. This technological advance allows rapid and reliable calorimetric measurements, supporting both scientific research and process control in cement manufacturing.

Figure 2: polabCal with temperature-controlled housing, connected to automated sampling system at customer site

Integration of an automated isothermal calorimeter into a laboratory set-up

An automated isothermal calorimeter system (polabCal) was successfully installed at a cement plant in Germany last year. Figure 1 illustrates the conceptual layout of the polabCal system integrated into a laboratory environment with automated sample integration.

During production, material is collected from five automatic sampling stations, thoroughly homogenised, dosed into capsules and sent to the central laboratory for quality control. The samples are received at the automatic receiving station (ART) within the polabAMT system, where an industrial robot manages the handling process. An integrated sample preparation module (AMT) processes the material for representative laboratory analyses. The prepared material is transported via a belt system to the X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysers, and afterwards to the polabCal for reactivity measurements. The calorimeter operates fully automatically within a temperature-controlled enclosure, ensuring stable thermal conditions throughout the sample preparation and real-time analysis of the early hydration reactions of cement and clinker (see Figure 2).

The customer operates two mills (ZM1, ZM3) during regular production, each providing one sample per hour. Depending on the type of material and the selected analysis duration, the automated calorimeter can process up to eight samples per hour. The clinker sampler is scheduled for completion in autumn this year and will be incorporated into both the laboratory automation and the polabCal system.

Modular approach with Intelligent Quality Control (IQCnet)

A core feature of IQCnet is its ability to predict material reactivity before conventional laboratory data become available. This is achieved through a neural network trained on large datasets from XRD, XRF and particle size distribution (PSD). The system generates a simulated reactivity value, enabling laboratories to intervene early in the production process and adjust parameters proactively. The model continuously incorporates new data, including actual reactivity measurements from polabCal, ensuring its adaptability to changing raw materials and process conditions.

The following sections briefly describe the individual modules of IQCnet and their respective roles in process optimisation, followed by two practical examples demonstrating the functionality of Modules 1 and 4 in active control operation at the above-mentioned cement plant.

Module 1: fineness control

Reactivity data obtained from polabCal, alternating with predictive reactivity values, are fed into the fineness control module at 30min intervals. Based on these inputs, the module adjusts the separator speed to either higher or lower fineness depending on the designated reactivity value. The module operates within predefined safety constraints and can function independently or in tandem with the composition control module for a more holistic approach.

Module 2: composition control

This module adjusts the proportions of raw materials via belt scales to control the chemical composition and reactivity of the final product. It ensures compliance with the specifications of the designated cement type while dynamically maintaining the target reactivity. This allows laboratories to achieve precise formulation control and simulate production conditions with high accuracy.

Module 3: economic optimisation

The module incorporates economic considerations through its return on investment tool, which assesses the cost-effectiveness of control actions by analysing current electricity prices and raw material costs. It combines input from both the fineness and composition control modules to identify the most economically viable combination of adjustments.

Module 4: prediction of compressive strength

The IQCnet software also supports forecasting compressive strength at two and 28 days, providing early insights into product performance. The AI model is trained on a minimum of 600 data sets, each comprising reactivity values from polabCal, compressive strength results, particle size distribution, chemical and mineralogical properties. Predictions are continuously refined by comparing them with actual strength measurements.

Case studies: application on real-time data from cement plant operation

Modules 1 and 4 were developed using a comprehensive data set from the above-mentioned cement plant, covering more than six months of operational data, including XRD, XRF, particle size and reactivity values from polabCal. Hourly control steps are applied to each mill and cement type whenever key parameters are not within the defined target ranges. The following two examples – each from a different mill and cement type – illustrate real-time performance during ongoing production in September 2025. The time frames are exemplary. Module 1 has been active at the customer site since February and Module 4 since August this year, during periods when the mills are in operation.

The first interval displays the grinding process of CEM III/A 42.5 N over a 10h period (see Figure 3). During this time, the software continuously adjusted the separator speed based on real-time strength predictions and calorimetric

Figure 3: control steps based on reactivity, strength, and particle size for CEM III/A 42.5 N at cement mill ZM3, during the period from 10 September, 23h to 11 September, 9h. Green area indicates target range. Orange lines mark limits. Grey dashed line represents reactivity setpoint of the cement type. Control steps are performed hourly; additional reactivity prediction allows smaller adjustments every 30min

