Impact of Oriented Clay Particles on X-Ray Spectroscopy Analysis

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Abstract. Understanding the engineering properties of the mineralogy and microfabic of clayey soils is very complex and thus very difficult for soil characterization. Micromechanics of soils recognize that the micro structure and mineralogy of clay have a significant influence on its engineering behaviour. To achieve a more reliable quantitative evaluation of clay mineralogy, a proper sample preparation technique for quantitative clay mineral analysis is necessary. This paper presents the quantitative evaluation of elemental analysis and chemical characterization of oriented and random oriented clay particles using X-ray spectroscopy. Three different types of clays namely marine clay, bentonite and kaolin clay were studied. The oriented samples were prepared by placing the dispersed clay in water and left to settle on porous ceramic tiles by applying a relatively weak suction through a vacuum pump. Images form a Scanning Electron Microscope (SEM) was also used to show the comparison between the orientation patterns of both the sample preparation techniques. From the quantitative analysis of the X-ray spectroscopy, oriented sampling method showed more accuracy in identifying mineral deposits, because it produced better peak intensity on the spectrum and more mineral content can be identified compared to randomly oriented samples.

Keywords: Clay particle, mineralogy, x-ray spectroscopy.

1. Introduction

Clay minerals are the most important chemical and dominating weathering product in the soil. They are formed by the alteration of existing minerals or by synthesis from elements when minerals weather to their elemental form [1]. Soils differ from other engineering materials because of its morphology characteristics which require a distributed particulate mechanics approach [2]. Specifically, clay mineral particles have commonly platy characteristics, the use of flat sample preparations for X-Ray Stereoscopy or X-Ray diffraction analysis are achieved from the naturally occurring random orientation of the clay particles. It is known that X-ray diffracted intensities from powdered samples depend on the orientation of the particles [3]. In theory, random orientation of powder form clay particles provides the maximum number of basal reflections but also gives a maximum overlapping reflections and this may cause reliable quantitative analysis inconveniently arduous [4]. Although analysis can be made with overlapping reflections, it is very convenient to use non-overlapping reflections. Non-overlapping reflections provides better peak intensities for X-Ray diffraction. However, there is still a dearth of published information on its effect on the elemental composition of clay particles.

This study aims to contribute by producing a well-crystallized and well oriented particle samples using appropriate techniques. The understanding of the quantitative evaluation of clay mineralogy is important for fundamental studies as it can be used to identify suitable methods for further civil engineering experiments. In this study, the oriented samples were prepared by placing dispersed clay in water and leave it to settle on a porous ceramic tile while applying a relatively weak vacuum suction to enhance the free fall to stable state of the particles. With that, the comparison on the elemental and mineral compounds with micro-imaging analysis between the random oriented and well oriented samples using X-ray stereoscopy and Scanning Electron Microscopy (SEM) was carried out. SEM image analysis can provide a better idea on how the clay particles are arranged between the two techniques. Ultimately, a thorough study on the impact of particle orientation on the elemental composition of the elements and its mineral compound can be achieved.

2. Materials and Method

In this study, three types of clay samples were used which are kaolin, bentonite and marine clay. The samples were first sieved through 63μm sieve (No. 200-mesh) to separate irrelevant non clay sized materials and turning it to a powdery form. This therefore maximized the amount of clay particles as it is defined as having sizes finer than 0.002mm [5]. The analysis was done on the Scanning Electron Microscope equipped with the Energy Dispersive X-Ray apparatus (SEM-EDX). This apparatus was used due to its capability of combining micro imaging and elemental compound distribution characterization analysis. Its characterization capabilities are due to its fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray emission spectrum [6].

Sample preparation was done carefully to achieve the required orientation of the particles. The random oriented samples were attained by applying the clay powder directly flat unto the specimen holder of the SEM-EDX apparatus. Well oriented samples were prepared by adopting the method as described by Shultz [7] and Kinter and Diamond [8]. Although the methods and apparatuses have considerably changed throughout the years, the concept of the test is still the same. In this study, the vacuum filtration technique (Figure 1) rather than centrifugal settling was used for separating approximately 1 gram of clay solid from a solvent and collecting the fraction on a porous disc. The clay fraction is seen to have a thin smooth layer on top of the porous disc. The thickness of the oriented clay was less than 1 mm. It was then dried at room temperature and left carefully undisturbed before it is taken for the SEM-EDX analysis.

