

# Instrumental Characterization of Pretoria Clay Soil by XRF, XRD and SEM

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## Abstract

The products of refractory materials are used for lining furnace, incinerators and kilns among other uses and they have the potential of withstanding high temperature without deformation. The objective of the research was to characterize the clay soil sample collected from Tshwane University of Technology in Pretoria. The sample, collected from a location Latitude 25.0969° S and Longitude 28.1624° E, was oven-dried, pulverized and sieved in the laboratory. Mineralogical and elemental compositions of the sample were determined by using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM) analytical methods. The XRF analysis revealed Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> as the major constituents, while the other elements occur in minor quantities. Mineralogically, the three samples contain Montmorillonite, Kaolinite and Bentonite, while Samples A contained Quartz in addition to earlier mentioned minerals, samples B and C contained Albite.

## Keywords

Characterization, Clay Minerals, Soil, XRD, XRF, SEM

## 1. Introduction

Refractory material has the potential of withstanding high temperature without deformation or breaking. The products of such materials are used for linings kilns, incinerators and furnace among others. They depend on their chemical nature which is neutral [1] [2]. Properties of refractory materials include: increase in melting points associated with their stability and high resistance to corrosion by slags. Research works have been carried out on the development of good refractory materials mostly in western countries [3] [4] [5] [6]. The out-

come shows that refractory materials are of utmost importance in the world of today. Clays are naturally occurring material composed of fine-grained minerals. They become hard at appropriate water content when fired, thereby exhibiting plasticity [7] [8]. Clay deposit was discovered more than ten thousand years ago in Africa [3]. They are useful in construction (building of houses), pottery and pore clays. Furthermore, the low prices of clay materials, make them mostly useful as adsorbents. When the structure of clay minerals occurs, there is an obvious net negative charge which comes from the capability of their absorption. Meanwhile, the surface area and the height also produce their absorption [9]. When chemical weathering of silicate minerals occurs at the earth surface, clay minerals are produced, which develop the percentage (40%) of the fine-grained sedimentary rocks which include clay stones. The clay deposit from the Tshwane University of Technology, Pretoria campus has many important uses including floor tiles and bricks making. When Clay is dried in the sun it can be used to produce bricks, utilized in the construction of mud houses and when combined with sand, it could be used as moulds, which are used for casting projects [4]. Various researches have been conducted on clay mineral analysis. These researches revealed the presence of a wide spectrum of minerals [10] [11]. Furthermore, other analyses [5] [6] [12] have revealed, in general, the presence by analyzing clay samples using XRD, XRF, SEM. Clay materials consist of alumina, silica, water as well as some quantities of iron and alkaline earth metals [13]. Generally, clay minerals bond is formed by both the tetrahedral sheets which are linked together through the sharing of apical oxygen atom.

Some research works done on clay using the Scanning Electron Microscope (SEM) have revealed that it obtains much powers of magnification than the standard visible light microscope [3] [4] [5] [6]. This is because the electrons are associated with a very short wavelength than the light wave, while the X-ray Diffraction analysis on clay was duly corrected by appropriate factors accounting for the variation of scattering due to their various angle. The aim of this study is to characterize as well as to critically investigate the nature of the clay soil from the study area.

## 2. Materials and Methodology

Clay soil sample was collected from the study area, Latitude 25.0969°S and Longitude 28.1624°E, about 1 km from the main campus of Tshwane University of Technology, Pretoria and adjacent to Building 3 Main Campus. The sample was collected using sampling cups.

In the laboratory, the sample was oven-dried at 115°C for 24 hours. The dried sample was later pulverized using Rawley Sussex grinder to obtain 500 µm particle size.

### 2.1. Sieve Analysis

The sample was sieved using pre-arranged sieves with different sieve sizes viz:

850  $\mu\text{m}$ , 600  $\mu\text{m}$ , 425  $\mu\text{m}$ , 212  $\mu\text{m}$ , 106  $\mu\text{m}$ , 75  $\mu\text{m}$ , –75  $\mu\text{m}$  stacked on each other. The mass of the clay soil retained on each sieve was determined by using a weighing machine. The –75  $\mu\text{m}$  fraction of the sample was, thereafter, split into three parts to provide samples for XRF, XRD and SEM analyses. The three samples were labeled Sample A, Sample B and Sample C. Thereafter, Sample B was milled for 2 hours and Sample C milled for 4 hours as well.

