SETTING UP PROCESSES AND STANDARDIZATION OF THE EQUIPMENT IN ORDER TO OPTIMIZE ANALYSES OF THE WAVELENGTH DISPERSION X-RAY FLUORESCENCE (WDXRF) SYSTEM

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ABSTRACT: For the purpose of operating and optimizing the analyses of the equipment: wavelength dispersion X-ray fluorescence (WDXRF)- model "S8 TIGER" from Enhancing Equipment Project (TCTTB) 2011-2012, we set up sampling and analytical process for different sample kinds; we constructed multi-elemental calibration curve for clay sample; we analysed elemental concentrations of 5 clay samples by XRF method and compared the results with the results given by NAA method. Equipment sensitivity was tested by analysing elemental concentrations of 2 Kaolin standard samples. The results show that S8-Tiger equipment is within good condition and is able to analyze powder clay sample exactly.

I. INTRODUCTION

Since its inception (1964) [1], X-ray fluorescence (XRF) analysis method has made a considerable progress. A lot of research improving the method have been done [4] to develop X - ray fluorescence analysis into one of the powerful methods for determining elemental concentrations - fast and convenient with many advantages. XRF equipment has been innovated to determine the elemental concentration with precision and higher sensitivity, as well as identifying a wide range of elements. XRF method has the advantages such as: compact, fast analysis, does not require a large investment like a nuclear reactor or a neutron generator (neutron activation analysis-NAA). A kind of XRF device based on the principle of wavelength dispersion (WD) has been produced and commercialized. Based on WDXRF equipment, many laboratories around the world have been studying to develop analytical techniques for different sample kinds such as materials, environment, biology, ...

II. THEORY

X-ray beam from Rh-target-tube irradiates sample and excites atoms in the sample. Excited atoms will emit characteristic X-rays. The characteristic X-rays emitted from the sample will be diffracted on a crystal in which distance (d) between the network layer have been known. The crystal will be rotated around one axis to change the angle of diffraction and diffracted beam is recorded by a detector. Bragg's law in X-ray diffraction:

$$n\lambda = 2d\,\mathbf{sin}(\theta)$$

Corresponding to diffractive peaks (2θ), we can deduce the wavelength (λ) of the characteristic X-rays and thereby infer the elements in the sample, which is a qualitative analysis [7]. At the same time, we handle the peaks with many different data processing techniques, then we could obtain relative intensities of the peaks, thereby we determine the elemental concentrations in the sample [7].

III. PROCEDURE

1. Setting up sampling process for different types of samples: solid, liquid, powder...

2. Setting up process of analysis, measurement mode corresponding to different sample kinds.

3. Buying 2 Kaolin standard samples (white clay) for the construction of multi-elemental calibration curve.

4. Experiment: Analysing elemental concentrations of 5 clay samples: 1 clay sample, 2 kaolin samples and 2 bentonite samples encoded: BNVN, SNN, KLTQ, BNAD & SMC by XRF nethod and NAA method collaborating with the Institute for Nuclear Research. Analysing elemental concentrations of 2 Kaoline standard samples by XRF method.

5. Surveying the matrix effects on the results of elemental analysis of the clay samples by XRF method.

IV. RESULTS AND DISCUSSION

Table 1: Result of sample BNVN**.**

Element	Unit	XRF (S8 Tiger)	NAA (ko-NAA)	Note
Si	%	27.03 ± 0.11	29.80 ± 4.70	PGNAA
Al	%	12.52 ± 0.08	10.40 ± 0.20	
Fe	%	5.76 ± 0.02	5.58 ± 0.29	
Ti	Ppm	6,700 ± 111	7,330 ± 1,070	
Mg	Ppm	10,500 ± 224	3,000 ± 160	
Ca	Ppm	8,700 ± 152	6,600 ± 2,000	
Mn	Ppm	600 ± 22	545 ± 5	
K	Ppm	14,500 ± 207	12,000 ± 1,700	

Table 2: Result of sample SNN**.**

Element	Unit	XRF (S8 Tiger)	NAA (ko-NAA)	Note
Si	%	16.68 ± 0.11	ND	ko-NAA ND
Al	%	2.24 ± 0.05	3.03 ± 0.07	
Fe	%	19.91 ± 0.03	17.00 ± 0.05	
Ti	Ppm	2,000 ± 64	<2,500	
Mg	Ppm	36,500 ± 672	1,700 ± 110	
Ca	Ppm	3,300 ± 98	3,420 ± 1,140	

Mn	Ppm	1,500 ± 35	1,240 ± 10	
K	Ppm	1,700 ± 76	1,790 ± 210	

Table 3: Result of sample KLTQ**.**

Element	Unit	XRF (S8 Tiger)	NAA (ko-NAA)	Note
Si	%	19.13 ± 0.12	22.20 ± 3.70	PGNAA
Al	%	21.70 ± 0.13	23.50 ± 0.34	
Fe	%	0.42 ± 0,01	0.42 ± 0.01	
Ti	Ppm	5,300 ± 105	4,600 ± 900	
Mg	Ppm	ND	5,690 ± 170	
Ca	Ppm	800 ± 53	<1,200	
Mn	Ppm	ND	8 ± 1	
K	Ppm	900 ± 57	600 ± 110	

Table 4: Result of sample BNAD.

