

ELEMENTAL ANALYSIS OF SOME CLAY SAMPELS BY A MODIFIED X-RAY FLUORESCENCE TECHNIQUE

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ABSTRACT

Elemental analysis of several Malaysian clays have been carried out using x-ray fluorescence technique with a modified sample preparation method. To minimize concentration and matrix effect, the dilution technique is employed in the preparation of samples for analysis. The results obtained show reasonable accuracy and repeatability.

INTRODUCTION

Due to its wide range of usage, clay has been considered as an important material for man. Manufacturing of building materials and pottery industry are two of the most important activities related to clay. Research on clay has become an interdisciplinary field involving Physics, Geology, Chemistry, Agricultural Science, Mineral Science, etc. The emphasis of most of the studies range from mineralogical studies, elemental analysis, up to studies on both chemical and physical properties of certain type of clays. For these studies, various methods have been employed. For elemental or chemical analysis, the most common method used is the wet chemical method. However, instrumental determinative methods such as x-ray Fluorescence (XRF) and neutron activation analysis have also been used. In some laboratories these methods are carried out on routine basis.

For the present study, XRF analysis was used for analysis of some Malaysian clays that are used in the building materials and pottery industries. The samples were obtained from various parts of North-Western region of Peninsular Malaysia.

EXPERIMENTAL

Sample Preparation

In this study, the dilution method was used in preparing the samples. 0.6 g of the sample which had been dried and pulverised, was mixed with 5.4 g analytical grade boric acid. The boric acid has also been dried and ground earlier. The mixture was compressed in a stainless steel die with a pressure of 20 tons, to form a circular disc of 4-cm in diameter and 0.5 cm in thickness.

For the preparation of the standards, known amounts of analytical grade of Al₂O₃, SiO₂, TiO₂, KCl, CaHPO₄ and Fe₂O₃ were mixed, homogenised and pulverised in the same manner as the samples. The percentage amount of these compounds in the mixture were made to be close to that of typical clay. The mixture was then divided into different portions and mixed with boric acid to produce a series of 6 gram standard with different elemental concentration. They were then prepared into disc form in the same manner as the samples.

Instrumentation

For the study, a Philip Model PW1410 XRF machine was used. The specifications employed were:

Target	: Chromium
Analyzing Crystal	: Pentaerythritol crystal (2d = 8.742 °Å)
Tube Ratings	: 50 kV 40 mA
Detector	: Proportional counter
Scanning Rate	: 1° /min
2θ range	: 58° to 147°
Path	: Vacuum