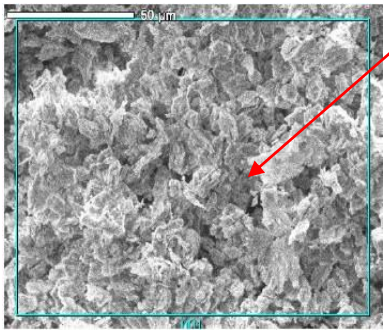
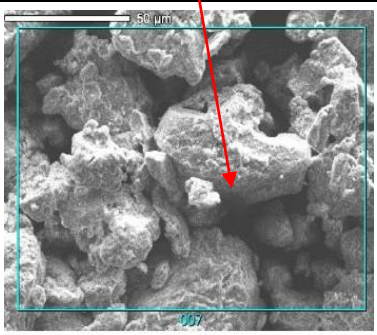
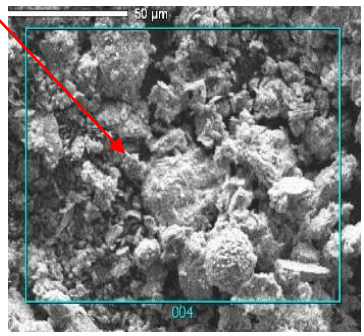
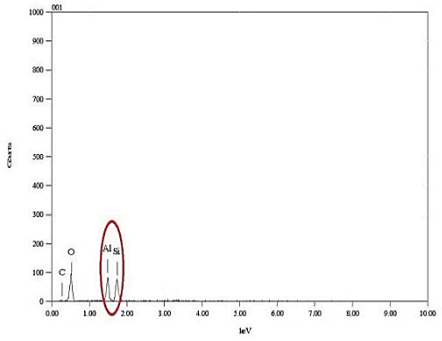
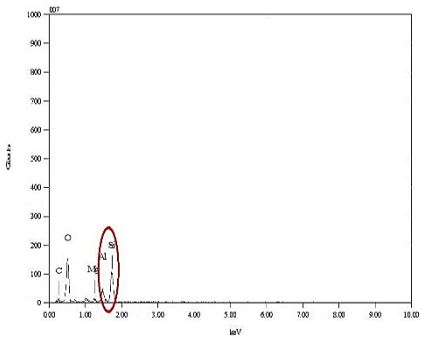
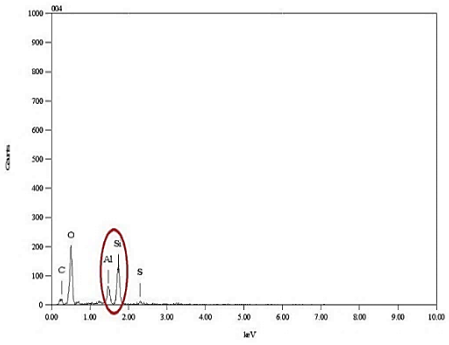
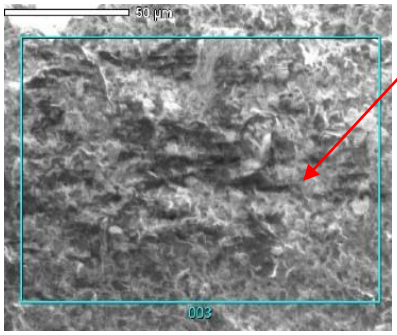
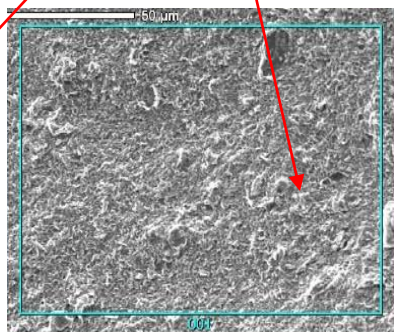
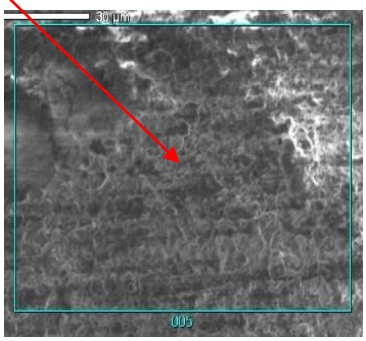
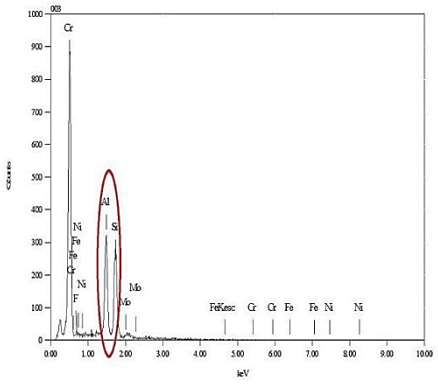
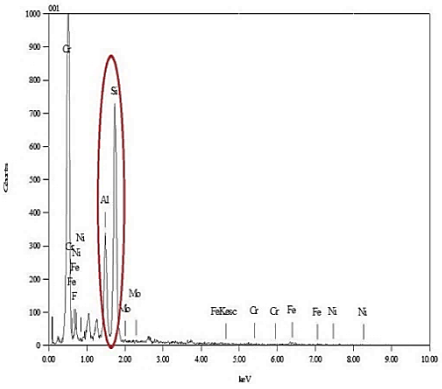
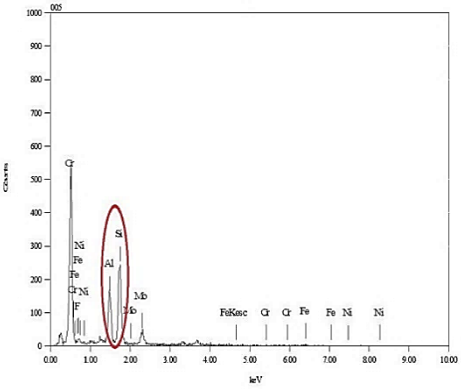
a)  b) 