## 2.2. Apparent Porosity

The three samples A, B and C were dried for 24 hours. They were, thereafter, dried further for another 24 hours at a constant temperature of 110°C and put into three well-rinsed and dried 250 ml beakers. The dried weights of the samples were determined. The beakers were then placed in a vacuum desiccator, after enough quantity of water that will dissolve the clay samples had been added. The soaked weights per the given time (thirty minutes) were determined and noted. The apparent porosity was calculated using the formula:

$$\text{Apparent Porosity} = \frac{W - D}{W - S} \times 100$$

where;

$W$  = Soaked weight;

$S$  = Suspended height;

$D$  = Dried weight.

## 2.3. X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) Analyses

The three samples A, B and C were compressed into pellets by using Hydraulic guage machine (**Figure 1**) and, thereafter, the major element composition determined by using Energy Dispersive X-ray Fluorescence Spectrometer (EDXRF). Analytical precision was ensured by the analysis of replicate samples. The mineralogical compositions of the three samples were also determined by using the X-ray Diffraction. The diffractions were collected from 10° to 90°  $2\theta$  and the diffractogram obtained were analyzed by using a software package.

## 2.4. Scanning Electron Microscopy (SEM) Analysis

Scanning Electron Microscopy (SEM) Analysis was also carried-out on powdered



Sample a, having 25Um each

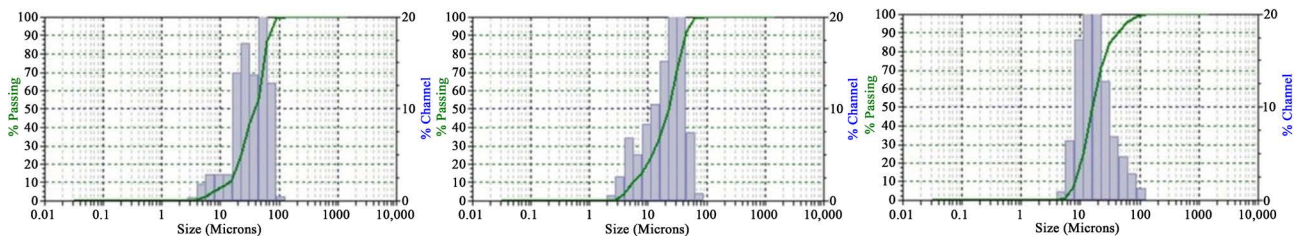
**Figure 1.** The three clay samples before compression into pellets.

samples A, B and C, after they had been gold-coated and prior to compression into pellets. The Carl-Zeiss Sigma Field Emission Scanning Electronic Microscope (Carl-Zeiss Sigma SEM) together with an Energy Dispersive Spectrometer (EDS) was used to determine the texture and the actual chemical composition respectively.

### 3. Results and Discussion

The particle size distribution of the three samples showed that using a defined tile to be 80%, the size for unmilled Sample A is 59.5  $\mu\text{m}$ . The sizes for samples B and C after milling for 2 hours and 4 hours are 35.44  $\mu\text{m}$  and 31.76  $\mu\text{m}$  respectively (**Figure 2**). This implies that the clay soil becomes finer as the time increases as a result of its mineral compositions. **Table 1** revealed that  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3$  are the major constituents, while the remaining components are in trace amounts in all the three clay samples.

The Microfabric structure of Sample A entailed grains of clean contacts, some connectors are within the grains of the clay at the magnification of 500 $\times$  (**Figure 3(a)**), and **Figure 3(b)** indicated that Titanium, Oxygen, Silicon have the highest Peaks, followed by aluminum and iron, while the other elements are minor elements. At the same magnification, Sample B consists of more dense clay matrices with a small visible interconnected assemblage of pore spaces within the sample (**Figure 4(a)**). The EDS spectra revealed that oxygen and silicon have the



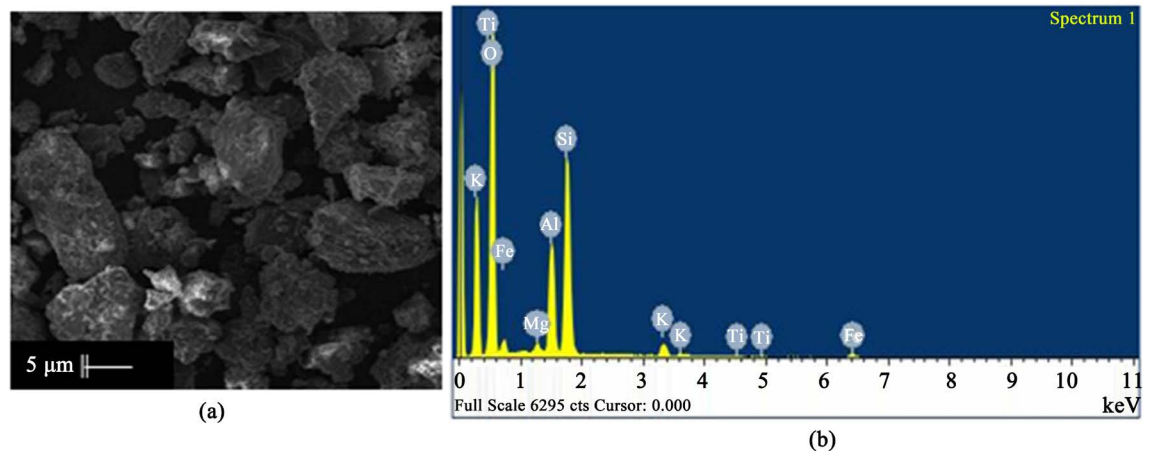
**Figure 2.** Particle size distribution for Sample A, B and C respectively.

