Online X-ray Elemental Analysis of Coal to Determine Ash, Sulphur, Calorific Value and Volatiles.

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Introduction

Indutech started in 1997 to develop an online analyzer based on energy dispersive XRF technology. Initially the main objective was to determine the sulphur content of coal. The patented technology allows an online XRF elemental analysis with bulky coal accurate with a particle size up to a quarter inch. This allows the calculation of the ash content from the elemental composition of the ash. In combination with a moisture meter, the calorific value can be determined.

Since that time the technology was continuously improved. This results in the following features:

- excellent energy resolution and spectra quality.
- excellent limit of detectivity and accuracy even for low elements such as magnesium, aluminium and silica.
- matrix compensation gives an excellent long-time stability of the calibration
- the Partial Least Square (PLS) regression method makes the calibration simple.
- a new XRF-based method for the ash determination improves accuracy and long-term stability.

Determination of the volatile matter content

Offline tests

The voestalpine Stahl GmbH contacted Indutech with the question, if in addition to the short proximate analysis also the volatiles can be determined to introduce these parameters in the thermal control of the coking ovens. Intensive investigations in Indutech´s laboratories showed, that the information taken by XRF measurements is sufficient to develop a model for describing the volatiles. These tests were carried out at over 100 samples. Six types of 'as received coal' were investigated, coming from Czech Republic and Poland.

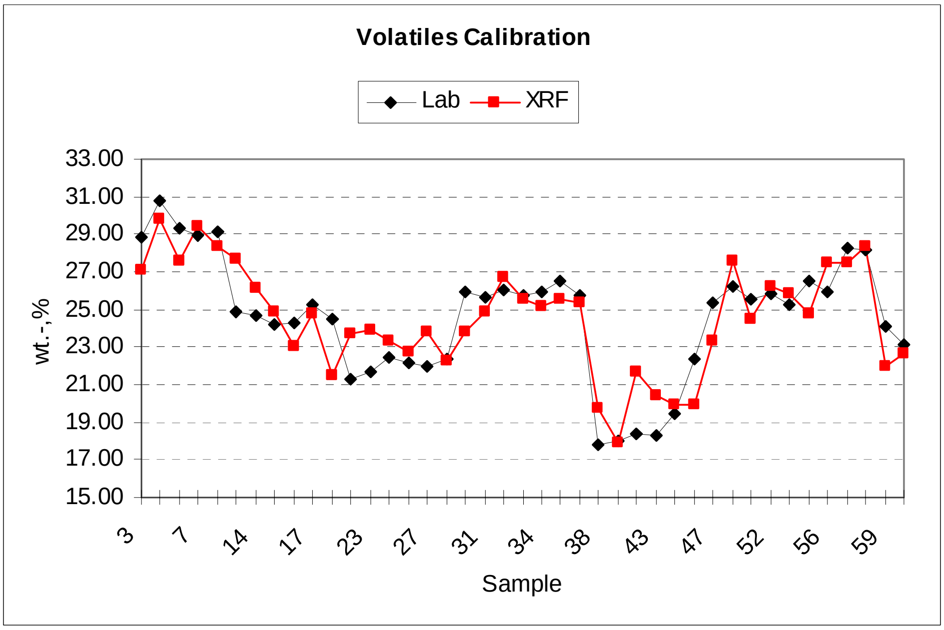

Fig. 1: Volatiles of the received coal

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All types are calibrated with one calibration curve. The accuracy for the volatiles is 1.5 wt.-% at one sigma. Of course, with separate calibration curves the result can be improved remarkably.

The second group of samples were the blended samples. This is the blend, which voestalpine wants to measure. In this case the standard deviation is 0.25 wt.-%, which is absolutely sufficient to control the gas fuel requirement of the coking process.

