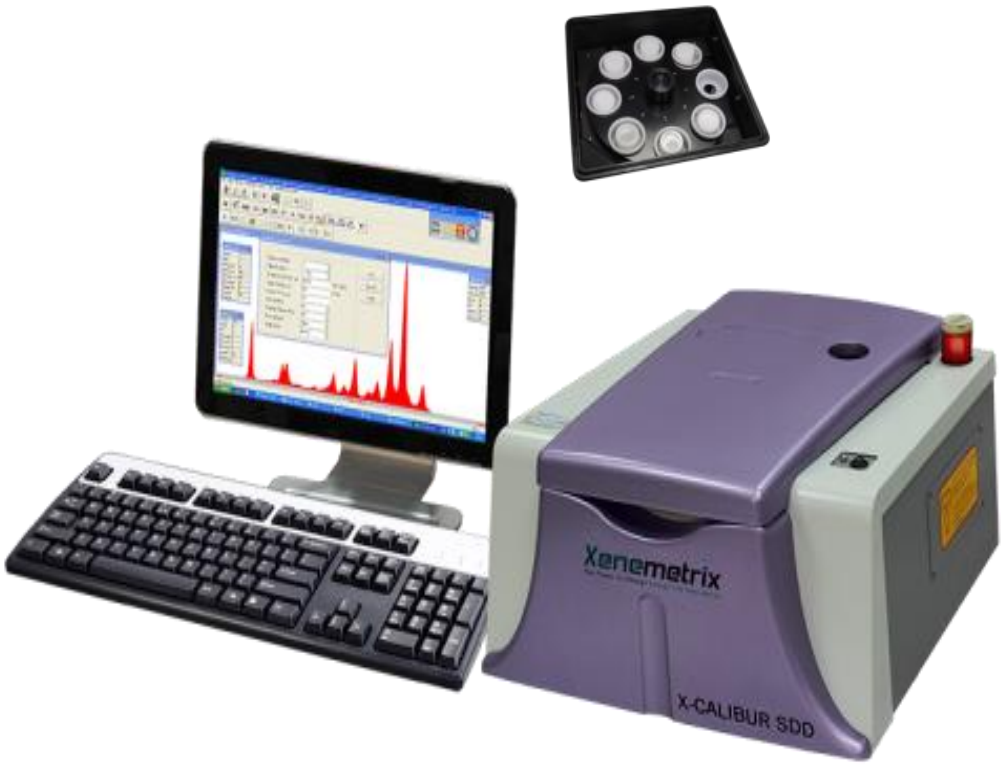
Xenemetrix

The Power to Change Energy Into Information

Quantitative elemental analysis of different cement mixtures

EDXRF Analyzer: **X-Calibur**

Equipped with Silicon Drift Detector (SDD)



Abstract

A set of different mixtures of cement samples were quantitatively analyzed by Xenemetrix X-Calibur benchtop EDXRF analyzer equipped with Silicon Drift Detector. Quantitative elemental analysis was performed using advanced Fundamental Parameter software.

Objective

- To develop quick and robust method for quantify elements content in different mixture samples
- To evaluate the instrument performance and method repeatability

Background

EDXRF is a fast and non-destructive technique that can quantify elemental contents in any type of sample such as solid, powder or liquids within few minutes. EDXRF can play an important role in assuring that consistent quality of samples is retained throughout a manufacturing process.

EDXRF is an ideal method for a quick and simple elemental analysis for industrial control purposes offering the following advantages: 1) Fast and minimal sample preparation, 2) An automated analysis process, 3) Limited or no exposure to corrosive reagents used by other analytical techniques, 4) Ease of use for operation by non-technical or non-specialized personnel.

Analytical Configuration

Table 1: Instrumental analytical configuration

Instrument	X-Calibur
Anode	Rh-Anode X-ray Tube, 50kV,50W
Detector	Silicon Drift Detector (SDD)
Environment	Vacuum
Excitation mode	Direct excitation
Type of analysis	Quantitative analysis
Analysis time	180 sec

EXPERIMENTAL

Five different mixtures of cement samples were received from the customer for elemental quantitative analysis. Samples ID, samples format and mixtures description are shown in table 2 below.

Table 2: Sample list

#	Sample ID	Format	Mixture Description
1	Raw sand	Small stones	Mix of coarse, medium and fine sand
2	Plaster	Powder+ particles	Mix of sand and cement
3	Tile Adhesive	Fine powder	Mix of sand, cement, polymers and other additives
4	Cement	Fine powder	Cement only
5	Skim Coat	Fine powder	Mix of fillers, white cement, polymers and additives

Each sample was received in powder format except the raw sand sample (#1) which contained small stones of variable sizes. In order to enable an optimal XRF analysis, the raw sand was grinded into a fine powder.