**Table 1.** X-ray fluorescence (XRF) analysis for the three clay samples.

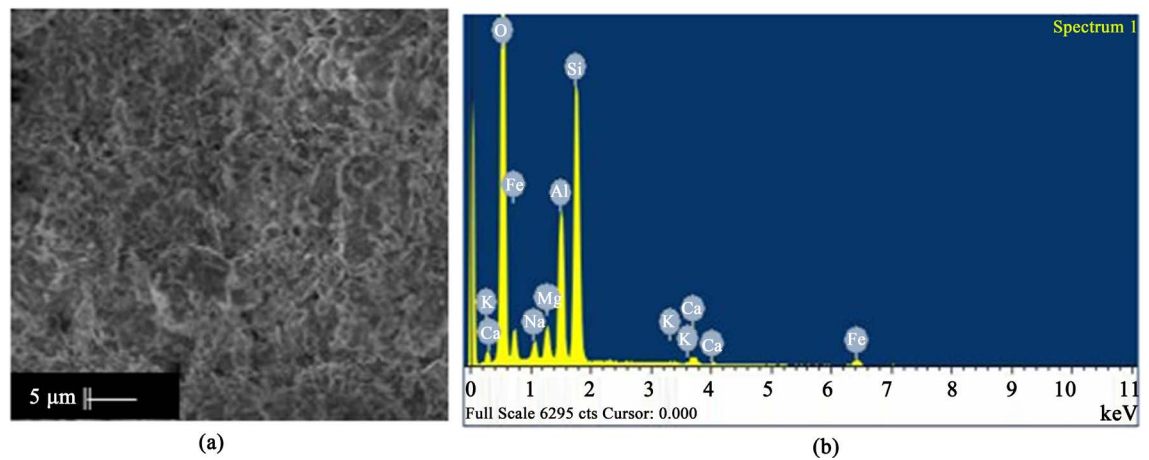
Chemical composition	Sample A	Sample B	Sample C
	Weight % (wt.%)	Weight % (wt.%)	Weight % (wt.%)
$\text{Na}_2\text{O}$	1.0857	1.1189	1.0218
$\text{MgO}$	3.7935	3.8349	7.8776
$\text{SiO}_2$	44.6240	43.6460	36.9253
$\text{Al}_2\text{O}_3$	18.6640	18.4357	15.6991
$\text{Fe}_2\text{O}_3$	23.6954	24.3772	16.6620
$\text{TiO}_2$	0.7863	0.8113	1.4407
$\text{CaO}$	5.5200	5.4987	12.2797
$\text{K}_2\text{O}$	0.6415	0.6073	0.3996
$\text{MnO}$	0.2897	0.8486	6.9253

highest peak (**Figure 4(b)**), while iron and aluminum have medium peak and the other elements were identified as trace elements.