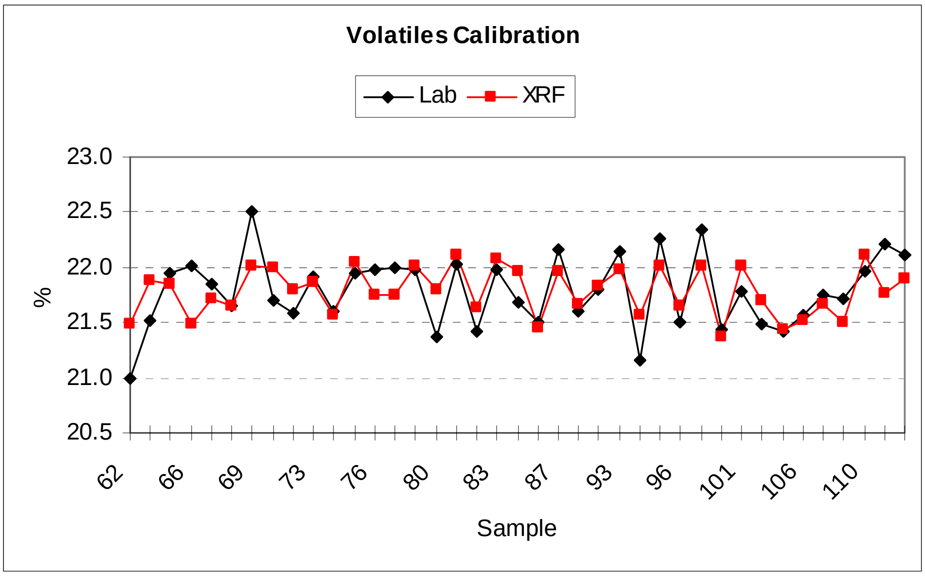

Fig. 2: Volatiles of the blended coal

Online installation and results

Based on these results an OXEA® Online X-ray Elemental Analyzer was ordered in 2003. The analyzer was installed on a sled at the belt transporting the blended coal from the blending facility to the coal towers. The load is 300 t/h, time of operation: 18 h/day, the belt speed is 2.3 m/s. The maximum particle size is less than 10 mm. OXEA® is installed on a sled, which slides upon the material. The sled can swing in arms, which are hinged at the sled as well as at the support frame. This allows the sled to slide always on the material independent of the load, as shown in Fig. 3.

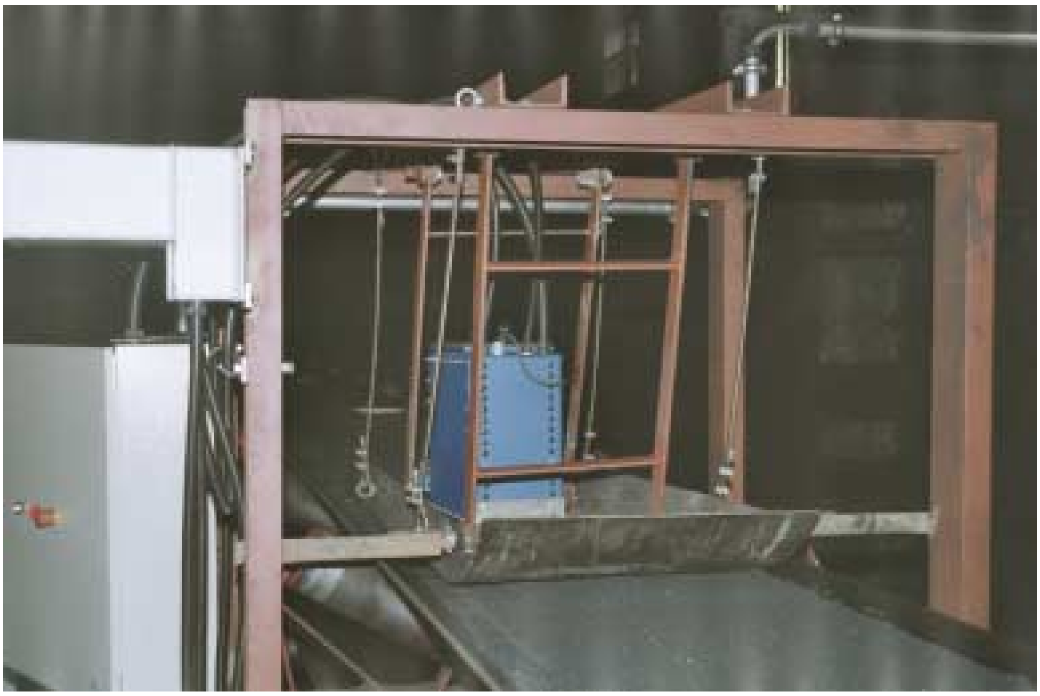

Fig. 3: OXEA® installed on a sled at a main belt of the voestalpine coking plant.