Element	Unit	XRF (S8 Tiger)	NAA (ko-NAA)	Note
Si	%	24.70 ± 0.07	24.20 ± 3.40	PGNAA
Al	%	10.42 ± 0.05	8.79 ± 0.12	
Fe	%	7.08 ± 0.01	6.85 ± 0.03	
Ti	Ppm	12,900 ± 94	10,000 ± 1,100	
Mg	Ppm	13,000 ± 173	2,400 ± 160	
Ca	Ppm	3,600 ± 60	<1,200	
Mn	Ppm	800 ± 15	840 ± 10	
K	Ppm	9,400 ± 103	8,270 ± 2,100	

Table 5: Result of sample SMC**.**

Element	Unit	XRF (S8 Tiger)	NAA (ko-NAA)	Note
Si	%	17.92 ± 0.12	23.30 ± 3.40	PGNAA
Al	%	2.48 ± 0.05	2.83 ± 0.04	
Fe	%	20.48 ± 0.03	17.50 ± 0.04	
Ti	Ppm	1,700 ± 60	<1,900	
Mg	Ppm	37,500 ± 690	1,380 ± 90	
Ca	Ppm	2,800 ± 90	<2,200	
Mn	Ppm	1,500 ± 34	1,370 ± 10	
K	Ppm	1,700 ± 76	1,570 ± 130	

Table 1 - Table 5 show results of the elemental analysis of the clay samples by XRF method and by NAA method. Most of the values given by the XRF method are in good agreement with the values given by the NAA method, with an exception of Mg. The analytical results of Mg are completely different between the two methods. This is due to matrix effect occurring in the clay samples. Magnesium absorbs the energy Al Kα1 and Si Kα1, therefore Mg Kα1 peak is significantly enhanced. We can fix this by setting standard curve for magnesium.

Table 6: Result of standard sample NCSDC60122 (GBW 03121) in Full Analysis mode.

Element	Certified value (%)	Analytical result (%)
SiO_2	54.55 ± 0.17	50.71 ± 0.23
Al_2O_3	31.41 ± 0.11	35.71 ± 0.17
Fe_2O_3	0.5 ± 0.03	0.46 ± 0.01
CaO	0.052 ± 0.008	0.06 ± 0.01
MgO	0.12 ± 0.02	0.10 ± 0.02
K_2O	0.34 ± 0.02	0.34 ± 0.01
Na_2O	0.015 ± 0.004	ND
TiO_2	0.69 ± 0.03	0.79 ± 0.02
MnO	0.0032 ± 0.0003	ND
P_2O_5	0.099 ± 0.009	0.11 ± 0.01
SO_3	0.53 ± 0.04	0.50 ± 0.02

Table 7: Result of standard sample NCSDC60123 (GBW 03122) in Best Detection mode.

Element	Certified value (%)	Analytical result (%)
SiO_2	44.53 ± 0.17	43.50 ± 0.12
Al_2O_3	38.62 ± 0.10	38.55 ± 0.10
Fe_2O_3	0.72 ± 0.04	0.85 ± 0.01
CaO	0.16 ± 0.03	0.16 ± 0.01
MgO	0.068 ± 0.005	0.050 ± 0.008
K_2O	0.049 ± 0.007	0.060 ± 0.003
Na_2O	0.069 ± 0.006	0.050 ± 0.010
TiO_2	0.39 ± 0.02	0.42 ± 0.01
MnO	0.0054 ± 0.0011	0.0068 ± 0.0008
P_2O_5	0.21 ± 0.02	0.25 ± 0.01
SO_3	0.12 ± 0.01	0.22 ± 0.01

The analysis results of standard samples in the table 6 and 7 show that elemental content is in good agreement with certified value. However, on the Full Analysis mode, the analysis capability (sensitivity) of the equipment is limited to hundreds of ppm, such as MnO and Na2O content as show in table 6. The Best Detection mode has the best sensitivity, it's around tens of ppm which can detect the MnO content (54ppm) as show in table 7.

V. CONCLUSSION

The project has been done with these contents: setting up sampling process for different sample kinds; setting up process of analysis; constructing multi-elemental calibration curve for clay sample; analysing elemental concentrations of 5 clay samples by XRF method and comparing the results with the results given by NAA method; analysing elemental concentrations of 2 Kaoline standard for testing equipment sensitivity. The above results show that XRF (S8-Tiger) equipment is within good condition and is able to analyze powder clay sample exactly.

The application of XRF S8-Tiger equipment after the end of the project

- Undertaking XRF analyses in Center for Nuclear Techniques in HCM city.
- Participating in Institute Project 2014.
- Participating in Nafosted Project 2014-2015.

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