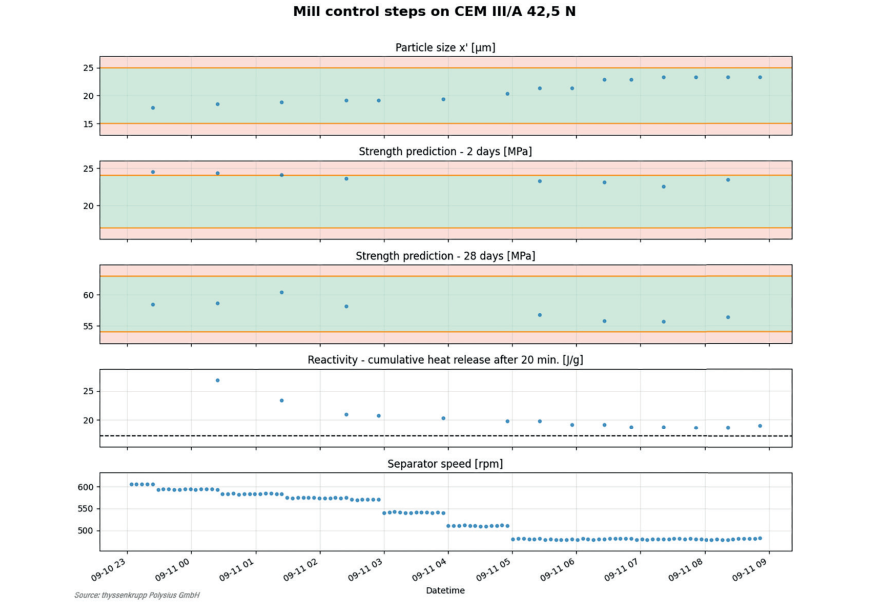
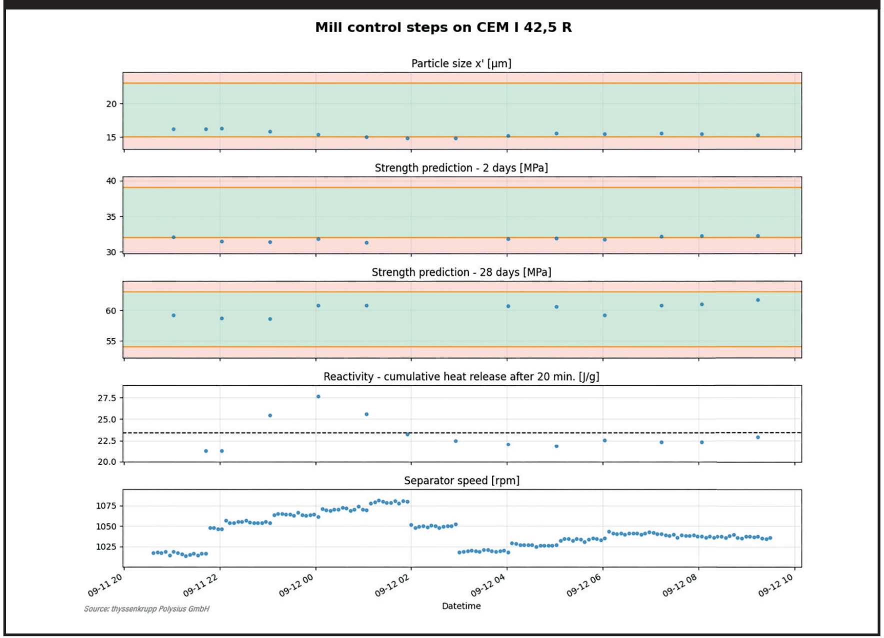


Figure 4: control steps based on reactivity, strength, and particle size for CEM I 42.5 R at cement mill ZM3, during the period from 11 September, 20h to 12 September, 12h. Green area indicates target range. Orange lines mark the limits. Grey dashed line represents reactivity set-point of the cement type. Control steps are performed hourly; additional reactivity prediction allows smaller adjustments every 30min



measurements. Initially, both the predicted two-day strength and reactivity (17J/g) exceeded their target values. In response, the separator speed was reduced from 605rpm to 570rpm in four incremental steps. As both early strength and reactivity were still outside the target range, a final adjustment was made, decreasing the separator speed to 480rpm. A temporary interruption in XRD measurements led to a 3h gap in strength predictions. However, the control loop mechanism remained active based on the available calorimetric data from the polabCal, allowing the regulation to continue.

After five total adjustments all key parameters – compressive strength, reactivity and particle size – were within their respective range, requiring no further changes due to constant process conditions. The predicted 28-day compressive strength remained within the target range throughout the entire control period.

The second time-frame presents results from a 16h control period, where the grinding process for CEM I 42.5 R was conducted on another mill (see Figure 4). At first, both the predicted two-day compressive strength (32MPa) and the measured reactivity (23J/g) were slightly below their respective targets. Therefore, the separator speed was increased in four steps from 1020rpm to 1080rpm, resulting in a gradual increase in both reactivity and two-day compressive strength.

Due to the control adjustment, reactivity increased to 26J/g – slightly above the target – while early strength reached the lower limit of the range. To realign reactivity with the set-point, separator speed was reduced in two steps to 1020rpm.

Over the next three hours, only minor adjustments were made, with the separator speed briefly increased to 1040rpm as the parameters remained within the lower target range, where no further adjustments were necessary. The predicted 28-day compressive strength consistently remained in the upper target range.

Outlook

IQCnet, in combination with automated isothermal calorimetry, marks a shift towards intelligent process control in cement production. By integrating real-time reactivity data with AI-driven analysis, it enables rapid, data-based decisions on actual measurements to maintain product targets and optimise quality. As the industry moves towards lower clinker content and increasing material complexity, new approaches and mechanisms are required. Stable operation and adaptive responses to raw material fluctuations depend on real-time feedback. The development of advanced technologies is crucial for cost-effective and sustainable cement manufacturing. ■

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