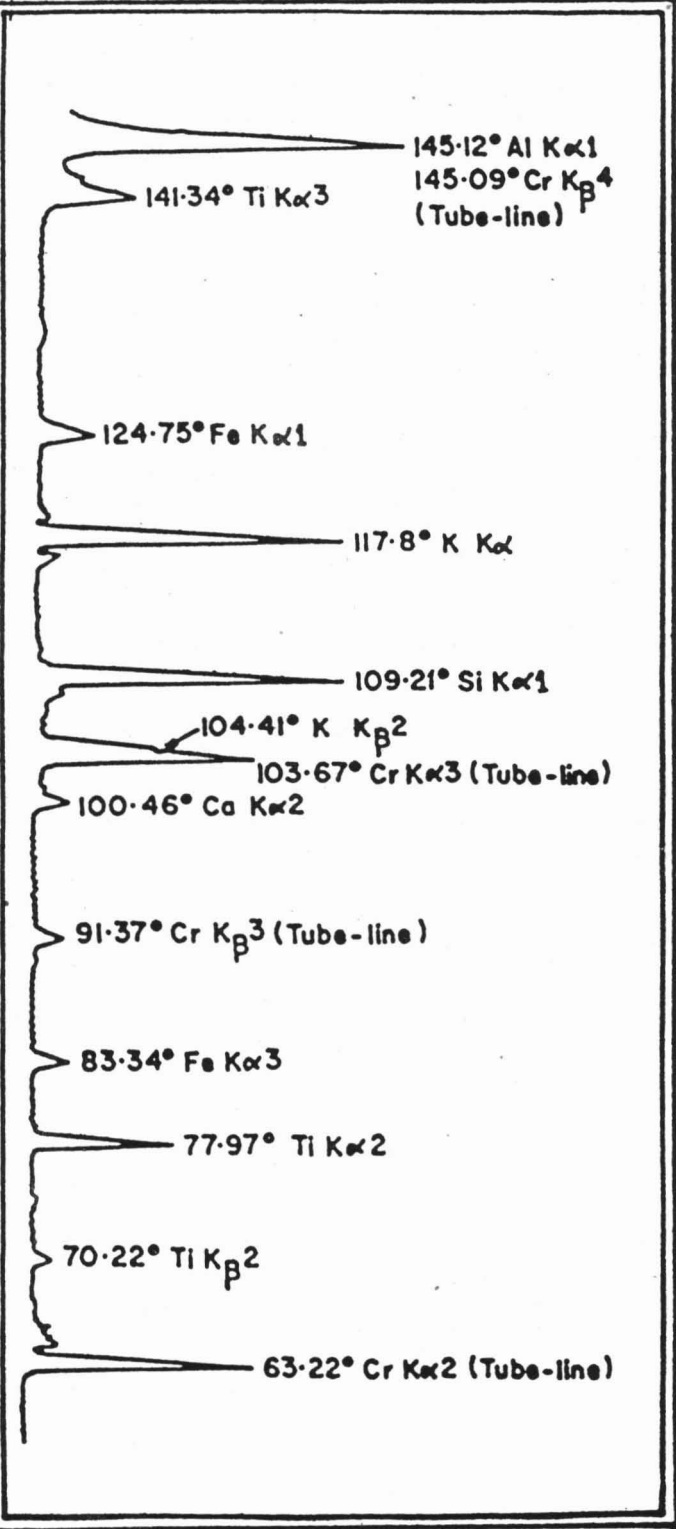
With these specifications, both major and minor elements in the clay could be detected. A preliminary XRF study was also carried out using LiF-200 crystal analyser and a scintillation counter to detect elements of higher Z values; the results showed that these elements were not present in the samples.

RESULTS AND DISCUSSIONS

The prepared standards were used to obtain the calibration curves for Al, Si, Ti, K, Ca and Fe for the experimental specifications mentioned earlier. All calibration curves were straight lines with correlation factor of $r < 0.98$. This indicates that variation in compositional percentage of the element concerned has little effect upon direct correlation of the elemental concentrations and the fluorescence peak intensities. Hence the elemental concentration can be obtained directly from the calibration curves [Ahmad (1988)]. Figure 1 shows an x-ray fluorescence spectrum of a clay sample. Here fluorescent peaks of the major and minor elements in the sample can be seen clearly, and these peaks together with the calibration curves can be used to determine the concentration of such elements.

The chemical contents of the samples under study are listed in Table 1. The deviations indicated together with the data are estimates of experimental error contributed by the calibration curves and the relative peak heights in the spectrum concerned. These results are normally reproducible on any one day, but may vary slightly from day to day due to the change in the x-ray machine conditions. As can be seen in Table 2, the results of repeatability test on three

Figure 1 : X-ray fluorescence spectrum of a clay sample.



samples show a considerably good agreement between the two test. This also indicates that the samples were quite homogenous.

The reliability of the present analysis was checked using a synthetic sample prepared in this laboratory. The results shown in Table 3 are quite satisfactory for all the compounds investigated. However, it has to be mentioned here that the matrix of the synthetic sample was quite different from that of typical clay.