Of course, conditioning of the material stream is required to enable a smooth sliding of the sled. This is done in 3 steps: At first a fixed plough removes the piles on the belt. Then two hinged plates smooth the surface. Figure 4 shows such a hinged plate.

Fig. 4: Hinged plate to smooth the surface

If the belt is empty, the sled is hanging above the belt, so the sled will never touch and damage the belt. Fig. 4 shows the sled with the analyzer in empty state.

Fig. 5: Sled on empty belt

The presence of material is detected by a proximity switch, which also controls the electronic shutter, by which the X-ray tube is switched. The lifetime of the X-ray tube is hereby not reduced.

The figures 6-9 show the online results for ash, volatiles, bulk density and sulphur.

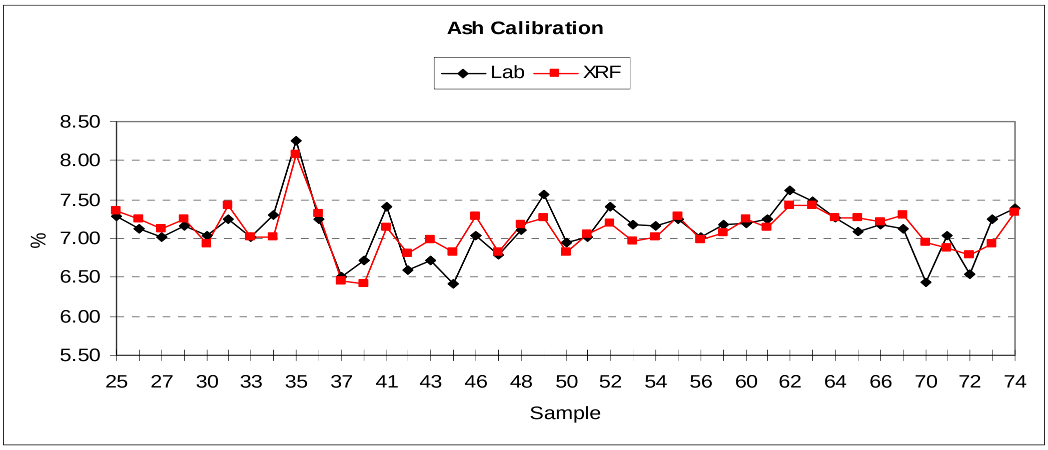

Fig. 6: Online measured ash content

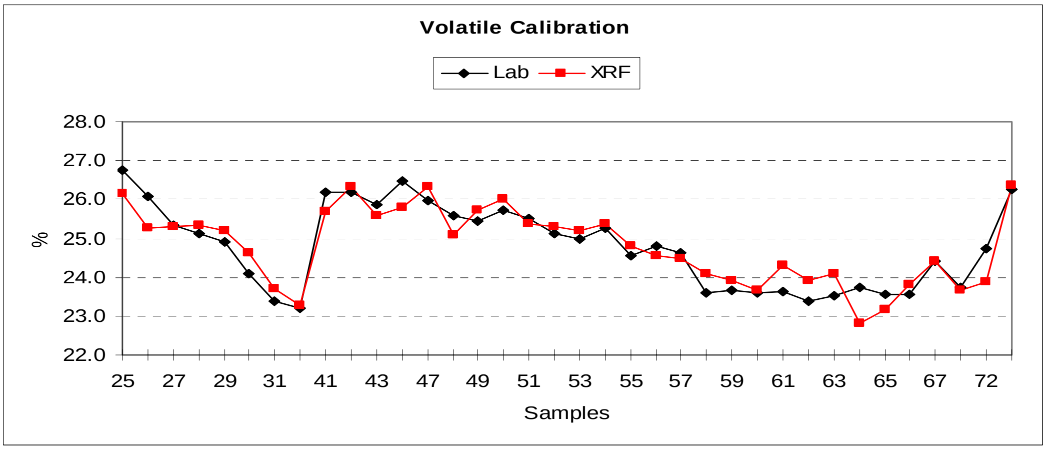

Fig. 7: Online measured volatiles

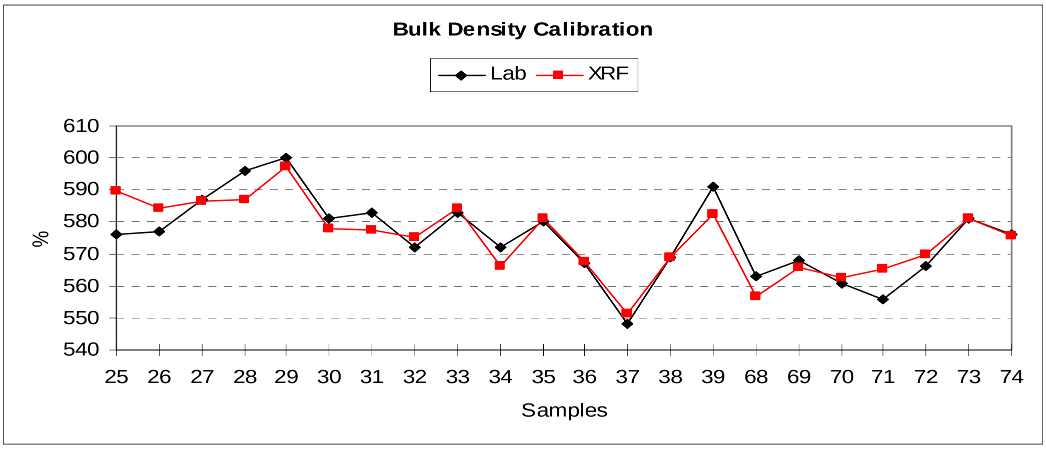

Fig. 8: Online measured bulk density

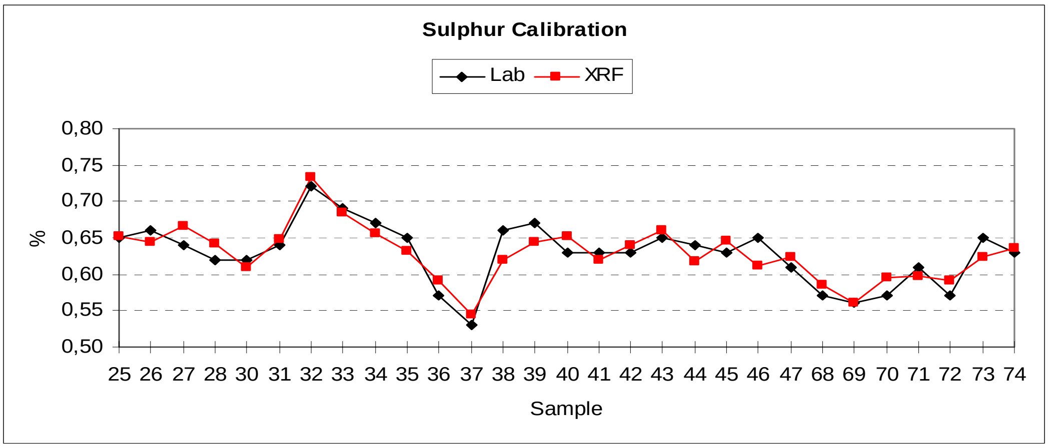

Fig. 9: Online measured sulphur content

Table 1 shows the achieved accuracies of the online calibration for the most important parameters.

