



Lab Report XRD 88

Characterization and Quantification of Australian Iron Ore and Gangue Minerals

- Minerals and amorphous quantification using the D8 ENDEAVOR with an external standard method and DIFFRAC.TOPAS

Iron ore mining and processing corporations are facing continually growing demands on making their operation more economic and environmentally compatible. For implementation it is necessary to control feedstock sorting, adapt beneficiation processes and tailor the chemistry of sintering products, such as iron ore pellets. The key to success is fast mineral identification and quantification using X-ray diffraction.

Introduction

Iron ore is one of the most important resources for the global economy: 98% of it is used to produce steel, which is essential to infrastructure, transportation and manufacturing. The vast majority of the 2 billion tons of iron ore mined annually come from China, Australia, Brazil, India and Russia.



Figure 1: The D8 ENDEAVOR diffractometer can be operated manually, or in automated mode for full flexibility in an analytical laboratory.

Although the iron-ore deposits are vast, they have been estimated to be exhausted by the end of the century. "Direct-shipping ores" containing predominantly hematite (Fe_2O_3 , 70% Fe) are already depleted in the United States. But improvements in beneficiation processes have allowed processing of lower grade ores containing e.g. magnetite (Fe_3O_4 , 72% Fe), goethite (FeOOH , 62% Fe) and siderite (FeCO_3 , 48% Fe).

Lower grade ores typically require selective mining to minimize extraction of unwanted deposits, as well as beneficiation techniques to increase the iron concentration. The efficiency of these processes can be increased whilst reducing cost, if

- less valuable or problematic minerals are identified for separation,
- beneficiation processes are adjusted if gangue minerals are present,
- the composition for iron ore pelletizing is tailored to minimize cost of additives and reducing agents.

X-ray diffraction (XRD) can address these issues by delivering the mineral composition and content. Our D8 family of XRD instruments is easy to use, fast and results are operator-independent. Sample preparation, measurement and data analysis can be highly automated enabling 24/7 production and process control.

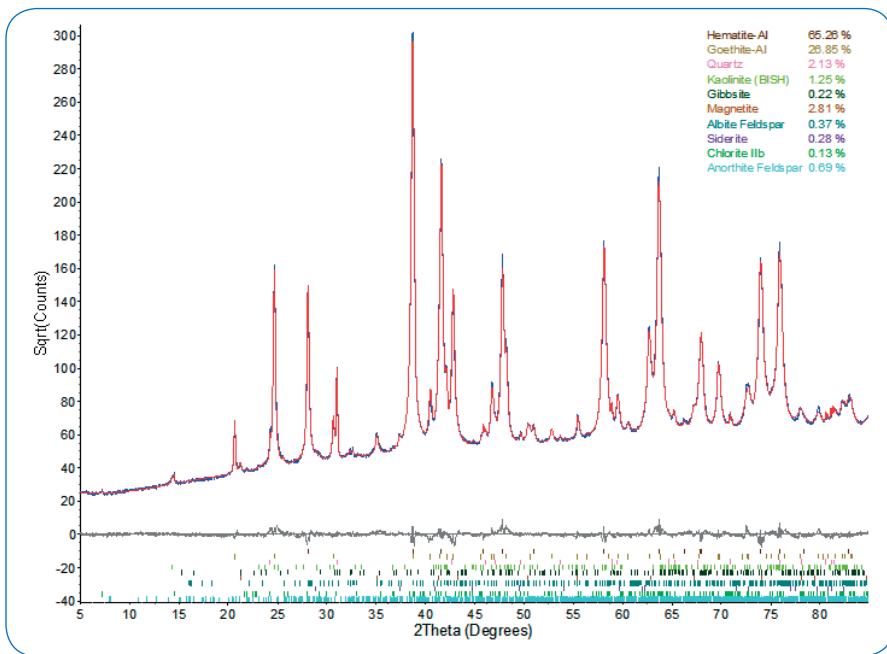


Figure 2: TOPAS result for ASCRM035: The Rietveld fit (red) matches well the measured data (blue); their difference is shown with the grey curve. The colored bars at the bottom indicate the peak positions of the different mineral phases according to their order in the top right corner list of concentrations.