Figure 1. (a) Vacuum with porous disc for filtration (b) Smooth layer on porous disc.

3. Results and Discussions

The EDX analysis will produce results that shows the percentage by mass of the chemical elements and the mineral compounds respectively. Table 1 shows the SEM images of all the non-oriented and oriented clay samples. The magnification of the images was up to x850, where the non-oriented clay samples seem to show that the particle assemblage is unstructured. The oriented samples showed that the clay particles were more compacted and well structured. With that, the high-energy beam from the spectroscopy is assumed to be focused consistently on the smooth surface of the oriented particle of clay sample.

Table 1. SEM image analysis and the peaks of the X-ray Spectrum of the Element of Koalin, Bentonite and Marine Clay respectively.

Kaolin	**Bentonite**	**Marine Clay**
Non-oriented clay particles	Non oriented particles are unstructured and uneven	
		
		
Oriented clay particles	Oriented particles are well structured and more compacted	
		
		

The table also confirms that the peaks of the X-ray emission spectra demonstrate convincingly that the oriented clay particles have an increased in the basal peak intensity of the Aluminum (Al) and Silicon (Si) elements as compared to the non-oriented clay particles. Figures 2 to 3 show the percentage by mass of the chemical elements and the mineral compounds found present within the Kaolin, bentonite and marine clay samples respectively.

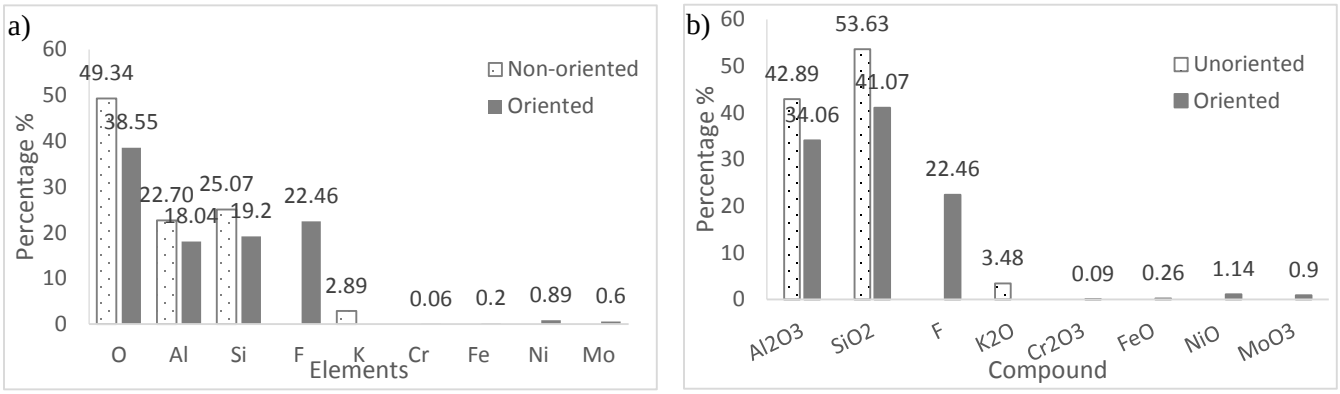


Figure 2. Percentage by mass of (a) chemical element and (b) mineral compounds of Kaolin.

