

X-ray diffraction

The X-ray diffraction (XRD) analysis was undertaken to investigate *qualitatively* the composition of the clay mineralogy in the soil samples. The main purpose of this study was to identify the clay mineralogy of the soil samples. It is important to identify clay minerals, for example kaolinite, illite, montmorillonite, chlorite etc. as they may influence the behaviours and properties of the clay materials. The cation exchange capacity, specific surface area, plasticity, and retention capability very much dependent on clay mineralogy.

a. Sample preparation

The glass slide method was utilised in this study because of its ease of application (Figure 3.18). All of the sample preparation must be accomplished with a minimum of chemical and physical damage to the clay minerals present. The rule is to do as little as possible to the sample before presenting it to the x-ray beam. Organic-free soils less than $63\mu\text{m}$ (after the organic test from Section 3.3.1) were used. According to Moore and Reynolds (1989), organic matter can produce broad x-ray diffraction peaks, and may increase the background, and inhibit dispersal of other minerals.

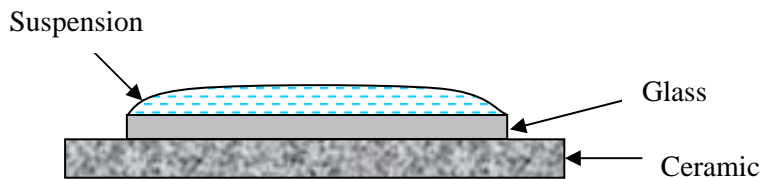


Figure 3.18. The suspension on the glass slide is hold by the surface tension. The ceramic/tile is used to catch the spills.

About 1g of soil was then placed into 10ml measuring cylinder. About 5ml of distilled water was then added in to it and was mixed thoroughly using a glass rod. After 15-20 minutes, the upper solution was then pipetted (using an eyedropper) and placed slowly on the clean glass slide so that the liquid covered the entire surface of the slide. A porous ceramic plate was used under the slide to catch any spills. It was assumed, according to Stoke's Law, that the solution that was sampled contained the clay particles of less than 2

μm . The slide was dried at the room temperature. High drying temperature should not be used because poorly crystallised clay minerals in soils can be damaged by high temperature. Several treatments have been undertaken to verify the clay minerals in the soil. These treatments were: -

Glycol treatment

The sample was exposed to the vapour of the reagent ethylene glycol solvation. It was dried in the oven at 60°C and then re-analysed to determine the presence of montmorillonite. Montmorillonite is easily identified by comparing patterns of air-dried and ethylene glycol-solvated preparations. The glycol-treated preparations give a very strong 001 reflection at about $5.2^{\circ}2\theta$ (16.9 \AA), which, in the air-dried condition, shifts to about 6° (15 \AA) (Moore and Reynolds, 1989).

Heat treatment

The slide was heated in the furnace at a temperature of 550°C for 30-40 minutes to verify the chlorite and kaolinite peaks. Heating chlorite to 550°C for 1 hour causes dehydroxylation of the hydroxide sheet with attendant changes in the diffraction pattern. The intensity of the 001 reflection increases greatly and shifts to about 6.3 to $6.4^{\circ}2\theta$, and the 002, 003 and 004 reflections are much weakened. At this temperature, kaolinite becomes amorphous to X-rays and its diffraction pattern disappears. This test may suggest that chlorite is present or that chlorite is absent (Moore and Reynolds, 1989).

Hydrochloric acid treatment

0.2g of organic-free soil sample was placed into the centrifuge tube. About 5ml of concentrated hydrochloric (HCl) was added and heated for 10-15 minutes. It was washed with distilled water for 3-4 times to neutralise the acid. The sample was centrifuged, the wash water was discarded and the soil left was oven dried and powdered. The sample was then used for preparation of another glass slide. This treatment was used to destroy the chlorite peaks and to distinguish chlorite from kaolinite.

b. Qualitative analysis

The clay minerals were identified using the X-ray diffraction patterns (diffractogram) which by the peak's position, intensity, shape and breadth. Peak position is determined using Bragg's law which is written as $n\lambda = 2d\sin \theta$. Most of the important clay peaks are at the 2θ values of 40° or less; therefore, θ is 20° or less. The identification of clay minerals was accomplished by careful consideration of peak positions and intensities. The qualitative identification procedure began by searching for a mineral that explains the strongest peak or peaks, then confirming the choice by finding the positions of weaker peaks for the same mineral. Once a set of peaks was confirmed as belonging to a mineral, these peaks were eliminated from consideration. From the remaining peaks, a mineral that will explain the strongest remaining peak or peaks was searched and then confirmed by looking for its peaks of lesser intensity. This method was repeated until all peaks were identified.

The determinations of clay minerals were based on the treatment results described earlier. Illite and glauconite profiles are unaffected by ethylene glycol solvation and heating to 550°C . Glauconite has a higher 001/003 intensity ratio than illite, but the main difference is the very weak or non-existent glauconite 002 reflection whose weakness is caused by heavy scattering from octahedral iron (Moore and Reynolds, 1989).

Chlorite has a basal series of diffraction peaks based on a first-order reflection of 14.2 \AA . Kaolinite has reflections based on a 7.1 \AA structure. Most kaolinites have the 002 peak at $24.9^\circ 2\theta$, and chlorite has its 004 reflection at $25.1^\circ 2\theta$. Positive identification of chlorite is provided by peaks at 6.2 and $18.8^\circ 2\theta$, but these peaks are often weak and may not be detectable if the chlorite concentration is low. The chlorite and kaolinite can be differentiate by treated chemically or heated and then re-examined. Montmorillonite is easily identified by comparing diffraction patterns of air-dried and ethylene glycol-solvated preparations (Moore and Reynolds, 1989).

Table 3.10. An identification of clay minerals based on observation after treatments.

Mineral name	Air dried	Heated 550°C	Glycolation	Acid
Montmorillonite	15.0 Å	10.0 Å	16.9 Å	Unchanged
Illite	10.0 Å	Unchanged	Unchanged	Unchanged
Kaolinite	7.1 Å	Peak disappeared	Unchanged	Unchanged
Chlorite	14.2 Å	Unchanged	Unchanged	Peak disappeared