Challenging sinter analysis using standardless ED-XRF on samples prepared as pressed pellet

Abstract

Pre-crushed iron ore sinter was prepared with an automatic mill (HP-MA) and an automatic press (HP-PA) in order to provide routine analysis of the chemical composition for the steel production. Here, we present the basic sample preparation used in a workshop acceptance test confirming repeatability for FeO with a standard deviation less than 0.1 %. This was achieved even with a standardless measurement using an ED-XRF.

Key words

• **Sinter** • **Automatic mill (HP-MA)** • **Automatic press (HP-PA)** • **XRF** • **Pressed pellet**

Introduction

Sintering of ore fines, concentrates, flux and coke breeze is a critical feed in the steel making process (SEETHARAMAN et al, 2014). To control the sintering process, several chemical and physical analyses are applied. Beside particle size determination and quantitative phase analysis (XRD – Rietveld analysis), the chemical composition is one of the critical aspects (KÖNIG et al, 2012). Fast and reliable sample preparation for XRF analysis by grinding and pressing is a common analytical technique for process control in steel industries. Here, we present the basic parameter and equipment configuration to achieve a standard deviation of 0.1 % for FeO.

Mill & press configuration

The preparation of sinter requires a specific configuration especially of the mill to meet the material specific and analytical requirements of iron ore sinters. With its abrasive properties the mill must be equipped with a tungsten carbide grinding vessel. To avoid cross contamination between each sample, the mill comes along with several features that can be set accordingly:

- Spoon dosing (blind sample)
- Dry cleaning (compressed air)
- Cleaning with granulate
- Wet cleaning.

Fig.01: Automatic mill (HP-MA) and the automatic press (HP-PA) for sample preparation in semi- or fully automatic laboratories.

Cleaning with a silica free granulate is essential to avoid cross contamination between the subsequent samples and introduction of biasing elements in the cleaning cycle that may remain in the preparation equipment.

Method

Before introducing the sample to the preparation system, approximately 20 g (+/- 0.02g) were weighed. At the end of the pulverization step 6 pills of the grinding aid HMPA 50 were added to the sample before discharging the material from the grinding vessel. The sample material was pulverized in a HP-MA down to a particle size of 90 % smaller than 45 µm.

HP-MA		
Grinding 1	Time:	80 sec.
	Speed:	1470 rpm
	Pills:	0 x
Grinding 2	Time:	20 sec.
	Speed:	1470 rpm
	Pills:	6 x
Discharge	Time:	40 sec.
	Speed:	700 rpm
Grinding aid		HMPA 50
HP-PA		
Pressing	Pressing force:	150 kN
	Ramp up:	10 sec.
	Hold:	5 sec.
	Ramp down:	10 sec.
	Force difference:	75 kN
Steel ring type:		40 mm

Tab.01: Basic parameter settings used with the automatic mill HP-MA and press HP-PA.

Pressing of the ground sample was done in the automatic press HP-PA equipped for a 40 mm steel ring. Before pressing, a defined portion was dosed into the pressing tool. After pressing, the pressed pellet shows a smooth surface (s_a = 0.18 µm / s_z = 1.71 µm). This surface roughness is very close to the surface finish of the counter pressure plate. The surface roughness was determined by using a digital microscope.

XRF measuring conditions

The 10 duplicate samples were measured using the energy dispersive spectrometer Epsilon 3XL with the standardless Omnian measuring program. The measuring conditions are summarized in Table 2, the results in Table 3.

Condition name	kV	Filter	Medium	Measuring mode	time (sec)
Nb-Sb	50.000	Ag	Helium	normal	20
Ni-Zr	50.000	Cu-300	Helium	normal	35
Cr-Co	20.000	Al-200	Helium	normal	20
Cl-V	12.000	Al-50	Helium	High Resolution	40
F-S	5.000	none	Helium	High Resolution	125

Tab.02: Measuring conditions used with the Epsilon 3XL.

