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european cement research academy

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NEWSLETTER

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Upcoming ECRA seminars/workshops:

- ECRA/CEMCAP Workshop "Carbon Capture Technologies in the Cement Industry"
  6–7 November 2017

- Secondary Abatement Techniques
  29–30 November 2017

Quality control of cement requires modern analytical methods

Online/inline analysis methods such as Rietveld and PGNAA provide new possibilities

Quality control of cement is absolutely indispensable, not only from a customer and authority perspective, but also for the cement industry itself. Cement is therefore – alongside foodstuffs and medicines – one of the best controlled products worldwide. Different physical and chemical methods have been developed to test the conformity of cement with different national requirements. The European harmonisation of standards for cement led to a consolidation not only of the requirements, but also of the testing procedures. Nevertheless, many test methods prescribed as reference methods for the purpose of quality assurance are still based on traditional wet chemical testing. For process control in cement plants such methods are less appropriate, particularly as they require too much working time. For this reason, modern online and inline analysis has become more and more established.

Already in the 19th century requirements on the product as well as corresponding testing methods were laid down to ensure the quality of cement and thereby the safety and durability of construction works. In the year 2000, EN 197-1 "Composition, specifications and conformity criteria for common cements" was published as the first harmonised European standard for a construction product. At the same time, the EN 196 series was released, defining the reference testing methods to be used for cement.

Quality control in cement plants

In addition to the reference test procedures, many other analytical methods have been developed. These methods can be applied during the cement production process in order to ensure an accurate and – even more important – constant quality of the cement.

For process control, the determination of parameters such as the chemical composition, the mineralogy, the fineness or the particle size distribution not only of the final products but also of raw materials and intermediate products are of interest. The test methods applied in industrial practise can be classified into offline analysis, atline analysis, online analysis and inline analysis.

Offline and atline analysis

For the purpose of offline analysis, the sampling, preparation and investigation of the samples are done manually. The wet chemical quantification of free lime is an example of offline analysis methods, which are usually carried out under "clean" conditions, e.g. in a central laboratory of the plant. Clinker microscopy (**Fig. 1**) requires even more expertise and is thus more often provided by external specialists then applied in the cement lab itself. Due to the fact that offline analysis requires time, the results cannot be used for direct process control. The fact that the work of experts is needed makes offline analysis less flexible with respect to personnel. Priority conflicts may occur in daily practise.

The principle of atline analysis is that samples are taken manually from the process, carried to the laboratory of the plant and analysed by automatic devices. For these methods, e.g. X-ray fluorescence analysis (XRF) or X-ray diffraction analysis (XRD), specified measuring equipment is needed. Specially trained personnel are required to operate and maintain the analytical devices. The results are available faster compared to offline analysis and the data can thus be better used for process control.

Figure 1: Clinker microscopy provided to support process optimisation of cement plants

Online and inline analysis

In online analysis, the automated analysis is combined with a fully automated sampling, sample transport and preparation system. A continuous data exchange between the analytical equipment and the process control system is possible. For example, XRF results from a laboratory automation system are used in many cement plants to control the kiln feed. XRD combined with Rietveld analysis (**Fig. 2**), laser granulometry and colour measurement systems are further examples of devices used for online analysis. Immediate process corrections can lead to significant process and cost optimisation and are the main advantage of online analysis. On the other hand, the capital costs are higher and specialised personnel is required for the operation of the system and for troubleshooting.

