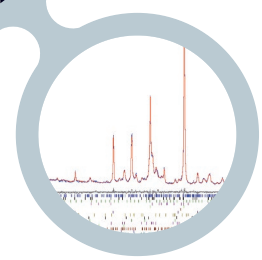

Lab Report XRD 72

Proactive blockage prevention in cement production by quantitative Hotmeal phase analysis

Introduction

One of the most critical processes during Portland Cement clinker production is the operation of the preheater system. Coatings or build-ups in the preheater tower increase the risk of blockages and downtimes. The origin of this operational problem is the formation of detrimental phases, which influence the flow of the hotmeal and lead to the build-up of cloggings.

The established strategy to manage these build-ups is to try to influence the chemical composition by using a bypass system that removes sulphur, alkalis and chlorine.

Now, for the first time, the detrimental phase formations can be monitored directly by X-ray diffraction and TOPAS Rietveld phase quantification in real time. This not only helps to completely understand the process, but also allows proactive strategies for reducing and solving blockage problems.

Blockage Formation

Blockage tendencies depend on parameters such as the concentration level of highly volatile compounds in the gas phase (e.g. alkalis, sulfur and chlorine), the temperature profile and the resulting mineralogical reactions in the preheater. The risk of coating formation is traditionally expressed by a chlorine-sulfur chart (Figure 1). However the knowledge of the hotmeal phase composition has been assumed to provide much deeper insights, which allows a better understanding of the reasons behind coating problems.

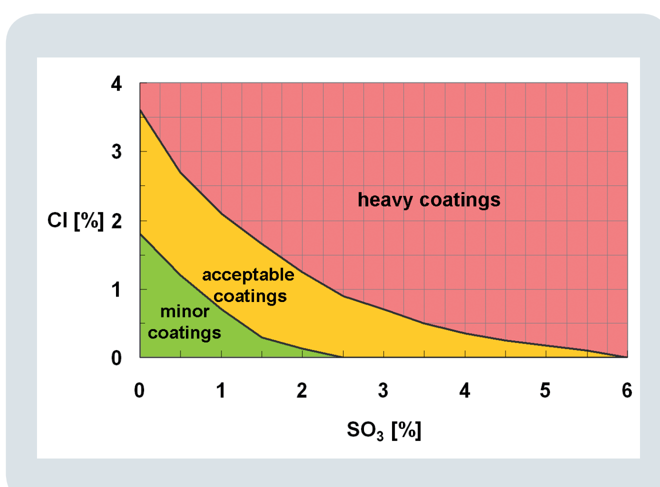

Figure 1: Risk of build-up formation in hotmeal related to the Cl and SO_3 concentration.

The phase composition of the hotmeal directly reflects the gas solid equilibrium and therefore the efficiency and possible malfunctions of the preheater system. Table 1 provides an overview of major phases commonly present in hotmeal. Other phases may be present in minor concentrations as well. Due to significant software developments X-ray diffraction combined with TOPAS Rietveld analysis became one of the most powerful techniques for doing quantitative phase analysis. Modern detector technology reduces measurement time to minutes, allowing real process control. Analytical details are summarized in the experimental setup section.

Table 1: Typical hotmeal phases (for some phases the cement chemical nomenclature system is used).

Phase name	Formula	Phase name	Formula
Calcite	$CaCO_3$	Carbonatespurrite	$Ca_5 (SiO_4)_2 CO_3$
Quartz	SiO_2	Jasmundite	$Ca_{22}(SiO_4)_8O_4S_2$
Freelime	CaO	Ellestadite	$Ca_{10}(SiO_4)_3(SO_4)_3(Cl,F,OH)_2$
alpha Belite	C_2S alpha	Ye´elimite	$Ca_4Al_6O_{12}SO_4$
beta Belite	C_2S beta	Mayenite	$C_{12}A_7$
Aluminate	C_3A	Halite	NaCl
Ferrite	$C_2Fe_{2-x}Al_xO_5$	Sylvite	KCl
Periclase	MgO	Ca-Langbeinite	$K_2Ca_2(SO_4)_3$
Alite	C_3S	Anhydrite	$CaSO_4$

Experimental Setup

The measurement data were acquired using the D4 ENDEAVOR diffractometer in Bragg-Brentano geometry equipped with the 1-dimensional LYNXEYE compound silicon strip detector. Instrument settings are given in Table 2. Quantitative phase analysis was done using the TOPAS (Version 4) software.

TOPAS defines a new generation of Rietveld software. Its unrivalled performance for quantitative phase analysis is based on the Fundamental Parameters Approach [1], powerful minimization algorithms, mathematical stability and unmatched speed of calculation.

Table 2: D4 ENDEAVOR setup with the LYNXEYE detector.

D4 ENDEAVOR	
Goniometer	D4 ENDEAVOR Theta/2Theta
Measurement circle diameter	401 mm
Tube	2.2 kW Cu long fine focus
Tube power	35 kV / 40 mA
Primary optics	Divergence slit fixed to 0.5°
	4° Soller slit
Sample stage	Spinner
Secondary optics	Cu Kß filter
	4° Soller slit
Detector	LYNXEYE (opening 3.9°)
Step size	0.02°
time per step	0.1s
Angular range (2Theta)	10° to 65°
Total measurement time	4 min 55 sec

Figure 2: The D4 ENDEAVOR process diffractometer.

