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Automated Quality Control in the Apatite-Staffelite Ore Benefication

Abstract

Processing samples of different grade can cause contamination during automatic sample preparation for XRF analysis. This report summarizes the trials undertaken to define a satisfying and minimum time consuming sample preparation method for the automatic analysis for process control in apatit-staffelite ore beneficiation.

Keywords

Apatit-staffelite ore • cellophane • XRF • HP-M • HP-P • contamination • automation

Introduction

Froth flotation of apatit-staffelite (collophanite) ore requires a steady process control for a successful beneficiation. For this assignment HERZOG provides fully automatic sample preparation equipment covering slurry drying, sample splitting, fine grinding and pressing into steel rings for XRF analysis (see Fig. 1).

Pretests had shown that during sample preparation contamination arouses by grinding and pressing the basic raw material which has to be beneficiated (Mill feed samples). This can be overcome as the test had shown by running a blind sample or cleaning with quartz sand before grinding the subsequent sample. For fine grinding and pressing the automatic mill HP-M and press HP-P was used.
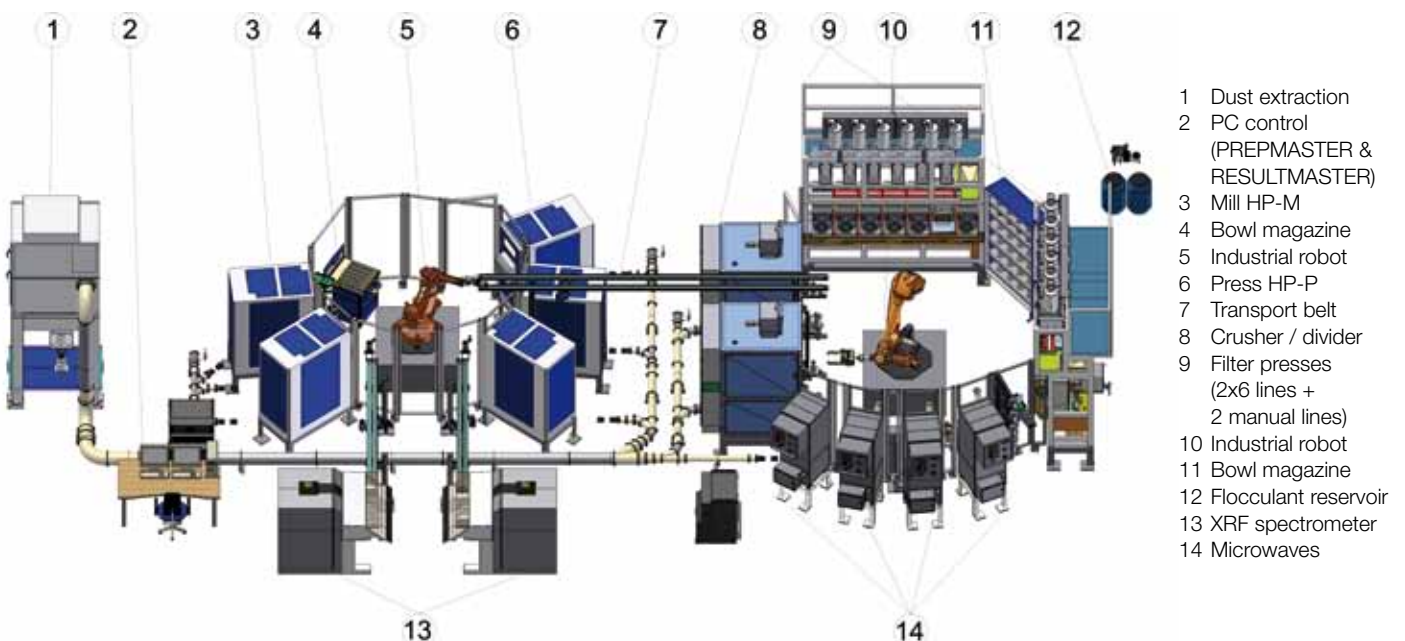

1	Dust extraction
2	PC control (PREPMASTER & RESULTMASTER)
3	Mill HP-M
4	Bowl magazine
5	Industrial robot
6	Press HP-P
7	Transport belt
8	Crusher / divider
9	Filter presses (2x6 lines + 2 manual lines)
10	Industrial robot
11	Bowl magazine
12	Flocculant reservoir
13	XRF spectrometer
14	Microwaves

Fig. 1: Main components of an automated laboratory for quality control for froth flotation of apatite ores.