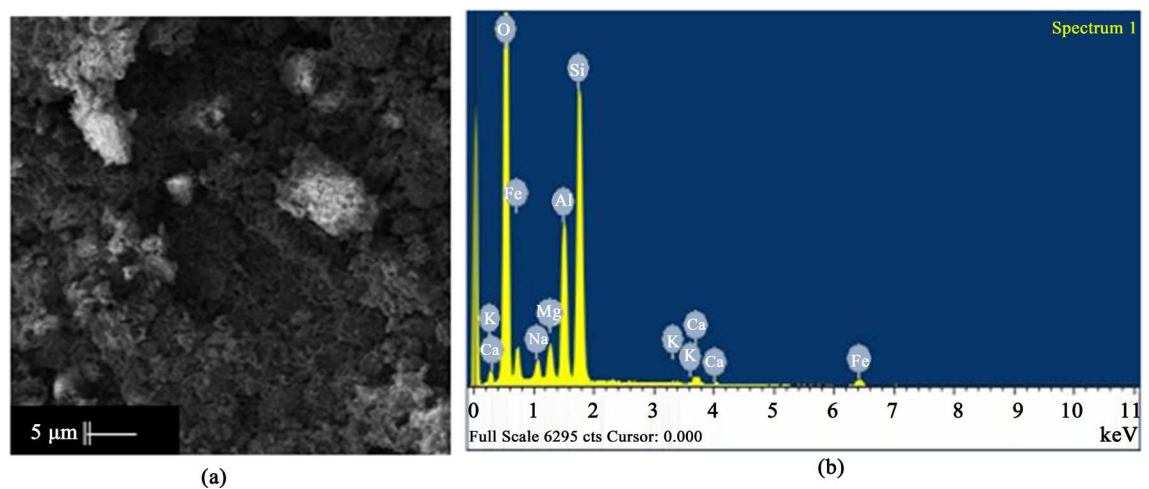
**Figure 5(b)** identified oxygen, silicon with the highest peak followed by



**Figure 3.** (a) Microfabric structure of Sample A and (b) Peak of Sample A.



**Figure 4.** (a) Microfabric structure of Sample B and (b) Peak of Sample B.



**Figure 5.** (a) Microfabric structure of Sample C and (b) Peak of Sample C.

aluminum, iron and carbon while the rest of the elements are minor elements. The result of the XRD revealed that five minerals were present in Sample A: Quartz, Montmorillonite, Bentonite, Kaolinite and Anorthite. Four Constituents were identified in Sample B: Bentonite, Montmorillonite, Kaolinite and Albite while Sample C contains Montmorillonite, Kaolinite, Albite and Bentonite as well (Figures 6-8, Table 2).

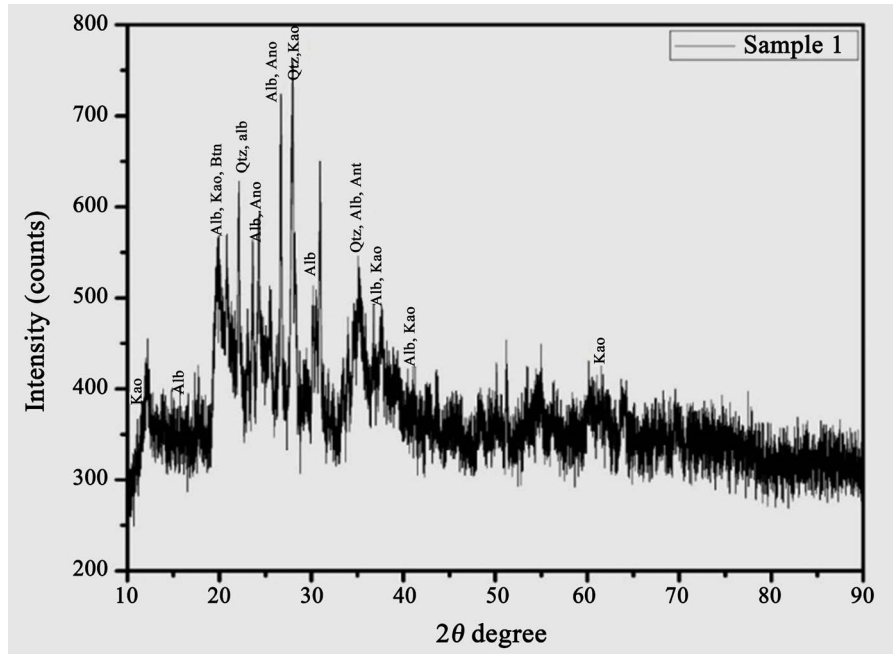


Figure 6. X-ray diffraction of Sample A.

