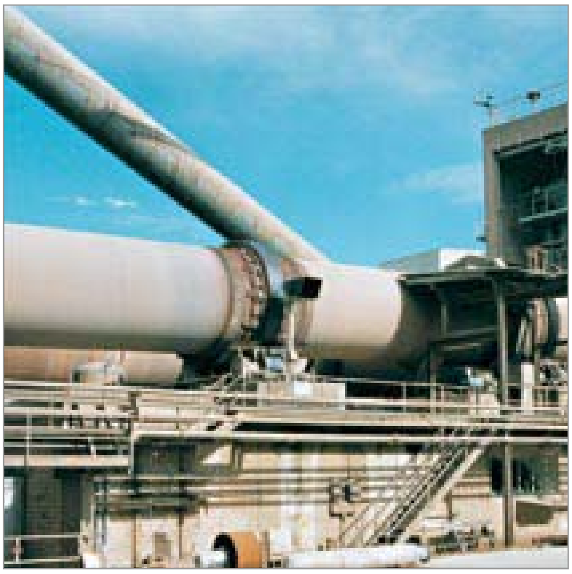
ACCURATE QUANTITATIVE RIETVELD ANALYSIS WITH TOPAS IN THE CEMENT INDUSTRY

Introduction

X-ray powder diffraction (XRD) combined with quantitative Rietveld analysis is a powerful tool for the determination of relative phase amounts in mixtures such as clinkers and cements. Such analysis is of high importance, as the performance of the products (and, therefore, of the plants) is governed by its mineralogy and not its elemental composition as determined by X-ray fluorescence (XRF).



With XRD and Rietveld analysis a full quantification of the crystalline components of a sample can be obtained providing detailed mineralogical information about the clinker and cement products. As a result, important parameters such as grindability of raw materials and clinker, burnability of raw feed, cement strength development, and cement setting time can be controlled.

Until recently, the application of the Rietveld method was hampered by several drawbacks of existing software such as complexity, low performance and instability. In particular the latter was prohibitive for automated use.

With a completely new developed Rietveld software, TOPAS, now the decisive breakthrough has been achieved. TOPAS not only allows the analysis of extremely complex phase mixtures in the shortest time possible, but also a fully automated on-line phase analysis for production control and quality management - without any user input.

LAB REPORT XRD 33

Experimental

In this study two NIST clinkers (standard materials RM 8486 and 8488) have been measured using a D5000matic process control diffractometer. The system configuration is provided in Table 1.

The NIST clinkers have been selected, as their phase composition is best known. Certified phase amounts have been determined by means of chemical and microscopical analysis.

In Fig. 1 an illustrative example for the possible phase abundance in a typical Portland cement is shown. The extremely high degree of peak overlap makes quantitative phase analysis a straight-forward task, which can not be performed with classical quantification methods at all.

In contrast to this, the Rietveld method perfectly addresses the overlap problem and allows the full quantification of all phases present within a negligible time. This is shown in Fig. 2 for a TOPAS refinement of RM 8486. Note the total calculation time of about 10 seconds on a Pentium III PC system for this example!

The refinement results for both standards, RM 8486 and RM 8488 are shown in Tab. 2a and b, respectively. The accuracy of the results is significantly better than +/- 3% with respect to the NIST certificate. In contrast to this, the deficits of the Bogue calculations, which are also provided in the tables, are clearly seen. Differences of up to 10% for the main phase C_3S compared to the microscopy and XRD results demonstrate that Bogue calculations are not suited for reliable phase quantification.

A detailed look at RM 8486 shows another deficiency of microscopical point counting. Often identification and distinction of the interstitial phases C_3A and C_4AF are difficult. In this case, it could be shown that the XRD results are more reliable and a reinvestigation of this standard material, which was done recently by NIST, confirmed this result. (Stutzman, P.E. and Leigh, S., "Compositional Analysis of NIST Reference Material Clinker 8486", Accuracy in Powder Diffraction III, Gaithersburg, 2001, in press)

Instrument Specifications	
Tube	2.2 kW Cu long fine focus
Tube power	40 kV, 40 mA
Incident beam optics	variable divergence slits fixed to 0.7° 4° Soller slit
Diffracted beam optics	variable anti-scatter slits fixed to 0.7° 4° Soller slit 0.2 mm detector slit
Detector	Scintillation counter
Measurement Range	10° - 55° 2θ
Step size	0.02° 2θ
Step time	1 s per step
Total measurement time	about 38 min.