Sample Material

The sample material includes three different sample types retained from different locations important for the flotation processes. The sample types are following:

- Mill feed
- Phosphate concentrate
- Technology tailings.

The preparation of pressed pellets with HP-M and HP-P is described to favor cross contamination which is characterized by red colored inclusions. Mill Feed and Technology tailings will be processed at one preparation line and the Phosphate concentrate on a separate one.

Methods & machine parameter

To estimate the amount of cross contamination, high grade copper matte (65 % Cu) was added to the sample as internal indicator. This was necessary because the chemical composition of the original sample material doesn't allow clear evidence. Copper was chosen because it's only present in small quantities (± 0.05% Cu). It also has a higher tendency to stick on the grinding vessel compared to the Mill feed sample. This property will support the conclusion that a successful cleaning of mill and press can be also assumed for processing only the original material with even better results. For testing the extent of contamination following composition and sample flow was chosen:

Machine settings

HMPA 40 (HERZOG Mill and Press Additive) was chosen as a grinding aid/binding agent instead of boric acid, because latter was classified as harmful and should be replaced by other additives. HMPA 40 is a successfully used mixture of wax and cellulose for different XRF applications.

Main parameter HP-M:

Grinding time:	60 sec
Grinding aid:	6x HMPA 40
Output time:	40 sec
Cleaning time:	35 sec 700 r/min
Cleaning speed:	1400 r/min
Vessel/puck & ring:	wolfram carbide

Main parameter HP-P:

Pressure:	120 kN
Ramp up:	10 sec
Ramp hold:	10 sec
Ramp down:	10 sec
Pressure difference:	50 kN

Tab.2: Machine parameter of pulverizing and pelletizing machine.

The XRF analysis was done using the EDXRF spectrometer Epsilon 3 XL with the Omnian software for standardless analysis. Following conditions were used for analysis:

Condition name	kV	Filter	Medium	detector mode	Measuring time (sec)
Nb-Sb	50.000	Ag	Helium	normal	15
Ni-Zr	50.000	Cu-300	Helium	normal	30
Cr-Co	20.000	Al-200	Helium	normal	30
Cl-V	12.000	Al-50	Helium	High Resolution	30
F-S	5.000	none	Helium	High Resolution	30

Tab.3: Conditions for XRF analysis.

Test 1		type
Sample A:	12 g sample material + 6x HMPA 40 + 0.3 g copper matte	Mill feed
Sample B:	12 g sample material + 6x HMPA 40	Mill feed

Test 2		
Sample A:	12 g sample material + 6x HMPA 40 + 0.3 g copper matte	Tailings
Sample B:	12 g sample material + 6x HMPA 40	Tailings

Test 3		
Sample A:	20 g sample material + 6x HMPA 40 + 0.2 g copper matte	Tailings
Sample B:	20 g sample material + 6x HMPA 40	Tailings

Test 4		
Sample A:	20 g sample material + 6x HMPA 40 + 0.2 g copper matte	Tailings
Sand cleaning:	20 g quartz sand (QS) milled for 10 sec./ output time 30 sec.	Quartz sand
Sample B:	20 g sample material + 6x HMPA 40	Tailings

Test 5		
Sample A:	20 g sample material + 6x HMPA 40 + 0.2 g copper matte	Mill feed
Blind sample:	12 g of material milled for 10 sec./ output time 30 sec.	Tailings
Sample B:	20 g sample material + 6x HMPA 40	Tailings

Test 6		
Sample A:	40 g sample material + 12x HMPA 40 + 0.4 g copper matte	Mill feed
Blind sample:	10 g of material milled for 10 sec./ output time 30 sec.	Tailings
Sample B:	30 g sample material + 10x HMPA 40	Tailings

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Results

Test 1 and test 2 showed that the biggest problems arise by preparing sample of the Mill feed type. This material has a higher tendency to cause cross contamination than the Technology tailings. For running sample of type Technology tailings a standard air cleaning procedure can be chosen to clean the grinding vessel (test 3). After running a sample of the Mill feed type a blind sample should be used to avoid cross contamination (test 5). Test 6 was set up to produce proof that the results are reproducible with a bigger sample amount which is necessary to allow an automatic grinding with a blind sample included. Detailed information can be found in table 4 and in figure 3.