For inline analysis, samples are neither taken, transported to the central

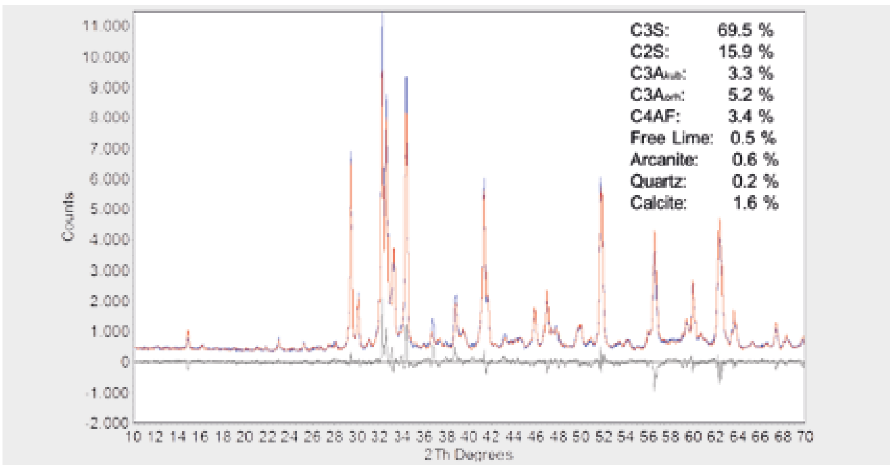

Figure 2: Rietveld analysis of a cement sample

laboratory nor prepared for the testing. Instead, the material is analysed during the process itself. This leads to a continuous flow of data that can be used directly for process control. Errors from sampling and sample preparation are excluded and no personnel is required for the analysis itself. On the other hand, the methods are often not as precise as analyses under "clean" laboratory conditions. The enormous amount of data can in most cases statistically compensate this disadvantage. The capital costs and the requirements on the robustness of inline devices are even higher than for online analysis. A concept for maintenance and quick repair is necessary.

Prompt Gamma Neutron Activation Analysis (PGNAA) is an example of inline analysis. This method can be used for the determination of the chemical composition of raw material, i.e. limestone, marl and clay from the quarry. The material is transported on a belt conveyer through the analysis cabin, where it is continuously irradiated with a beam of neutrons. Prompt gamma rays emitted from the material are detected to identify the chemical elements and their concentrations. The results can be used to optimise stockpiling for the purpose of homogenisation to optimise the production process.

Which methods are recommended?

A decision on the implementation of analytical methods should always take into account the local conditions of the cement plant. If the homogeneity of the quarry is poor, a method like PGNAA may lead to significant improvements. The high investment costs and effort for maintenance may not be justified for other cement plants with more homogeneous quarries. The use of secondary materials, the given technical circumstances in the cement plant, the number of cements and the requirements of the market should also be considered. Last but not least, the availability of trained personnel must be taken into account.

ECRA and CEMCAP jointly examine carbon capture technologies

EU research project CEMCAP uses ECRA CCS project results for experiments and modelling

CO_2 capture is seen as a potential breakthrough technology for the significant CO_2 reduction of process emissions in the cement industry. CEMCAP is a project funded by the EU's Horizon 2020 programme addressing CO_2 capture from cement production through the demonstration of different CO_2 capture technologies in an industrially relevant environment (TRL 6) based on the previous work of ECRA and Norcem.

ECRA is involved as an industrial advisory group to give guidance to the CEMCAP research consortium and communicate the results within the European cement industry. Furthermore, an important contribution has been made by providing results from ECRA's CCS research project, which formed a starting point for the design of CEMCAP research experiments and modelling studies. This information base has recently been summarised in the so called "CEMCAP Framework" document: (https://www.sintef.no/projectweb/cemcap/results/).

Project partners in CEMCAP Workpackage 9 "Oxyfuel clinker cooler prototype" are IKN GmbH, HeidelbergCement AG, VDZ gGmbH and SINTEF as coordinator of the CEMCAP project.

A primary objective of the research project CEMCAP is to prepare the ground for the large-scale implementation of CO_2 capture in the European cement industry. Although there are still significant open issues to be resolved, CEMCAP's goal is to identify technologies with the greatest potential to retrofit existing cement plants and formulate a techno-economic decision basis.

Oxyfuel clinker cooler prototype at HeidelbergCement in Hannover

CEMCAP's experimental research aims to reach Technology Readiness Level (TRL) 6 for oxyfuel capture technology in cement plants and three post-combustion capture technologies, with a targeted capture rate of 90 %. In 2017, tests on the first oxyfuel clinker cooler prototype in a cement plant and on an oxyfuel burner and calciner have been concluded.

Furthermore, a portfolio of cost- and resource-effective options for CCS and thus the range of available options for reducing greenhouse gas emissions will be described. This is intended to serve as a foundation for European cement producers to calculate plant-specific CCS business cases, and enable them to be at the forefront of adapting to an emerging carbon-constrained world.

2nd ECRA/CEMCAP Workshop

On 6–7 November 2017 ECRA and CEMCAP will hold their 2nd joint workshop in Duesseldorf. The workshop will highlight the draft conclusions from CEMCAP experiments and research and discuss the technological and economic framework for the application of carbon capture technologies in the cement industry. Presentations will include options for calcium looping and direct separation capture technology in the cement industry. More information and details of how to register can found at: https://ecra-online.org/seminars-and-events/overview/

More information about CEMCAP can be found at: www.sintef.no/projectweb/cemcap

Phase II of ECRA's Future Grinding Technologies project launched

Focus on a modelling approach combined with a practical technology evaluation

In June 2017 the newly formed steering committee of ECRA's new research project "Future Grinding Technologies" met for the first time in Duesseldorf. The committee, consisting of researchers and experts from both the cement industry and suppliers, discussed the details of Phase II of the project. In the first phase of the project, the research agenda was developed, based on comprehensive studies on existing comminution technologies and on the cement industry's need for new grinding solutions. These investigations have led to an approach that combines comminution modelling with a practical evaluation of existing technology. Fig. 1 shows the overall research agenda of the project.