Table 1: D5000matic process control diffractometer

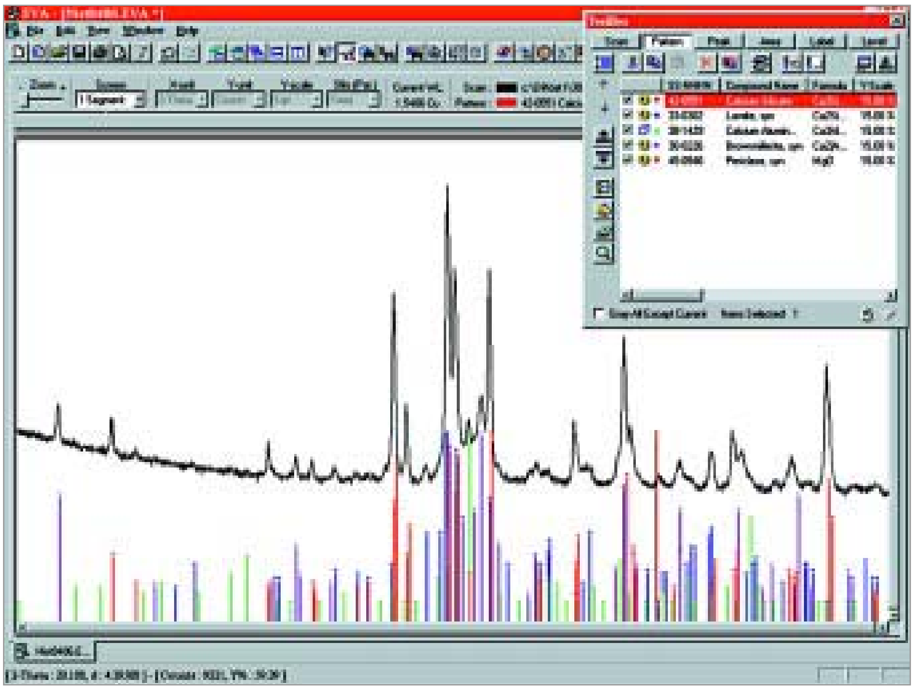


Fig. 1: Phase abundance in RM 8486. Intensities on a square root scale to show more pattern details.

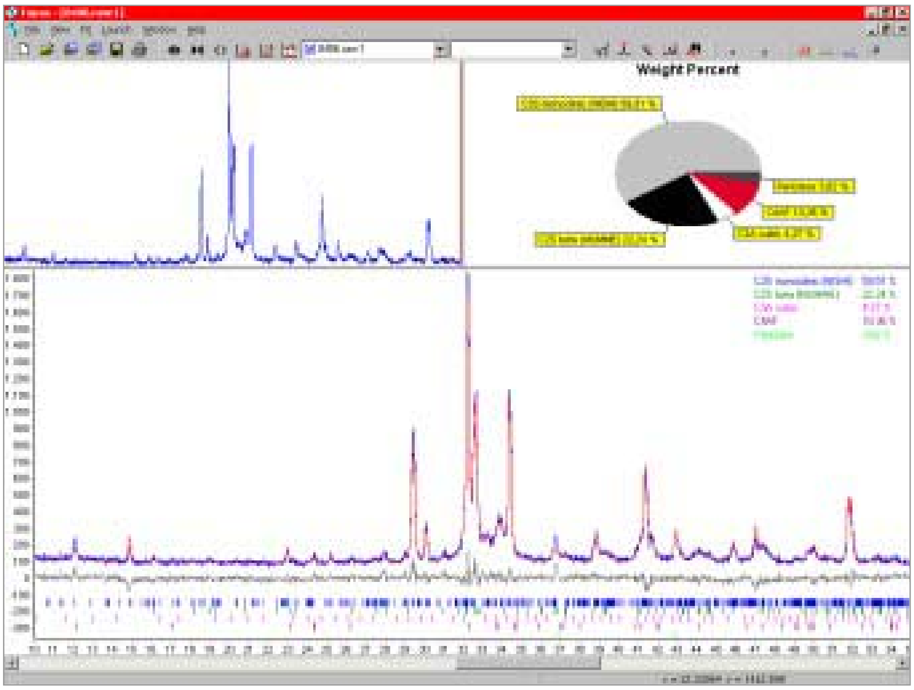


Fig. 2: Quantitative Rietveld analysis of RM 8486 with TOPAS. Note the total calculation time of about 10 s on a Pentium III PC system for this sample.

Compound	TOPAS Results	NIST	Bogue
Alite C_3S	59.36 (0.65)	58.47 (1.15)	48
Belite C_2S	22.43 (1.09)	23,18 (1.94)	28
Aluminate C_3A	4.27 (0.25)	1.15 (0.10)	7
Ferrite C_4AF	10.33 (0.86)	13.68 (0.63)	11
Periclase	3.61 (0.21)	3.21 (0.72)	

Table 2a: TOPAS Quantification results for NIST RM 8486

Compound	TOPAS Results	NIST	Bogue
Alite C_3S	66.91 (1.17)	64.97 (0.56)	57
Belite C_2S	18.01 (1.11)	18.51 (0.58)	22
Aluminate C_3A	3.19 (0.10)	4.34 (1.35)	7
Ferrite C_4AF	11.89 (0.42)	12.12 (1.50)	12

Table 2b: TOPAS Quantification results for NIST RM 8488

Conclusions

The unique capabilities of the new TOPAS Rietveld software allow the full quantification of cements and clinkers with high accuracy. With TOPAS the mineralogical phase amounts in clinkers and cements can be quantified directly - in real-time and on-line.

Typical calculations times of less than 30 seconds and the automation capabilities of TOPAS allow its fully automated use for production control and quality management.

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