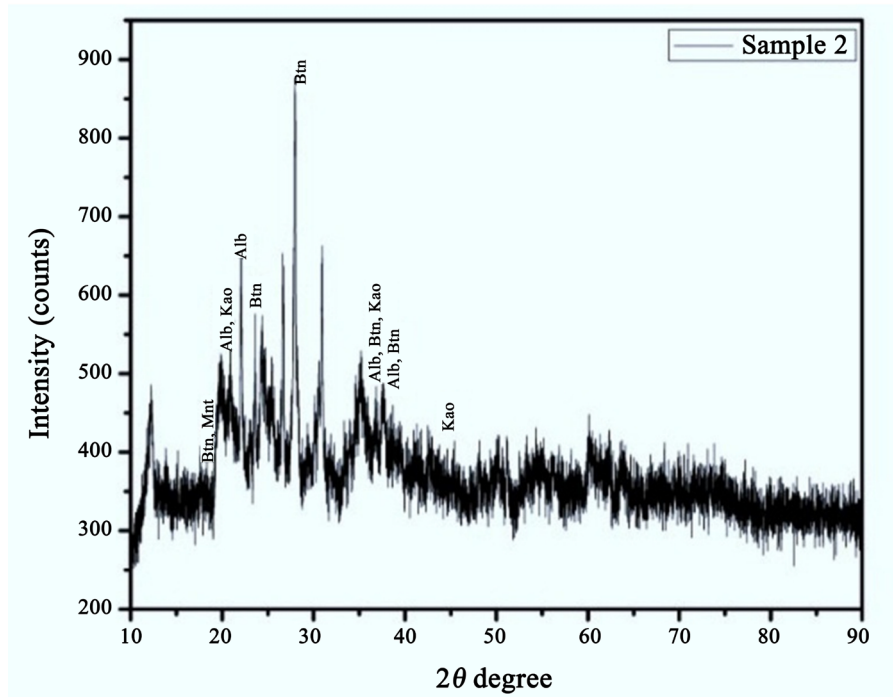
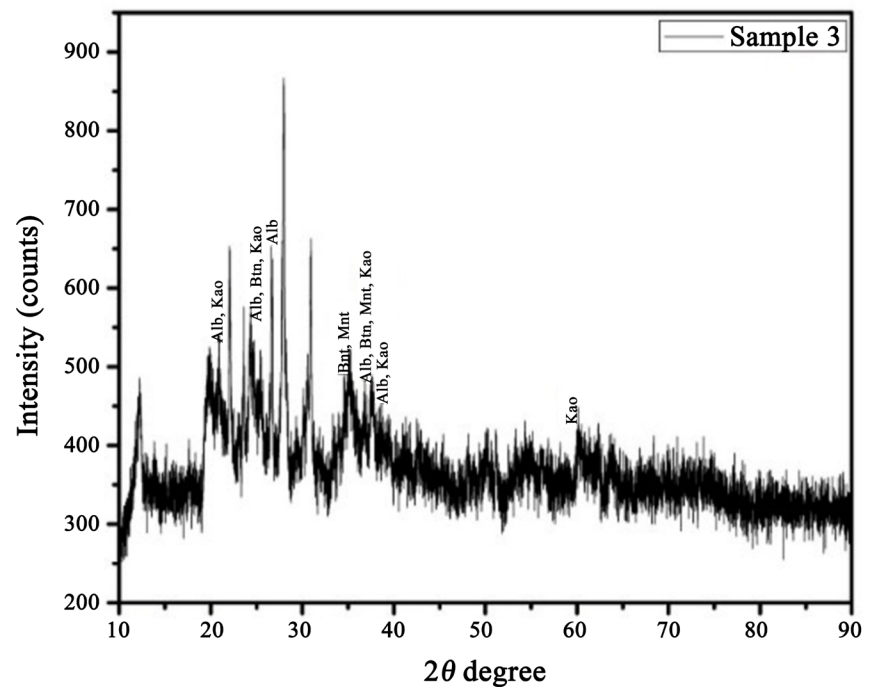


Figure 7. X-ray diffraction of Sample B.



**Figure 8.** X-ray diffraction of Sample C.

**Table 2.** XRD analysis of clay samples.

Samples	Kao	Btn	Mnt	Qtz	Ant	Alb
Sample A	+++	+++	+++	++	+++	–
Sample B	+++	+++	++	–	–	+++
Sample C	+++	+++	++	–	–	+++

Qtz (Quartz); Mnt (Montmorillonite); Btn (Bentonite); Kao (Kaolinite); Alb (Albite); Ant (Anorthite); (+++) major; (++) minor; (+) trace elements; (–) not detected.

## 4. Conclusions

It can be concluded from this research work that Sample A contains a small amount of quartz which cannot be applied in the making sand blasting and grit for sawing due to its high percentage of kaolinite. Kaolinite is useful as a filler for rubber and plastic because of its durability. The mineral phases in Sample B which include Kaolinite, Bentonite are important for large castings and the production of glossy papers used in the production of magazine.

Furthermore, Sample C is useful in the production of Iron ore as a result of the low occurrence of quartz and Anorthite and a major presence of Kaolinite and Bentonite.

However, it is imperative further researches be conducted in this area so as to consider the effect of reducing the impurity level of the clay samples to acceptable limits.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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