Measurement data

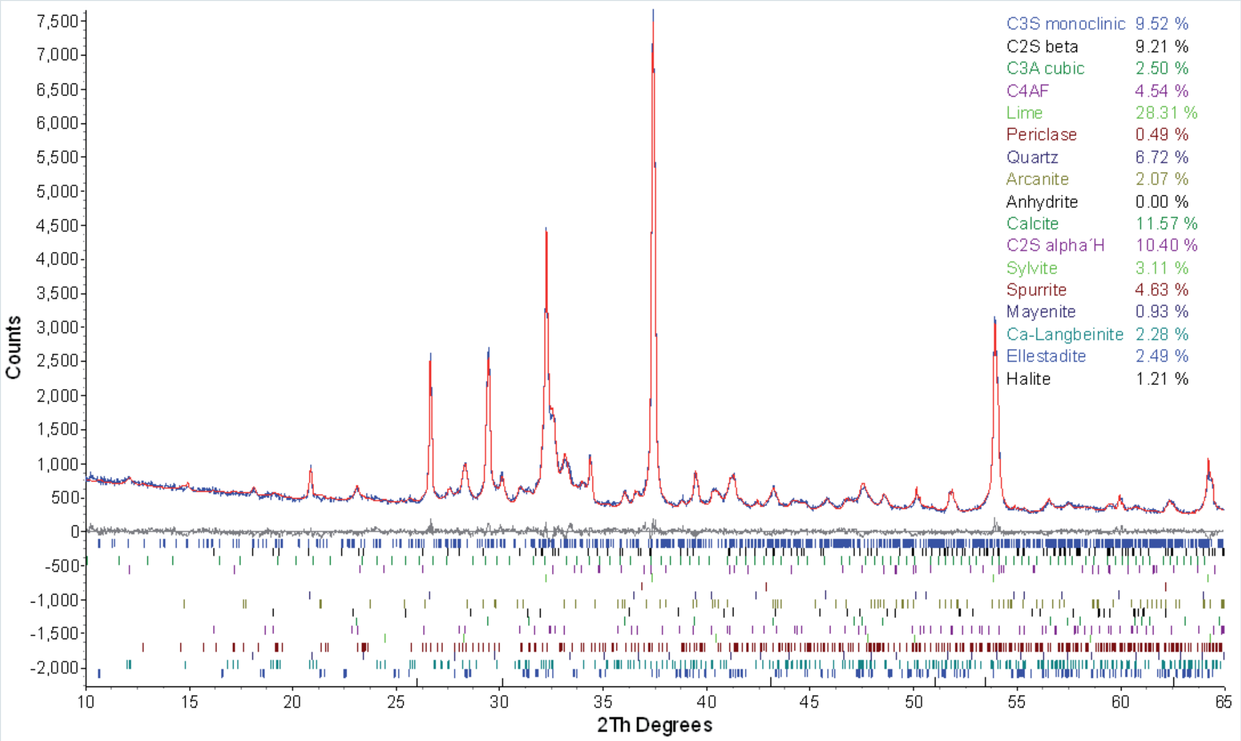

Figure 3: TOPAS Rietveld quantification of a hotmeal sample. With the ultra fast LYNXEYE detector, measurement data are obtained in minutes. The TOPAS software allows the automated analysis of complex phase mixtures in a few seconds.

Real-time Hotmeal Phase Quantification

A typical TOPAS quantification result of a hotmeal sample is shown in Figure 3. It presents the experiment data together with the fit of 18 mineral phases and the corresponding phase concentration.

The abundance of the different phases is directly related to process relevant parameters. Some obvious examples are:

- Calcite and freelime allow monitoring of the decarbonation in order to control the efficiency of the preheater.
- Carbonatespurrite and Ellestadite are typical blockage minerals resulting in hard cloggings that are difficult to remove.
- The flow characteristics depend on sulphates and chlorides.
- Alite in hotmeal is a measure for clinker dust cycles.

Consequently, this knowledge can be applied to adjusting the temperature profile and the material input in order to move the stability field of the blockage building phases into uncritical parts of the preheater and kiln system.

Conclusion

The hotmeal phase analysis, in contrast to mere chemical analysis, is the basis of a detailed understanding of the preheater processes. The D4 ENDEAVOR, combined with the latest generation TOPAS Rietveld analysis, provides useful process relevant parameters quickly and simultaneously such as

- Degree of decarbonation
- Formation of blockage minerals
- Chloride and sulfate cycles
- Clinker-dust cycles

By using X-ray diffraction blockage tendencies can be recognized at an early stage in order to control the gas solid equilibrium and to keep the system stable. This not only helps to minimize blockages and kiln shutdowns, but also results in a smooth kiln operation. The economical benefits are a better energy efficiency and increased productivity.

Keywords

XRD, quantitative phase analysis, TOPAS Rietveld, Hotmeal, Blockages

Authors

Rainer Schmidt, Bruker AXS GmbH

References

[1] Cheary, R.W., Coelho, A.A. & Cline, J.P.: "Fundamental parameters line profile fitting in laboratory diffractometers", Journal of Research of the National Institute of Standards and Technology, 2004, 109, 1-25

Bruker AXS GmbH

Karlsruhe · Germany
Phone +49 721 50997-0
Fax +49 721 50997-5654
info.baxs@bruker.com

www.bruker.com