From the results of this study, it was found that the analyses performed by XRF on samples prepared by the dilution method provide a reasonably accurate chemical analysis of clay. However, it should be made clear that the method was carried out with the assumption that the matrix effects constitutes the single source of inaccuracy. In practice, compositional diversity [Baird and Welday (1968)] and instrumental instability [Shio-Chuan Sun (1959)] might also contribute significantly towards the inaccuracy. Moreover, as stated by Murad (1973), the dilution method would not totally eliminates interelements effect. Thus, this explained why variations up to 10% are found in Table 2 and Table 3.

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Table 1 : Chemical contents of some Malaysia clays

Contents % Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ *	TiO ₂	K ₂ O	LOI**
SP ₁	65.6 ± 4.5	25.5 ± 2.3	0.23 ± 0.02	1.88 ± 0.13	4.09 ± 0.27	5.2 ± 0.1
SDB ₁	69.4 ± 4.9	26.1 ± 2.3	0.23 ± 0.02	0.63 ± 0.05	0.59 ± 0.04	6.4 ± 0.1
BS ₁	59.8 ± 4.3	20.4 ± 1.9	2.93 ± 0.20	0.55 ± 0.03	1.01 ± 0.07	9.7 ± 0.2
S ₁	59.4 ± 4.1	23.8 ± 2.1	0.77 ± 0.06	0.13 ± 0.01	2.80 ± 0.19	8.2 ± 0.2
P ₁	64.5 ± 4.5	30.0 ± 2.6	0.76 ± 0.06	0.32 ± 0.02	2.51 ± 0.18	6.8 ± 0.1
V ₁	53.1 ± 3.6	32.7 ± 3.0	0.41 ± 0.03	0.35 ± 0.02	0.99 ± 0.07	8.2 ± 0.2
K ₁	68.1 ± 4.7	29.3 ± 2.6	0.13 ± 0.01	0.18 ± 0.02	0.54 ± 0.04	4.7 ± 0.1
TK ₁	57.6 ± 4.1	35.9 ± 3.2	0.51 ± 0.04	0.22 ± 0.02	3.01 ± 0.22	4.9 ± 0.1
TB ₁	57.2 ± 4.1	31.0 ± 2.8	0.37 ± 0.03	0.22 ± 0.02	2.98 ± 0.22	8.9 ± 0.2
KT ₁	58.7 ± 4.1	31.4 ± 2.8	0.03 ± 0.21	0.45 ± 0.03	1.80 ± 0.12	7.7 ± 0.2

* Iron is assumed to be in the form of Fe₂O₃ only.

** Values obtained by firing the sample at 600°C for two hours. LOI means loss on ignition.

Table 2 : Repeated estimations of chemical contents of clay sample

Contents % Sample		Chemical contents (%)				
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃ *	TiO ₂	K ₂ O
TK ₁	1	57.6 ± 4.1	35.9 ± 3.2	0.51 ± 0.04	0.22 ± 0.02	3.01 ± 0.22
	2	58.9 ± 4.1	37.9 ± 3.4	0.43 ± 0.03	0.22 ± 0.02	3.01 ± 0.22
S ₁	1	59.4 ± 4.1	23.8 ± 2.1	0.77 ± 0.06	0.13 ± 0.01	2.80 ± 0.19
	2	59.8 ± 4.2	25.1 ± 2.3	0.71 ± 0.06	0.15 ± 0.02	2.72 ± 0.19
TB ₁	1	57.2 ± 4.1	31.0 ± 2.8	0.37 ± 0.03	0.22 ± 0.02	2.98 ± 0.22
	2	52.8 ± 3.6	30.6 ± 2.8	0.33 ± 0.03	0.23 ± 0.02	2.70 ± 0.19

* Iron is assumed to be in the form of FE, O, only

Table 3 : Chemical contents of a synthetic sample determined by this procedure

Compound	Actual percentage	This method (%)
SiO₂	49.9	52.0 ± 3.9
Al₂O₃	48.7	47.8 ± 3.9
Fe₂O₃	0.35	0.37 ± 0.03
TiO₂	0.35	0.31 ± 0.02
K₂O	0.30	0.31 ± 0.02
CaHPO₄	0.27	0.31 ± 0.03