Typically, analysis of XRD data provides:

- qualitative and quantitative mineralogy, including iron-deposit minerals such as gibbsite, goethite and ferrous dolomite (ankerite)
- Limit of detection (LOD) for minerals < 1%
- LOD $\text{FeO} \sim 0.3\%$
- Total Fe content and $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio
- Si and Al minerals and content

Materials and Methods

In this report, we illustrate the capabilities of XRD for iron ore mining on six iron ore standard reference samples from Western Australia (ASCRM030 – ASCRM035) with certified elemental composition. The fine grained powders were prepared via back-loading in steel sample holders with $\varnothing 31$ mm cavity.

The samples were measured on a D8 ENDEAVOR Minerals diffractometer (figure 1) equipped with a Co-tube (35 kV/ 40 mA) and a LYNXEYE XE-T detector to minimize micro-absorption and reduce iron fluorescence. A very low background signal was achieved for all samples with the Dynamic Beam Optimization (DBOTM) approach, which uses a variable divergence slit and a motorized anti scatter screen. Data was collected from 5 - 85° 2θ, with a step of 0.017° within 15 min.

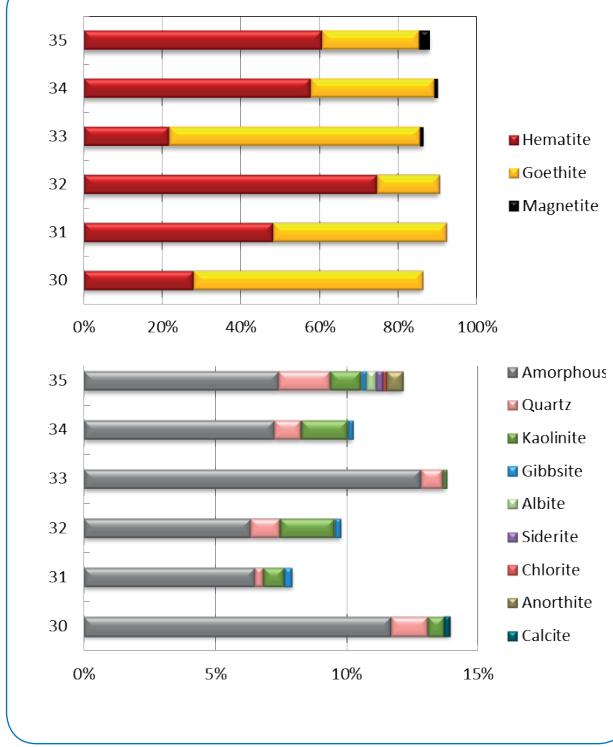


Figure 3: Quantitative Rietveld results for the ASCRM materials 030 - 035. All samples are high in iron (total Fe: 55 – 62%) and low in gangue (Al, Si: 1 – 4 wt%).

The mineral components were identified using the DIFFRAC.EVA software and the ICDD PDF2 database. The different phases were quantified with the external standard method (see box) implemented in the Rietveld software DIFFRAC.TOPAS.

The quality of the Rietveld fit was further improved by taking into account aluminum substitution on the iron site in both goethite and hematite, as well as preferred orientation effects in the later. These models are easily implemented in DIFFRAC.TOPAS and improve the overall accuracy on the quantification result.

