

Towards an online energy dispersive X-ray fluorescence analytical system for iron ore grade evaluation

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Abstract Having studied some of the factors that influence the results of iron grade analysis during the iron ore production process, we established an online analytical system based on energy dispersive X-ray fluorescence. The system can determine the iron ore grade within 5 minutes. Compared with similar systems, this online analytical system has the ability to deal with the problems of poor sampling, poor on-site conditions for online iron ore analysis, variation in the moisture content of samples, the matrix effect and spectral drift. The system has been applied to iron ore grade evaluation in the Panzhihua Iron and Steel Company, China. Practical tests illustrated that the system can effectively improve iron ore grade evaluation.

Key words EDXRF, Iron ore, Online analysis, Nondestructive

1 Introduction

To control the quality of iron ore production, iron ore grade should be limited to between 0.52 and 0.58 in the iron ore concentration plant. Thus there is a need for a fast, reliable technique for the analysis of iron grade in iron ore samples. Energy dispersive X-ray fluorescence (EDXRF) is such a technique; it offers fast, nondestructive, and reliable analysis for a wide variety of materials, and the technique has been widely used for elemental analysis of pharmaceutical materials, analysis of heavy metal pollution, analysis of ancient coins, determination of the elements in mineral ores, etc^[1-5].

Previously, we developed an online automatic titanium grade analyzer based on EDXRF technology, and the method proved to be reliable for online quality control in the titanium ore concentrate production process^[6]. Recently we have studied factors that influence EDXRF-based online iron ore analysis at the production site, and we developed an EDXRF-based automatic iron grade analyzer (OAAS-FE) equipped

with an automatic sampling unit and an automatic sample-making unit. In this paper, the OAAS-FE system is introduced, and its industry application at the Panzhihua Iron and Steel Company are discussed.

2 Influencing factors

2.1 Representation of samples

In the iron ore concentration plant of the Panzhihua Iron and Steel Company, the unstable ambient temperature (-10°C to 45°C), humidity in the plant environment, and irregular shapes and sizes of iron ore particles are the most challenging problems affecting accurate measurement^[7]. When production lines employ online analysis based on EDXRF, the excitation source and detector are fixed directly above the conveyor belt (transmission speed is 1.27 m·s⁻¹), and the count rate of the Fe X-ray line changes as the height of iron ore piled on the belt changes. Thus, when carrying out element analysis by the EDXRF method, sample representativeness plays an important role in the iron ore concentrate analysis process.

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As a result of our research, an automatic sampling unit was designed to reduce the impact of this effect. According to modern mechanical and electrical precision control technology, the automatic sampling unit was designed to send iron ore samples into an enclosed space for measurement, reducing the effects of the problems mentioned above. The automatic sampling unit picks up 15 samples every 1.2 minutes. This, to some extent, resolves the problem of poor representative sampling.

2.2 Influence of variation in moisture content in samples

Variation in the moisture content of iron ore influences the intensity of the X-rays^[8]. Thus, the effect of moisture was investigated experimentally as described below.

At 26°C, four different types of iron ore samples were prepared: (1) 55% Fe and 12% moisture, (2) 55% Fe and 14% moisture, (3) 58% Fe and 12% moisture, and (4) 58% Fe and 14% moisture, as listed in Table 1. These iron ore powders were made into 5 mm-thick samples, all under the same condition. The distance between source and sample was 16 mm. The system ran continuously and collected data every 5 minutes for 24 hours.

Over time, the water in the samples evaporates, and the count rate of the Fe X-ray line changes, as shown in Table 1 and Fig.1. We plotted trends in the count rate by linear plus polynomial curve fitting. After 24 hours, the characteristic X-ray count rate of the Fe spectral line for the four types of samples increased from 10% to 11%.

Table 1 Four different iron ore sample types in detail

	Iron ore / g	Fe / %	Water / g	Water / %	Evaporated water / g
Type I	20.0016	55%	2.7643	12%	0.9812
Type II	20.0021	55%	3.4331	14%	1.5817
Type III	24.9922	58%	3.4075	12%	0.7907
Type IV	24.9941	58%	4.0801	14%	1.2492