Already in 2013, ECRA's Technical Advisory Board had identified grinding as a future field of research activity, in particular with respect to the low efficiency of the process. In order to trigger a systematic approach towards innovation in cement grinding, ECRA began a pre-study on grinding equipment before assessing the situation in the industry in a two-day workshop.

Starting point: Round-table event

During a round-table event with over 50 comminution experts from various industries, the requirements on comminution for today and tomorrow were defined. The technology database developed by ECRA and the discussions during the round-table showed that a wide range of interesting comminution technologies already exist, but in order to learn from these a systematic evaluation approach is required. One of the most remarkable outcomes was the high priority placed on product quality and operational flexibility. Energy efficiency was ranked the third most important criterion for comminution processes. The detailed evaluation of all data from the round-table event can be found in the technical report on Phase I of the project, which is available on the ECRA website.

Theory and practice well-balanced

In order to provide energy-efficient equipment, it first has to be ensured that product quality can be maintained at all times. Predicting residence times and particle size distributions for products manufactured in various new mill systems and blended afterwards is a complex task. This cannot be determined by a stand-alone experimental approach. Modelling and simulation toolboxes, which are a common standard in the mining industry, enable very efficient virtual testing of equipment and plant layouts. Today there are also many powerful models for comminution available, but none that can fully match the requirement of the cement industry to master full particle size distributions and energy demand. Therefore, a work package on the development of a basic new comminution model coupling existing theories including methods for parameter determination has been assigned to the Institute for Particle Technology of the University of Braunschweig.

The modelling approach is combined with a second practical work package that will further evaluate ECRA's technology database. Methods commonly used in product development will be applied to the data in order to better understand technical solutions for all the relevant sub-processes of comminution. Based on a desk study, the first practical trials are being planned. ECRA has assigned a study on this topic to a master student from the National Technical University of Athens.

The research project and membership of the steering committee are open to all interested parties willing to contribute to the project's budget.

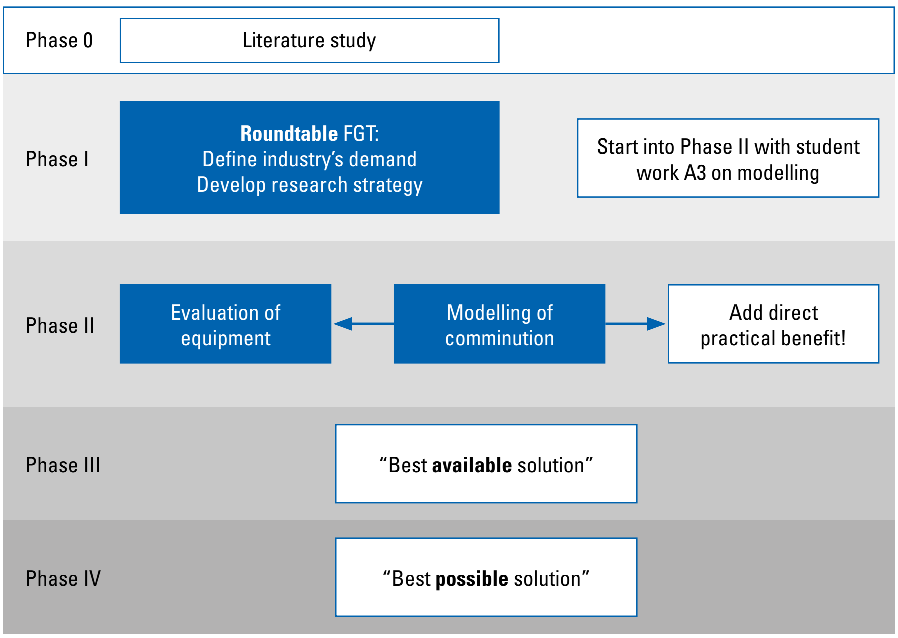

Figure 1: Research agenda for ECRA's Future Grinding Technologies project

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