Results

The diffraction pattern was analyzed by full pattern modelling using the TOPAS-Rietveld method, taking into account all mineral phases and also instrument settings simultaneously. Figure 2 shows the diffraction pattern and Rietveld fit for ASCRM035 as representative example for all samples. Quantitative phase analysis (QPA) indicates that iron-deposit minerals (hematite, goethite and magnetite) constitute 87.9(2) wt% of the sample (total Fe: 60 wt%), various gangue minerals (quartz, kaolinite, gibbsite, etc.) amount to 4.7(5) wt%, and 7.4(3) wt% of the sample are amorphous (*i.e.* non-crys-

The external standard method

Classic Rietveld analysis only considers the phase fraction of crystalline components while amorphous fractions are considered background and thus ignored. Consequently, the weight fractions of the crystalline phases are overestimated. This limitation can be easily overcome with the external standard method, where the crystalline fractions are corrected using the so-called K-factor and the sample's mass absorption coefficient (MAC).

The K-factor is determined by measuring a highly crystalline material using the same diffractometer settings as for the actual sample. No mixing of sample and standard material is required. In this application note, corundum (Al_2O_3 , 99.9% crystallinity) was used as such a standard.

The external standard method is implemented in the DIFFRAC.TOPAS software version 5 (2014) and can be automated as part of the TOPAS.BBQ software package.

talline). The phase quantifications including the amorphous content for all samples are illustrated in figure 3. Table 1 compares the total iron and gangue content to the certified elemental composition of the ASCRM standards.

Discussion

The D8 ENDEAVOR is ideally suited for high-throughput measurements of iron ores:

- Outstanding measurement speed and LODs guaranteed by the proprietary Dynamic Beam Optimization (DBO) and LYNXEYE XE-T technology
- Highly accurate iron mineral analysis ensured by the use of Co-radiation and TOPAS-Rietveld quantification
- Automation options from push-button operation to operator-free sample handling, measurement execution and data analysis

The quantitative mineral analysis can be started as soon as the measurement finishes – and the results are then available in the database to provide feedback to mine operators immediately. This allows to control feedstock sorting based *e.g.* on the total iron content, or to adapt the beneficiation processes to the gangue mineralogy.

XRD is also a reliable tool to determine the Fe²⁺/Fe³⁺ ratio, an important factor in iron-ore pelletizing and smelting, which is used to optimize the reducing agent (coal) required for iron reduction. In addition to the mineral composition, XRD allows to assess the crystallite size, which may impact the reactivity of a mineral. This information allows tailoring the type and quantities of additives required to optimize processing conditions in the blast furnace.

Summary

XRD analysis delivers insight into the iron ore materials at all stages, from mining to beneficiation and pig-iron production. Where XRF provides elemental analysis, XRD also sheds light on the chemical environment of iron and its oxidation state – both important parameters in reducing cost in iron-production.

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The micro-absorption effect

All materials absorb X-rays more or less strongly depending on their chemical composition and the wavelength of the X-rays. A high absorption contrast between strong and weak absorbers negatively influences the phase quantification. This effect, called micro-absorption, can only be reduced by choosing a wavelength which gives low absorption contrast. For example, Cu-radiation gives a high absorption contrast between iron-ore and gangue minerals, whereas the contrast is low with Co-radiation.

Mineral	wt%	Fe(II)O	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Na ₂ O	CaO	MgO
Hematite	60.43		60.43					
Goethite	24.87		22.34					
Magnetite	2.60	0.81	1.79					
Quartz	1.97			1.97				
Kaolinite	1.16			0.54	0.46			
Gibbsite	0.21				0.14			
Albite	0.35			0.23	0.07	0.04	<0.01	
Siderite	0.26	0.16	0.18					
Chlorite	0.12	0.02	0.02	0.04	0.02			0.03
Anorthite	0.64			0.29	0.23	<0.01	0.12	
Fe ₂ O ₃			SiO ₂	Al ₂ O ₃	Na ₂ O	CaO	MgO	
XRD	85.75		3.06	0.92	0.04	0.13	0.03	
XRF	89.15		4.26	1.36	0.08	0.16	0.25	
Bias	-3.39		-1.19	-0.45	-0.03	-0.04	-0.22	

Table 1: Chemical analysis of ASCRM035 derived from quantitative phase analysis of XRD data and compared to XRF analysis.



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