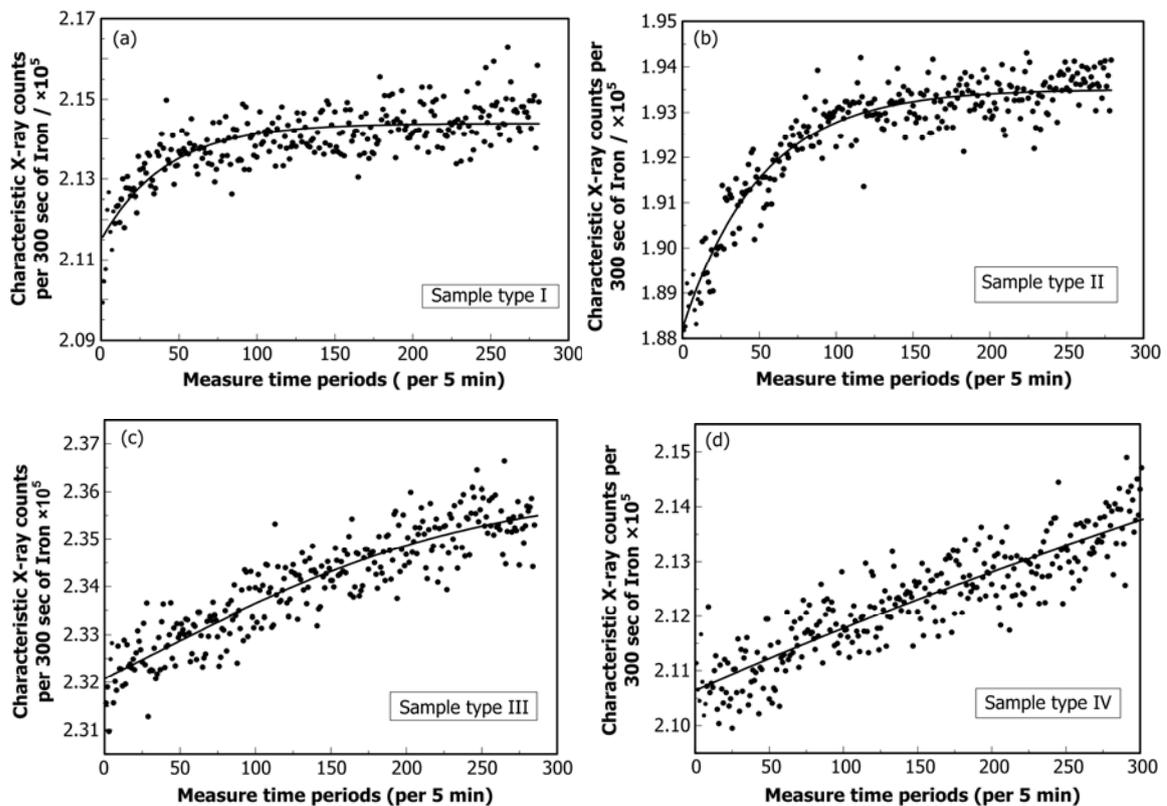


Fig.1 Effects of moisture content in samples on characteristic X-ray count rate. The meaning of the label on the x-axis is the measurement time, and each point result was recorded every 5 minutes and the total measurement time was 300 minutes.

To solve this problem, an automatic sample-making unit was designed to automatically press the water out of samples three times and produce the same surface roughness and uniformity. The effectiveness of the unit was experimentally tested in iron ore samples with 0%, 5%, 10%, 12%, 14%, 16%, 18%, 20%, 22% and 25% moisture, each weighing 25 ± 0.0020 g. Measurement was carried out in two ways: by an X-ray fluorescence analyzer with the automatic sample-making unit (such as OAAS-FE system) and by an X-ray fluorescence analyzer without the automatic sample-making unit. The results, shown in Fig.2, indicate that the system significantly reduces the moisture content in iron ore samples.

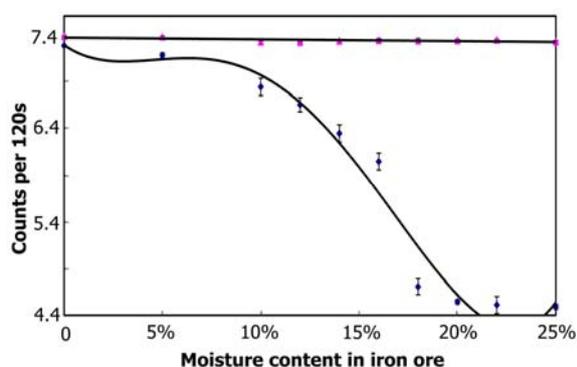


Fig.2 Comparison of measurement by the OAAS-FE system and the X-ray fluorescence analyzer in iron ore samples that differed in water content.

2.3 Matrix effect and spectral drift

By adding an automatic sampling unit and an automatic sample-making unit, the accuracy of the X-ray fluorescence analyzer increased. In addition, a hardware/software-based spectral stabilization method and an advanced ratio measurement technology were used to resolve spectral drift problems.

The system uses an amplifier circuit with a software algorithm to stabilize the spectrum during operation^[9].