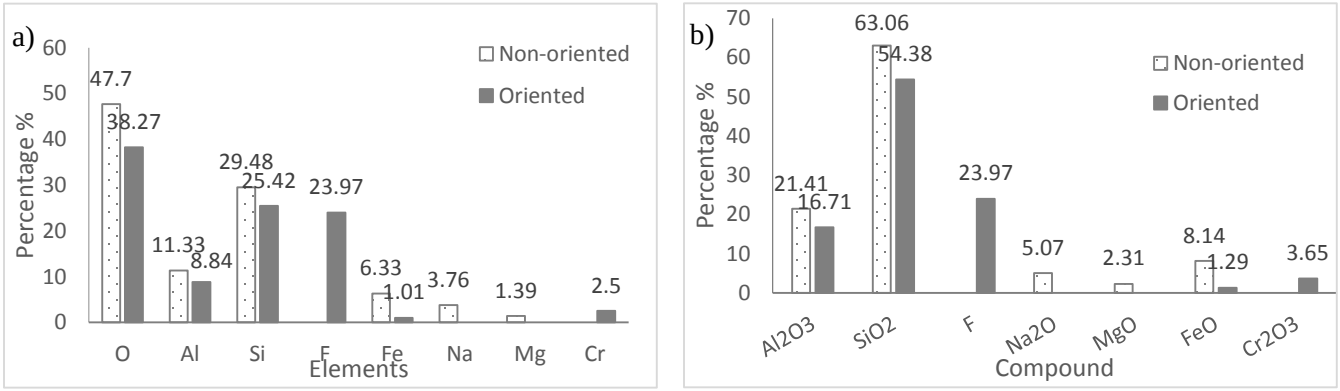


Figure 3. Percentage by mass of (a) chemical element and (b) mineral compounds of Bentonite.

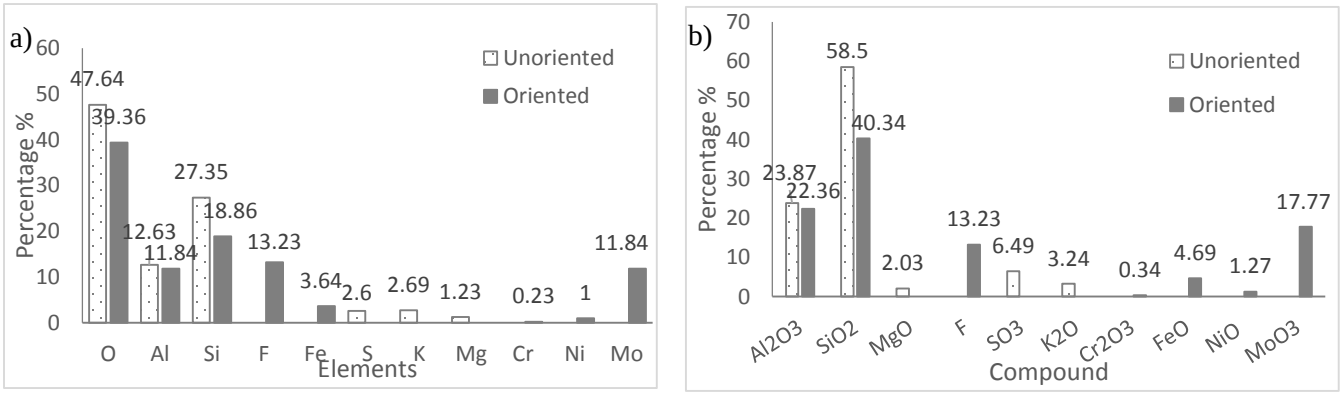


Figure 4. Percentage by mass of (a) chemical element and (b) mineral compounds of Marine Clay.

For all the samples, the most dominant elements by percentage of mass are Al, Si and Oxygen (O). These being the dominant elements in all clay minerals. However, oriented samples showed lower percentages of Al, Si and O to compensate for the small additional traces of other elements such as Chromium (Cr), Iron (Fe), Nickel (Ni) and Molybdenum (Mo). Furthermore, the amount of mineral compound by mass percentage can also be determined. The most dominant compound for all the samples was found to be Silicon Oxide (SiO2) and Aluminum Oxide (Al2O3). Again, additional mineral compounds were found for the oriented clays particles such as Chromium Oxide (Cr2O3), Iron Oxide (FeO), Nickel Oxide (NiO), and Molybdenum Oxide (MoO3). The analysis also showed that the dominant mineral compounds of SiO2 and Al2O3 for all the samples are almost similar where seemed to comprise of more than 15% of its mass. This is in accordance with the typical mineralogical content of clayey soils.

4. Conclusion

From this study, it was found that the vacuum filtration technique to create a solid form of clay solvent to settle on a porous disc with suction allows the clay particles to be in an oriented pattern. Free falling of sediments occur in the natural gravitational compaction of clay. This is often hindered by the presence of coarse grained particle and by a chemical environment that causes the electricity charged fine particles to flocculate rather than be dispersed and form a pack of cards type structure. Image analysis from SEM showed that the oriented particles have a sample surface of a more structured and compacted appearances, compared to the non-oriented clay particles which is seen to be more scattered and less dense. From the EDX analysis, it was found that both the oriented and non-oriented clay particles identified the dominant chemical element and mineral compound. However, the oriented clay particles showed additional elements and minerals in the samples. Therefore, in order to achieve a more accurate quantitative evaluation of clay mineralogy, a proper technique of obtaining oriented soil particles is essential in sample preparation for quantitative mineral analysis.

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