	Correlation	Std. Deviation
Ash	0,826	0,192 wt.-%
Volatiles	0,950	0,475 wt.-%
Bulk density	0,899	5,424 kg/m³
Sulphur	0,866	0,019 wt.-%

Table 1: Results of the online calibration

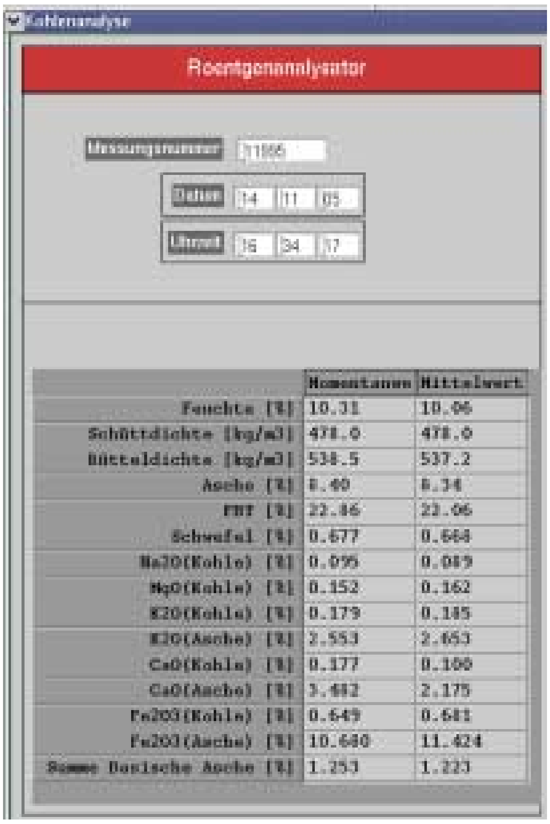

Fig. 10: Numerical display in the control room with all readings given by OXEA®

After the calibration the analyzer was observed by the R&D group of voestalpine. The OXEA® worked absolutely stable. During this final test period the signals of the analyzer were transferred to the PLC of the coking plant and visualized in the control room. The figures Fig. 10 and Fig. 11 show the alphanumerical and graphical visualization in the control room.