In addition, relative analysis technology is applied in the analyzer to resolve the influence of the matrix effect and the complexity of humidity in the environment. The algorithm for element content can be expressed as follows:

$$W_e = \frac{\mu_0 + \mu_e}{KI_0} I_e \quad (1)$$

where W_e is the content of one element; I_0 is the primary fluorescence exposure rate of the excitation source (cps); μ_0 is the absorption coefficient of the sample to primary radiation; μ_e is the absorption coefficient of the sample to the element; I_e is the X-ray count rate of the element; and K is a constant relying on the characteristic of the element (branching ratio, fluorescence yield, geometric effect, detect efficiency, etc.).

It is difficult to obtain accurate coefficients when analyzing one element in compound samples. The calibration equation for the EDXRF method is based mainly on the so-called empirical calibration approach; that is, it is derived from the intensities measured on standards using relative analysis:

$$\frac{W_e}{W_c} = \frac{I_e}{I_c} \Rightarrow W_e = \frac{I_e}{I_c} W_c \quad (2)$$

where I_e and I_c are the element content and X-ray intensity of the standards, respectively. However, the equation is only valid under the condition that the samples and standards are in the same analysis configuration.

A standard with a composition similar to that of the samples in the iron ore concentration plant is fixed in the system. The standard is made by adding 40% (volume ratio) of low-pressure polyethylene powder to iron ore, abrading it with a agate bowl with pressure to 20 tons, heating it for 40 minutes in an oven at 150°C, then pressing again to 20 tons, and finally painting a strong adhesive at the bottom. The standard is measured before every measurement of the iron ore, which efficiently reduces the influence of the matrix effect and the humidity. In addition, it also solves the problem of the decrease in count rate caused by gas consumption in proportional counters.

2.4 Optimal source-sample distance

The distance between source and sample is one of the most important factors in the system. In order to determine the optimal distance, the Fe-K α (6.4 keV) line of a 30-g iron ore sample containing 55% Fe is measured at various source-sample distances from 1 mm to 20 mm.

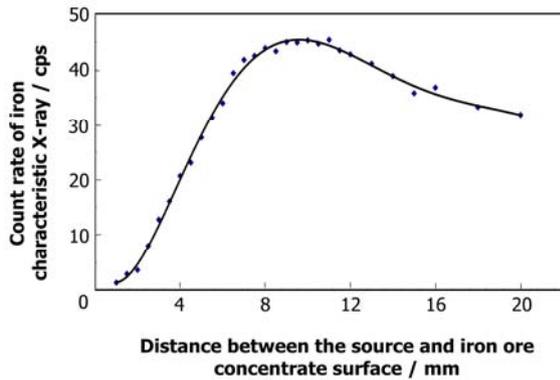


Fig.3 Influence of source-sample distance on the characteristic X-ray count rate.

In Fig.3, the characteristic X-ray count rate of Fe increased with increases in the source-sample

distance between 1 to 9 mm, the count rate reached a peak value at a distance of 9 to 11 mm, and the count rate decreased at a distance of more than 11 mm. From these experimental results, the optimal source-sample distance for the OAAS-FE system was found to be 9.5 mm after fitting the 6th degree polynomials.

3 OAAS-FE System

A schematic diagram of the OAAS-FE system is shown in Fig.4. The system consists of five components: (1) automatic sampling unit, (2) automatic sample-making unit, (3) X-ray fluorescence analyzer, (4) long-distance signal transmission unit and (5) indoor control and display unit.

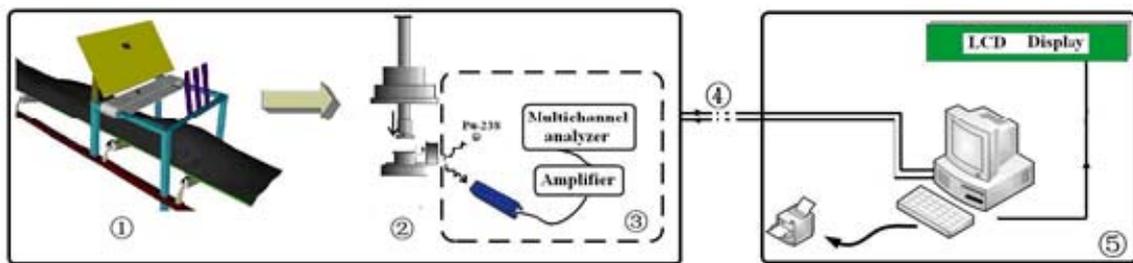


Fig.4 Schematic diagram of OAAS-FE system.