The spectrum of each sample was acquired in vacuum environment to avoid the air absorption of low energy fluorescence signal of low molecular weight elements. Typical spectra of raw sand and cement samples are shown in figures 1 and 2 respectively.

The detected elements by qualitative analysis were quantitatively analyzed in "oxide" form using special Fundamental Parameter software for the type of samples where 100% of the elements are not detected in the XRF spectrum such as for example, C and H from the polymer in sample # 3 and 5.
Certified reference standards from NIST were used to calculate theoretical calibration coefficient used in the Fundamental Parameter software and to validate the quantitative analysis.
Two portions of each sample were analyzed. The quantitative results shown in table 3 are average of the two portions.

A Static precision test was performed on the cement sample (#4) to show the performance of X-Calibur Analyzer and the repeatability of the method. The precision was performed by acquiring the spectra ten times without moving the sample between acquisitions. The repeatability results; individual results, measured average± 1 standard deviation and relative standard deviation are shown in table 4.

RESULTS

Qualitative analysis results:

Figure 1a: Typical spectrum of raw sand sample (#1)

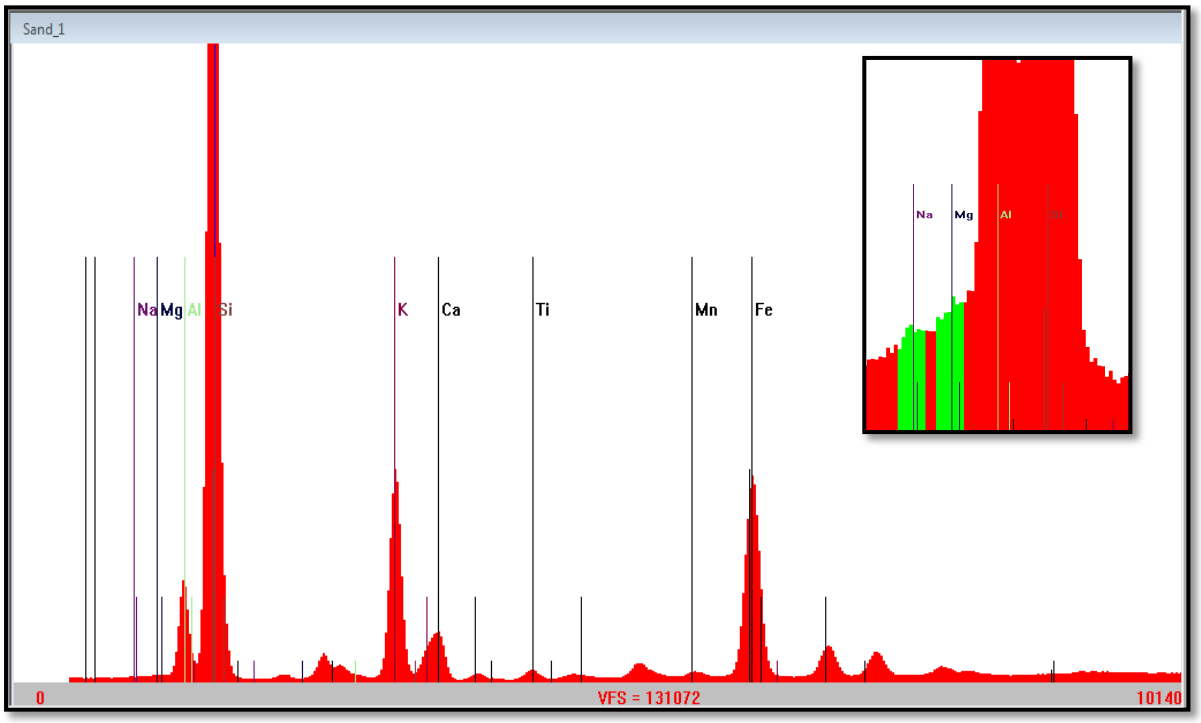


Figure 1b: Continuation of spectrum showing the Sr peak

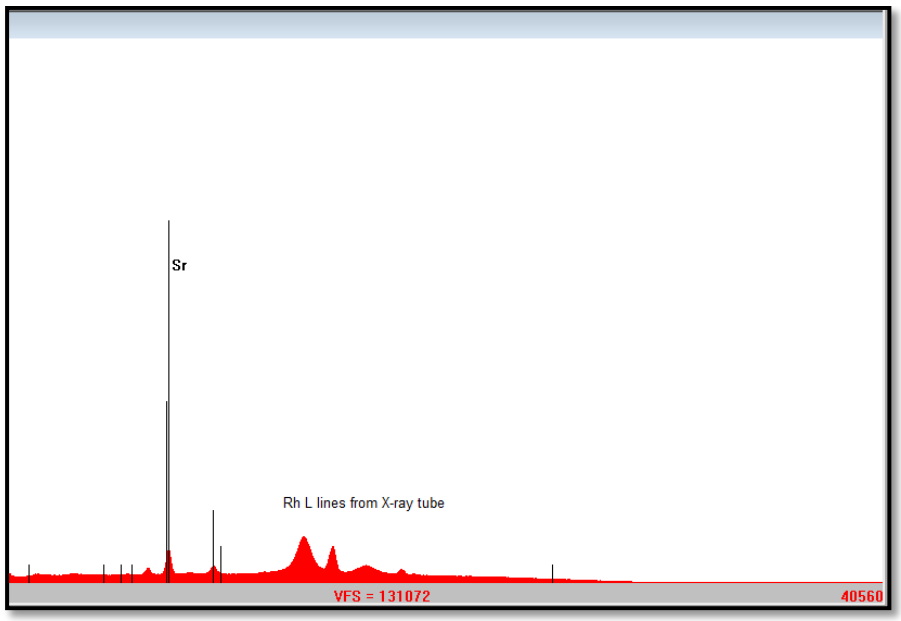


Figure 2a: Typical spectrum of cement sample (#4)

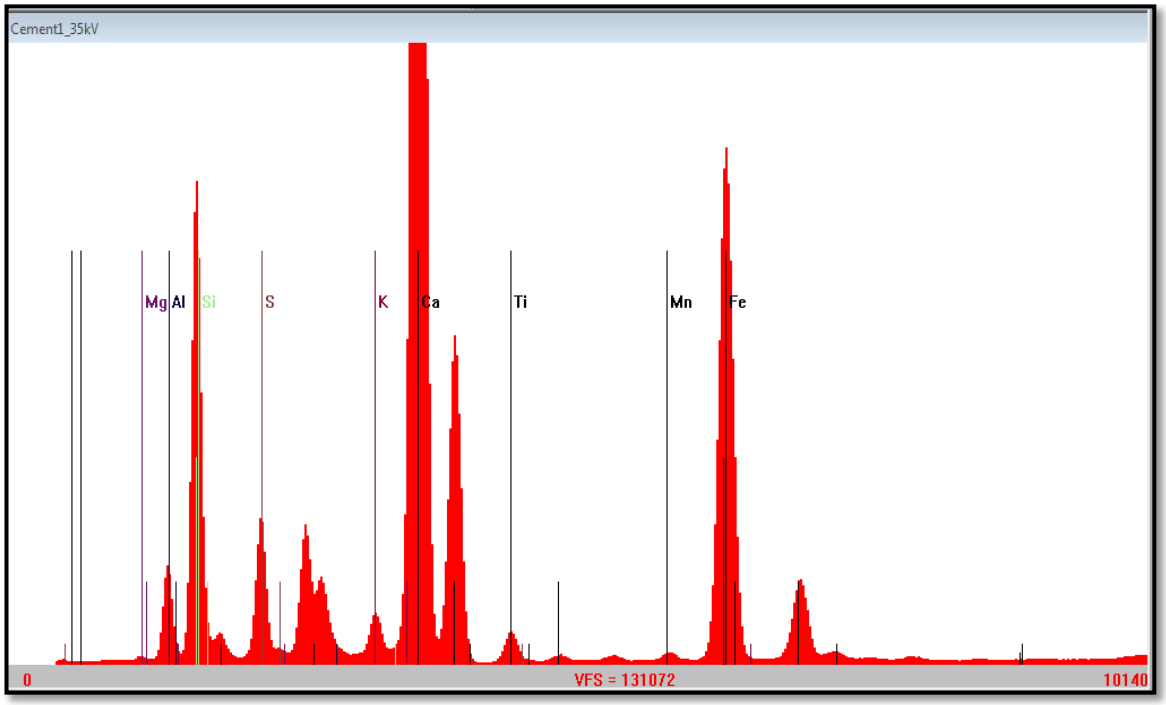
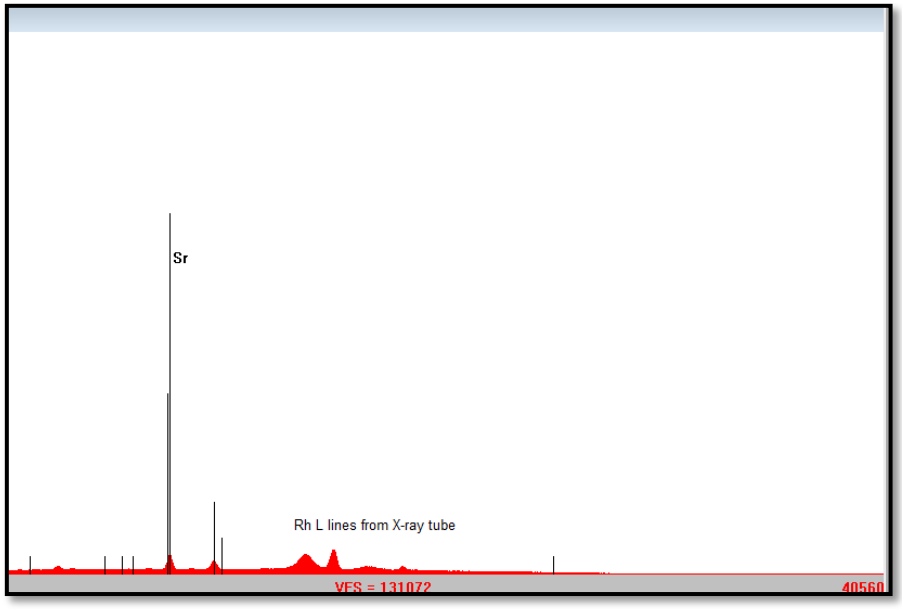