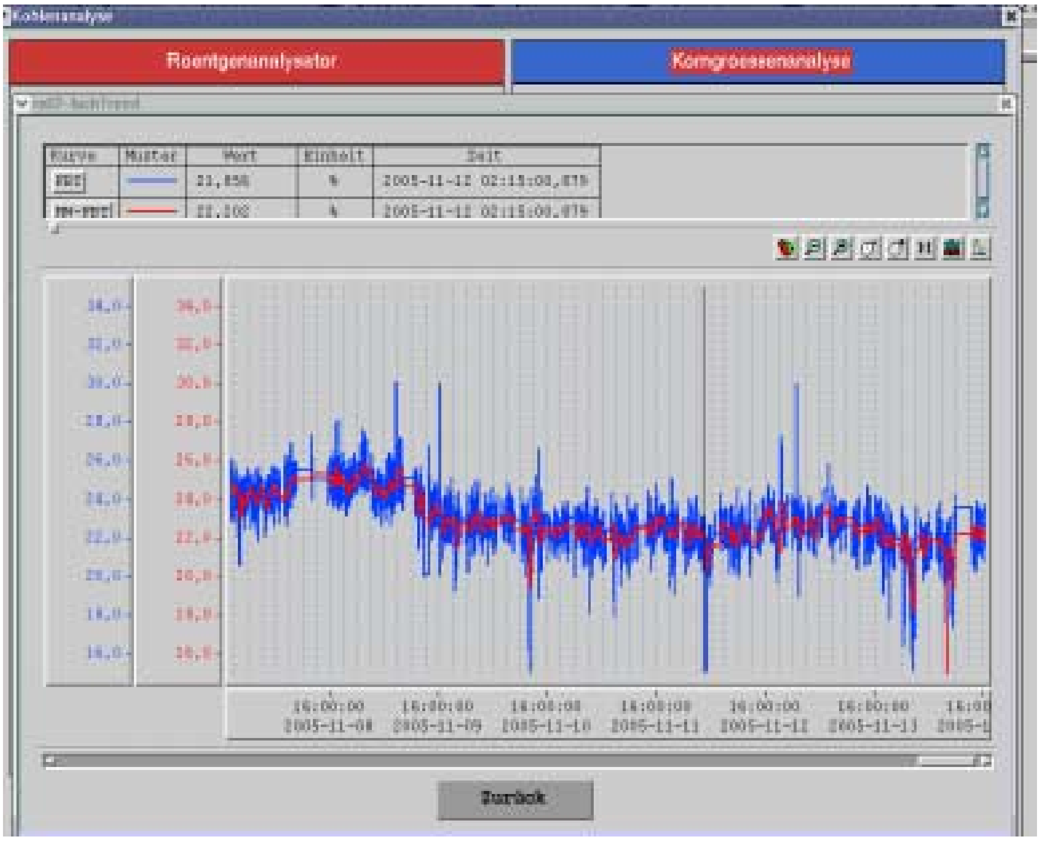

Fig. 11: Trendplot of the volatiles

After a successful online test of the analyzer, the volatiles were switched in a closed loop to control the gas fuel consumption of the coking oven. This test showed that the model for the volatiles works very stable. Adapting the model is only necessary if the received coal types are changed. The analyzer is now running since 5 years without noteworthy problems. The service personal of the coking plant maintains the analyzer. In the last 4 years Indutech was only called twice for service. This shows the high availability of the analyzer.

Importance of online quality control for the coking plant

Before the installation of OXEA® the analysis of ash, sulphur, volatiles and bulk density **was** only available on a daily basis. Fluctuations of individual parameters in the course of a day could not be registered. The elemental composition of the ash was determined once a month only. – The online moisture measurement was already installed.

With OXEA® the volatiles can be determined continuously. The desired final temperature of the coke can be achieved with a low standard deviation, because the required gas fuel can be calculated more exactly. Hereby the consumption of energy is reduces remarkably and the investment for the OXEA® is amortized within a short time. Furthermore, impurities such as ore can be recognized and a reaction is possible to avoid the production of low quality coke.

Long-term Experience

The analyzer now operates for more than 4 years. The stability of the calibration was observed during this period continuously on the basis of the daily samples and was found to be stable over this period, i.e. a recalibration was not necessary. Even after the exchange of a type of coal it was not necessary, to recalibrate the system.

However, recently the recipe of the blending was completely changed. The number of types of coal was increased. Therefore, a new calibration was started in August 2008. The samples were taken and the model for the volatiles will be adapted to the new types of coal once the laboratory results are available. This opportunity was also used to upgrade the system in hard- and software.

Further developments

In 2004 Indutech developed a specific geometry with a helium flush. Hereby the limit of detection and the accuracy of low elements Na, Mg, Al and Si are improved. The helium flush causes, that the height of the Si- peak is twice, the height of the Al peak is three times over. This method was developed within a project to determine low Silicon concentrations in magnesite ore. In the meantime this method is successfully used in different coal applications.

Another coking plant has installed OXEA® to analyze the received coal. In addition to the parameters measured at voestalpine this customer wants to distinguish between the coals of different origin. For this application Indutech developed a fingerprint method, which compares the actual spectrum with spectra of the used types of coal. Of course, impurities are also detected. This analyzer was installed in February 2008. The analyzer is installed at the main belt on a sled too. The maximal particle size is 50 mm and the load variation on the belt is dramatic. In spite of these much stronger installation conditions the analyzer works fine. This shows: the precondition, that the particle size should be less than 10 mm is too restrictive. Specific results about this application will be reported later.

Furthermore, Indutech developed a complete new generation of XRF analyzers. This allows to use the XRF technology for a coal with a particle size of up to about 100 mm.

After the success with the coal analyzer Indutech has also installed several OXEA® for non-coal applications. These applications will be discussed in another paper.

Summary

In this paper it is reported about an online XRF analyzer, which is designed to measure the ash- and sulphur content, the volatiles and the bulk density in a coal blend for coke production. In addition to these parameters, the elemental composition of the material is measured to detect impurities by ore. The analyzer was installed in 2003/ 2004 and is now running for 4 years of continuous use without major problems and an availability over 95%. The model for the volatiles works stable. A recalibration is necessary only if the types of coal are changed. The achieved accuracy allows the use of the readings of the analyzer in a closed loop to calculate the gas fuel consumption for the coking process. Because of the saved energy, the analyzer is amortized within a short period of time.