The automatic sampling unit and automatic sample-making unit are key components of the OAAS-FE system, which can reduce the effects of distance variation between source and sample, moisture variation in iron ore, surface irregularity, etc. These two units, by means of advanced precise mechanical and electrical control technology, control the mechanical hand sampling from the fast-running conveyor belt. After obtaining 15 samples, the automatic sample-making unit mixes all samples and presses them three times. The shelf then moves the sample to a certain position to be measured by the EDXRF system. After measurement, the automatic sample-making unit removes the sample and begins the next cycle.

The X-ray fluorescence analyzer takes ^{238}Pu which emits 14–17 keV X-rays as the excitation source. The proportional counter, which is filled with Argon and connected to a 3750-V high-voltage, collects the

characteristic X-rays emitted from the sample. The detector has a 25- μm -thick beryllium window. The detector energy resolution is less than 10% at 5.9 keV (^{55}Fe).

The indoor control and display unit includes a PC with control and analysis software. It is used to control the sampling flow direction, the sample making, movement of the detector and ^{238}Pu source, measurement time, and processing of the data obtained by the detector and multichannel analyzer (MCA). The final online analysis results are displayed on an LCD monitor to guide the production process. Data transfers between the workspace and indoor control room depend on a long-distance signal transmission unit. The total time from sampling to outcome is 4 to 5 minutes. A photograph of the OAAS-FE system installed at the Panzhihua Iron and Steel Company is shown in Fig.5.

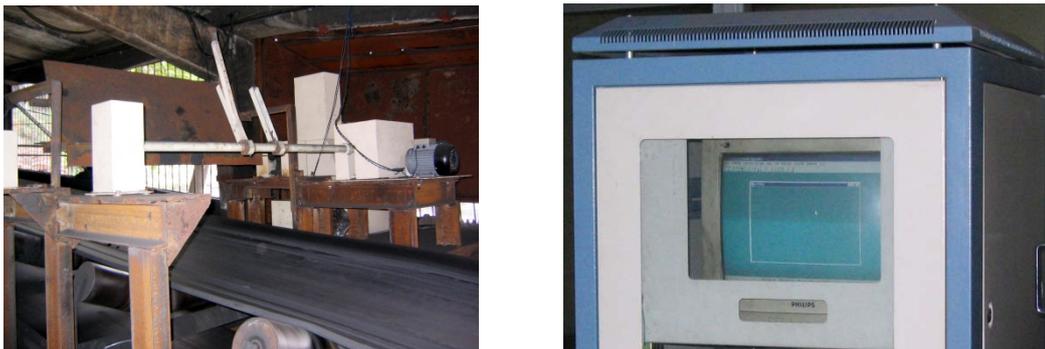


Fig.5 OAAS-FE system installed at the Panzhihua Iron and Steel Company. The image on the left is of the automatic sampling unit; the image on the right shows an analysis output by the PC host on the LCD monitor.

4 Application and stability testing of the OAAS-FE system

The OAAS-FE system was installed at the iron ore concentration plant of the Panzhihua Iron and Steel Company. A stability test was performed by continuously recording counts of Fe spectral line of an

Fe sample for 72 hours, which yielded 1995 data points. As shown in Table 2, the data are normally distributed, demonstrating the long-term operative stability of the OAAS-FE system under normal conditions.

Table 2 Statistical analysis of steady measurement for 72 hours yielding 1995 data points

Mean Value	86879.10	Maximum value	87666.55
Standard Error of the Mean	6.10719	Kurtosis	-0.173
Minimum value	86115.51	Skewness	0.009
		Percentage of data within $\pm \sigma$	68.4%
Standard deviation (σ)	272.78	Percentage of data within $\pm 2\sigma$	95.5%
		Percentage of data within $\pm 3\sigma$	100%

There were 734 effective datasets among the 801 datasets obtained by the OAAS-FE system, and the results were compared to those obtained by chemical analysis. We assumed the chemical analysis values to be true values. The OAAS-FE system operation rate was 95.38%; the pass rate for a Fe-grade test is 95.15% for an absolute error $\leq 0.5\%$, and 91.5% for an absolute error $\leq 0.4\%$.

Errors in analyses based on the OAAS-FE system may come mainly from the following two sources: (1) difference between sample weights used in ordinary chemical analysis (0.2 to 0.5 g) and those used in the OAAS-FE analysis (more than 50 g), and (2) the error of chemical analysis that is superimposed on the OAAS-FE analysis result.

5 Conclusion

An OAAS-FE system based on EDXRF was developed and employed in the analysis of iron ore in the concentration plant of the Panzhihua Iron and Steel Company. The automatic sampling and sample-making units of the OAAS-FE system are capable of providing precise and stable online iron ore analysis. The system can ensure product quality and increase production efficiency, and prospects for its application in the ore concentration industry are good.

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