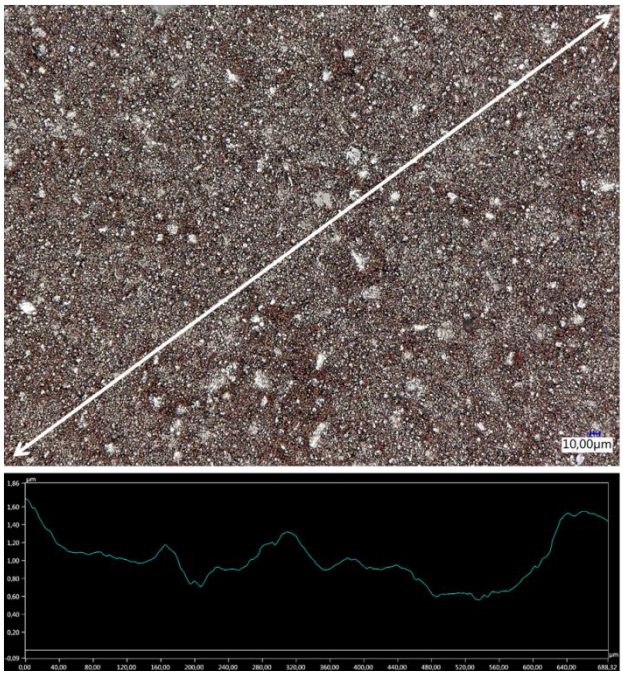

Fig.02: Surface roughness investigation of the pressed pellet using a digital microscope at 1000x magnification.

Automation possibilities

The grinding and pressing can be realized in various degrees of automation. The simplest approach is to use a mill and press as separate machines that are operated manually. To minimize the workload, both machines can be connected via an internal conveyor belt. Finished pressed pellets can be discharged on a magazine or directly forwarded to the XRF spectrometer. To increase capacity, also two mills can be combined with one press. In fully automated laboratories, similar mill and press types can be also integrated into a robotic system. Then, the sample handling is realized by an industrial robot.

The equipment can also be connected to an air tube system which is an additional step towards a fully automated solution. The sinter sample can be pre-crushed at the sampling spot at the

	Fe	CaO	Al_2O_3	SiO_2	P_2O_5	TiO_2	MgO	
Rep 1	70,893	11,585	1,636	6,232	0,039	0,088	1,543	%
Rep 2	70,894	11,579	1,672	6,925	0,039	0,090	1,547	%
Rep 3	70,791	11,525	1,641	6,290	0,039	0,087	1,546	%
Rep 4	70,821	11,519	1,625	6,204	0,039	0,087	1,536	%
Rep 5	71,043	11,624	1,642	6,238	0,041	0,088	1,537	%
Rep 6	70,844	11,561	1,635	6,209	0,039	0,087	1,546	%
Rep 7	70,944	11,565	1,639	6,223	0,039	0,089	1,545	%
Rep 8	71,088	11,631	1,640	6,226	0,039	0,084	1,542	%
Rep 9	71,065	11,612	1,656	6,319	0,039	0,087	1,560	%
Rep 10	71,034	11,590	1,640	6,270	0,039	0,089	1,549	%
Avg	70,942	11,579	1,643	6,314	0,039	0,088	1,545	%
Abs. Std. Dev.	0,109	0,038	0,013	0,218	0,001	0,002	0,007	%

Tab.03: Results from 10 duplicates prepared with the automatic mill (HP-MA) and press (HP-PA) achieved with an ED-XRF without calibration.

plant and send via the air tube carrier to the central laboratory.

Conclusion

The data show that a fast and reliable sample preparation is possible with the automatic mill (HP-MA) and automatic pelletizing press (HP-PA). Various automation concepts can be used to integrate the chemical analysis of sinter feed in a modern process control environment.

Here, we show that basic analytical requirements can be even achieved with non-calibrated ED - XRF spectrometer.

Contact us for further information about automation concepts and check how your laboratory performance may benefit from using even more sensitive and calibrated analytical instruments.

References

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