Figure 2b: Continuation of spectrum showing the Sr peak



Quantitative results:

Table 3: Quantitative results

#	Sample ID	Na_2O [wt.%]	MgO [wt.%]	Al_2O_3 [wt.%]	SiO_2 [wt.%]	SO_3 [wt.%]	K_2O [wt.%]	CaO [wt.%]	TiO_2 [wt.%]	Mn_2O_3 [wt.%]	Fe_2O_3 [wt.%]	SrO [wt.%]
1	Raw sand	2.875	1.472	11.085	70.109	N.D	4.580	0.815	0.150	0.033	1.050	0.040
2	Plaster	0.923	1.367	12.968	55.408	0.969	2.795	14.328	0.463	0.046	2.263	0.044
3	Tile Adhesive	0.072	1.21	5.409	26.266	2.474	0.732	43.914	0.54	0.053	2.861	0.046
4	Cement	0.000	1.888	11.571	27.787	2.141	0.904	45.309	0.764	0.080	4.802	0.060
5	Skim Coat	0.108	1.956	1.923	4.428	0.809	0.459	73.271	0.032	0.016	0.214	0.122

Table 4: Static precision test results on cement sample (#4)

Repeat #	MgO [wt.%]	Al_2O_3 [wt.%]	SiO_2 [wt.%]	SO_3 [wt.%]	K_2O [wt.%]	CaO [wt.%]	TiO_2 [wt.%]	Mn_2O_3 [wt.%]	Fe_2O_3 [wt.%]	SrO [wt.%]
1	1.805	11.406	27.892	2.220	0.885	45.079	0.756	0.082	4.808	0.059
2	1.808	11.462	28.006	2.219	0.888	44.990	0.756	0.083	4.804	0.058
3	1.927	11.438	28.081	2.238	1.057	44.371	0.749	0.083	4.855	0.059
4	1.907	11.471	27.983	2.199	0.881	45.014	0.759	0.082	4.811	0.059
5	1.783	11.628	28.277	2.213	0.880	45.051	0.756	0.079	4.839	0.059
6	1.944	11.574	28.129	2.224	0.884	44.995	0.764	0.082	4.852	0.059
7	1.963	11.575	28.435	2.222	0.879	45.069	0.756	0.079	4.848	0.059
8	1.849	11.617	28.274	2.212	0.877	45.003	0.752	0.081	4.826	0.059
9	1.849	11.629	28.657	2.227	0.945	44.526	0.759	0.082	4.893	0.059
10	1.907	11.744	28.649	2.218	0.890	45.112	0.761	0.083	4.873	0.060
Average±Std. Dev [wt.%]	1.874± 0.064	11.554±0.107	28.238±0.271	2.219±0.010	0.907±0.056	44.921±0.255	0.757±0.004	0.082±0.002	4.841±0.029	0.059±0.0005
Rel. Std. Dev.[%]	3.40	0.93	0.96	0.46	6.23	0.57	0.57	1.85	0.60	0.80

CONCLOUSIONS

This work demonstrates the excellent performance of Xenemetrix model EX-Calibur SDD EDXRF combined with advanced Fundamental Parameter software to analyze different cement mixtures.