Sample A			Sample B			Blank Sample		
Com-pound	Conc.	Unit	Com-pound	Conc.	Unit	Com-pound	Conc.	Unit
MgO	6.813	%	MgO	12.952	%	MgO	13.491	%
Al_2O_3	8.496	%	Al_2O_3	11.314	%	Al_2O_3	11.722	%
SiO_2	31.916	%	SiO_2	41.271	%	SiO_2	41.894	%
P_2O_5	15.177	%	P_2O_5	4.241	%	P_2O_5	4.297	%
S	0.108	%	S	0	%	S	0	%
K_2O	1.168	%	K_2O	1.334	%	K_2O	1.34	%
CaO	22.404	%	CaO	12.13	%	CaO	11.886	%
TiO_2	0.44	%	TiO_2	0.565	%	TiO_2	0.556	%
Fe	4.686	%	Fe	5.633	%	Fe	5.609	%
CuO	**0.78**	**%**	**CuO**	**0.024**	**%**	**CuO**	**0.029**	**%**
SrO	0.115	%	SrO	0.083	%	SrO	0.083	%
ZrO_2	0.055	%	ZrO_2	0.062	%	ZrO_2	0.058	%
Cr	0.014	%	Cr	0.016	%	Cr	0.013	%
Mn	0.186	%	Mn	0.18	%	Mn	0.177	%
Zn	0.015	%	Zn	0.015	%	Zn	0.013	%

Tab. 04: XRF samples analysis of the final method check – test 6. The Cu – value (<0.05 %) indicates that the cleaning of the grinding vessel was successful.
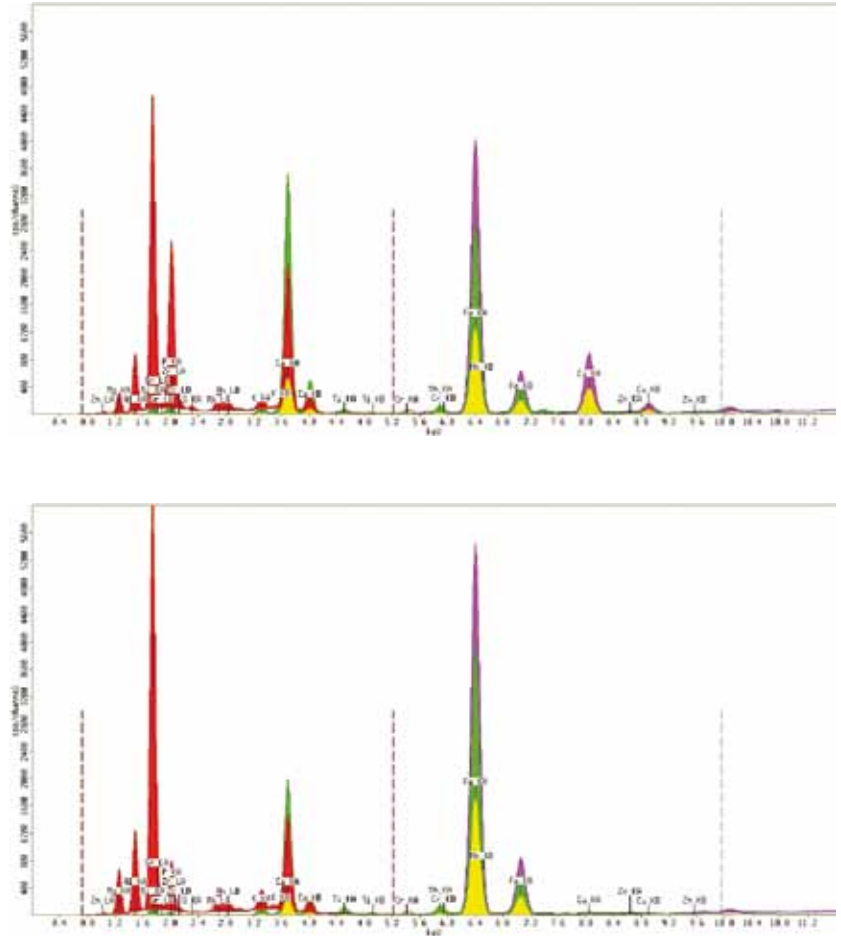

Fig.3: EDX spectra from Sample A (upper figure) and Sample B from Test 6. The EDX spectra show that the contamination of copper (8.040 keV) is successfully overcome by using a blind sample.

Discussion

This application note shows that all process stages for apatit-staffelite (collophanite) ore can be monitored with Herzog equipment. Avoiding contamination between the Mill feed and the Technology tailing samples proved to be the most complex step of sample preparation. However, using a blind sample between each grinding step prohibited contamination. With this method a normal dry cleaning with compressed air is sufficient. Sand cleaning will show the same cleaning results. However, use of a blind sample is more cost-effective and easier.

More information: