4. SOIL MINERALOGY

4.1 INTRODUCTION

Any definition of the term soil will take into account aspects of behaviour in which the user has a predominant interest. Four ways in which soil could be defined are:

- 1. The material on natural surfaces in which vegetation takes root.
- 2. A limited section (beneath natural surface) extending from decomposed rock to the surface.
- 3. Any loose fragmental material which can be readily broken into grains or masses of coherent clays.
- 4. The loose fragmental materials of the earth's crust.

Soil Mechanics could be loosely defined as the application of the principles of mechanics and hydraulics to the study of soil. In this context "study" would include examination of behavioural changes that occur when soil is subjected to changes of stress, deformation, or environmental conditions.

The examination of soil behaviour can be performed at two levels: a consideration that response can be explained by considering soil to be an assemblage of solid soil particles, air and water; or a study at the atomic level where surface forces are considered.

It could be thought that a study at the atomic level would provide a more fundamental understanding of soil behaviour than that conducted at the "assemblage" level. Whilst this is true for some limited aspects of soil behaviour, in particular the response of "fine grained" soils to changes in moisture, the major thrust in research has been the explanation of soil behaviour on the engineering (or assemblage) level.

This course will concentrate on studies of soil behaviour at the engineering level. To explain adequately an observed response to some change in condition reference will be made to atomic structure.

A first course in soil mechanics can be confusing. Problems posed in elementary statics and fluid mechanics are usually quite clear-cut and well defined, each question having

its own unambiguous answer. The solution to problems involving soil response is not so straightforward, because:

- (a) boundary conditions may be ill-defined, and
- (b) material properties are variable, and in some cases unknown.

Problem solving in soil mechanics usually entails;

- (a) a recognition of the physical problem.
- (b) an idealisation of this real situation into a model of behaviour.
- (c) the solution of the idealised model using relationships of solid mechanics or fluid mechanics.
- (d) a consideration of the applicability of the answers to this simplified problem with respect to the physical situation.

(A solution process common to all engineering problems).

4.2 SOIL FORMATION

4.2.1 Weathering

Implicit within the definition of soil is the notion that soil is the product of decomposition from rock sources. (An exception are the organic soil materials). A detailed study of soil formation will not be attempted in this introductory presentation.

Four stages can be identified in the formation of soil.

- 1. Weathering and Transportation.
- 2. Deposition.
- 3. Sediment alteration.
- 4. Movements of the earth's crust.

Not all are essential. Weathering may occur without transportation and subsequent deposition, in which case a "residual soil" is formed.

Weathering can be classified, simply, as physical or chemical. The more common agents associated with physical weathering are water, temperature and stress relief (uplift). Chemical weathering can occur at normal (surface) temperatures or can be associated with temperatures in excess of 100°C and pressures greater than one atmosphere. (For a discussion on transformation that occurs at elevated temperatures and pressures see, for example, Grim (1968), Ch. 13).

The products of weathering can be transported via wind, water, ice or mass movements (landslides). In all cases further degradation of the parent material or alteration of the sediment can occur.

When the transportation velocity is slow enough, particles in suspension will settle. As the sediment thickness builds up, further changes in the soil can occur. These changes could be chemical or physical. Taken to extremes of pressure or temperature a new rock form may emerge.

Any movement of the earth's crust, abrupt or gradual, uplift or subsidence, will change the environmental conditions under which the soil exists. The soil formation cycle then recommences.

Since all soils result from weathering of minerals (organic soils are excluded from this discussion) present in the parent rock, it can be argued that the presence of primary rock-forming minerals in soil is indicative of their stability or the time for which weathering has been proceeding.

4.2.2 Weathering of Igneous Rocks

For igneous rocks the most prevalent minerals are feldspars (60%), pyroxenes and amphiboles (17%), quartz (12%) and mica (4%). When this rock type weathers the feldspars disappear. This very low stability is brought about because of the low bond strength between feldspar units, and in an alkaline environment the feldspar commonly alters first to the clay mineral montmorillonite. If the environment is conducive to change, this clay mineral type will alter further. The stability of the other rock forming minerals is in the order

muscovite > > > amphibole > pyroxenes > > > biotite.

Thus it could be expected that muscovite may be present in a soil from igneous rocks, whereas the amphiboles, pyroxenes and biotites would be altered completely.

A general weathering schedule for igneous rocks has been described by Currey (1968). The agents or features which contribute to weathering of a surface are:

- (i) water charged with carbon dioxide,
- (ii) oxygen,
- (iii) acids derived from vegetation, and
- (iv) a system of joints through which water will penetrate.

The rock on either side of the joint is softened and removed by erosion. In some cases "tors" are left standing. See Fig. 1.1(a).

Grim (1968) lists five factors that have an influence on the weathering of rock and the subsequent formation of clay minerals. These are

- (i) Parent Rock composition and texture
- (ii) Climate principally the variation in temperature and rainfall
- (iii) Topography
- (iv) Vegetation in particular the products of decomposition
- (v) Time.

Table 2.1 is a summary of generalised patterns of weathering for igneous rocks.



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FIGURE 4.1(A) - GRANIT TOR FORMATION (AFTER CURREY, 1968)
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FIGURE 4.1(B) - PROGRESSION OF WEATHERING, SEDIMENTARY ROCKS (AFTER CURREY, 1968)

Rock Type		Climate and Topography	Vegitation	Time	Product
	a	Low rainfall or poor drainage (Magnesium remains)			Smectite Mineral
(u	b	High rainfall or good drainage (Magnesium removed rapidly)			Kaolinite Mineral
g Magnesiun	c	Intermediate between (a) and (b)	Production of organic acids	Long duration	Smectite then Kaolinite
Igneous(containing	d	Cold and wet Magnesium removed	No acid produced	Long duration	Clay minerals break down aluminium and iron leached out. Silica remains near surface.
Basic	e	Wet and hot Magnesium removed			Silica removed, Iron and Aluminium concentrated near surface(Lateritic Alteration)
derable otassium	a	Conducive to allow Magnesium and Potassium to remain			Illite and Smectite
Acid Igneous (Containing consi Magnesium and I	b	Rapid removal of Magnesium			Kaolinite Mineral
Acid Igneous (Low Magnesium)		Allow Potassium to remain			Illite
Acid Igneous (Low in	Potassium	Magnesium remains			Smectite Mineral

Table 4.1 Generalised Patterns of Weathering-Igneous Rocks (after Grim, 1968) (Note: For definition of products of weathering see Section 4.3.2)

4.2.3 Weathering of Sedimentary Rocks

Weathering of the sedimentary (Silurian Age) rocks which form the base for a large area around Melbourne is extremely variable. Very soft or hard beds can be formed depending on whether the parent material was mudstone, siltstone or sandstone.

Neilson (1977) identifies three phases in the weathering of the mudstones (and siltstones) of the Melbourne area.

- (a) Early to Mid-Tertiary
- (b) Lateritic weathering of pre-existing deeply weathered profiles
- (c) Later weathering the development of soil and soil minerals.

Cole, Lancucki and Nickson (1968) conducted mineralogical determinations of the unweathered and weathered material. The weathering sequence was described as:

Unweathered	Weathered	Lateritic Weathering
Quartz	unaffected Quartz	Quartz
Kaolinite	disorganised Kaolinite	reduced to Halloysite 2H ₂ 0
Muscovite	slightly altered Muscovite	severely degraded Inter-stratified
		mineral

4.2.4 Non-clay Components of Soil

The preceding discussion has centred around the formation of constituents of one component of soil - the clay fraction. As was be seen in Chapter 1, (Geomechanics 1) soil can be considered to be composed of a number of components, which can be classified according to particle size:

clay < 0.002 < silt < 0.06mm < sand < 2.00mm gravel.

To explain adequately the behaviour of clay soils, principles of colloid chemistry need to be considered. For the silt sizes and larger, gravity forces play a predominant role in soil behaviour: features such as particle shape, size, strength, and angularity need to be considered. These factors alone may not be adequate in understanding behaviour - a

knowledge of the composition and history, similar to that required for the clay fraction, may well be essential for the non-clay components of a soil.

Degradation of a parent rock will result in a wide variety of particle sizes. These particles may remain in place, or be transported and subsequently deposited. Where an environment is not conducive to further mineralogical modification, or the mineral from the parent is extremely stable, minerals present in the parent rock will be found in the soil. Examples include:

- 1. Silts: Loess A wind-blown silt essentially composed of feldspar and quartz, with some clay
- 2. Sands: Wind-blown, fluviatile, or river deposits. Composition may include silica, quartz, calcite, olivine or rock fragments.

Other soils of silt size and larger can result from chemical precipitation, eg. calcium carbonate, or from remains of marine organisms (coral - calcium carbonate: diatomaceous earth - silica). Of particular note in this category are the cemented carbonate sands and silts found in Bass Strait and the North West Shelf. The very low cementing forces between particles, coupled with low crushing strength of the particles themselves, pose problems in foundation design for offshore structures.

4.3 CLAY MINERALOGY

4.3.1 Silicate Structures

Where a cation is small, the co-ordination number of the ion is of the order 3-4, which indicates a tetrahedral structure. If the cation is highly charged, a high repulsion will exist between adjacent tetrahedra which means that joining of the tetrahedra will occur at corners or edges. Thus we can have silicate structures following a number of forms (see Fig. 4.2). The silicate structure of most importance in the study of clay mineralogy is the layer - minerals which have this structure are referred to phyllosilicates.



FIGURE 4.2 - DIAGRAMMATIC REPRESENTATION OF TYPICAL SILICATE STRUCTURES

4-9

Reference to the definition of a clay mineral^{*} will show that the structure of the aluminium or magnesium component needs clarification. These components exist as aluminium or magnesium hydroxides. The successive layering of the silicate sheets and the aluminium or magnesium hydroxides produces the structure for most clay minerals.

For the silicate sheets the tetrahedra share three corners in the same plane, with the fourth oxygen of each tetrahedron always pointing in the same direction. This arrangement produces open, hexagonal rings. (see Fig. 4.2 (f)).

An aluminium hydroxide (Gibbsite) has the aluminium cation co-ordinated by six hydroxyls to form an octahedron. Since the edges of the adjacent octahedra are shared, the resulting structure is a sheet composed of hexagonal units. The same structure exists for magnesium hydroxide (Brucite). Each cation has three anions above and three below, with the lower anions rotated through 60° to form an hexagonal close packed arrangement of the two hydroxyl sheets. This is illustrated diagrammatically in Fig. 4.3 (c), (d), (f) and (g).

Where one octahedral and one tetrahedral layer combine a 1:1 type of layer structure is formed. If a second tetrahedral layer is added a 2:1 layer structure is formed. The type of layer structure forms the basic classification system for clay minerals: each type is subdivided on the basis of the charge carried by the layer. Table 4.2 shows a classification proposed for clay minerals.

^{*} clay mineral: A hydrated aluminium (or magnesium) silicate, with variable amounts of calcium, magnesium, iron, sodium, potassium, usually occurring as very small crystalline particles with layered structure - A.S. 1726 - 1993 SAA Site Investigation Code.







Ion	Radius (Å)	Symbol
Al ⁺³	0.57	•
Si +4	0.39	●
0 -2	1.32	\bigcirc
он-1	1. 32	
Mg +2	0.78	●

	ь-мg	+1Z	
000000 00	6 – OH	- 6	
(g)			

FIGURE 4.3 - BASIC SILICATE UNITS. (A) & (B) SILICON TETRA-HEDRON. (C) ALUMINIUM OCTAHEDRON. (D) MAGNESIUM OCTAHEDRON. (E) SILICA. (F) GIBBSITE. (G) BRUCITE (AFTER LAMBE AND WHITMAN, 1969)

6-0H-6

TABLE 4.2 PROPOSED CLASSIFICATION (PHYLLOSILICATES/CLAY MINERALS)SHOWING LOCATION OF SELECTED CLAY MINERALS

Туре	Group (x = charge/formula unit)	Sub-Group	Species
1:1	Kaolinite-Serpentinte (x ≈ 0)	Kaolinites Serpentines	Kaolinite,
			Halloysite
2:1	Pyrophyllite - Talc (x ~ 0)	Pyrophyllites Talcs	Pyrophyllite Talc
		Smectites or	Montmorillonite
	Smectite or Montmorillonites	Montmorillonites	
	$(x \approx 0.25 - 0.6)$		
2:1	Vermiculite	Dioctahedral/	Dioctahedral
	$(x \approx 0.6 - 0.9)$	Trioctahedral	Vermiculite
		Vermiculites	Trioctahedral
	Mica		Vermiculite
	(x ≃ 1)	Dioctahedral/	Muscovite
		Trioctahedral Micas	Biotite
2:1:1	Chlorite	Dioctahedral/	Donbassite
	(x variable)	Trioctahedral	Cookeite
		Chlorites	

4.3.2 Clay Mineral Structures

(a) The 1:1 Mineral - Kaolinite

Two-thirds of the hydroxyls in one plane of the octahedral layer are replaced by apical oxygens of the tetrahedral layer. The remaining OH ions are in the centres of hexagons formed by oxygen ions at the apex of the silica tetrahedra. Because of a slight difference in the cation spacing between the tetrahedral and octahedral layers, some distortion of the ideal hexagonal tetrahedral network occurs. The vertical Si-0 bond is tilted slightly to fit the overlying octahedral layers, resulting in a triclinic rather than monoclinic system.*

Bonding between the basal oxygens of one Kaolin unit and the hydroxyl associated with the 'top' of the next is both Van der Waal's and hydrogen. Thus a "tightly bound" stack of 1:1 units can be constructed. This is shown diagrammatically in Fig. 4.4 (b).

Where the octahedral layer is Brucite a mineral of the Serpentine sub-group is formed, whilst a mineral of the Kaolinite sub-group is formed if Gibbsite is the octahedral layer.

If a monomolecular layer of water is present between the unit Kaolinite layers, a clay mineral, Halloysite is formed.

As discussed previously, the b spacing for the 0 plane in the silica tetrahedra is greater than the b spacing for the OH plane of the Gibbsite ($8.93A^\circ$ and $8.62A^\circ$ respectively). In Kaolin the OH spacings are stretched to match the silica sheet.

The interlayer water present in Halloysite reduces the bond between successive units, and thus the stack tends to form tubes, as shown in Fig. 4.4 (c). For these tubes the c spacing is $10.1A^{\circ}$. Where the interlayer water is removed by drying the tubes tend to unroll: this dehydration is irreversible, and can take place at temperatures as low as $60^{\circ}C$.

(b) 2:1 Minerals

* CRYSTAL STRUCTURE

SYSTEM	AXES	AXIAL ANGLES
Triclinic	$a \neq b \neq c$	$\alpha \neq \beta \neq \gamma \neq 90^{\circ}$
Monoclinic	$a \neq b \neq c$	$\alpha \neq \beta = 90^{\circ} \gamma = 90^{\circ}$



FIGURE 4.4 - STRUCTURAL AND SCHEMATIC ARRANGEMENTS KAOLINITE, HALLOYSITE AND MONTMORILLONITE

Included in this sub-group are the smectite minerals (montmorillonite) and illite (hydrous mica). The structural arrangement is indicated in Fig. 4.4 (e).

The 2:1 minerals differ from the 1:1 through the addition of a second silica sheet which sandwiches the octahedral layer. As in the case of the 1:1 minerals the hydroxyls of the octahedral layer are replaced with apical oxygens from the silica sheets (see Fig. 4.4 (f).

Bonding between successive 2:1 units can vary from very weak Van der Waal's Bonds in the case of montmorillonite to the very strong bond arising from the interlayer potassium ions in the case of the illites.

(c) 2:1:1 Minerals

If a brucite layer is located between adjacent 2:1 units the structure typical of chlorites is obtained.

4.3.3 Source of Charge on a Clay Mineral

The formula/unit cell for a kaolinite is

 $(OH)_8$ Si₄ Al₄ 0₁₀

whilst for a pyrophyllite (the prototype structure for montmorillonite) the formula/unit cell is

(OH)4 Si8 Al4 020

Both structures are electrically neutral. However both mineral types carry a charge: the manner in which this charge deficiency is balanced, coupled with the bonding between successive layers, is critically important to the explanation of behaviour.

A major source of charge imbalance is attributed to isomorphous substitution, ie. a cation in the lattice is replaced by an ion of similar size, but (usually) carrying a lower charge. For the four most common clay minerals this replacement has been suggested as:

- 1:1 Kaolinite Mg^{2+} for Al^{3+}
- 2:1 Montmorillonite 1 Mg²⁺ for every 6th Al³⁺
 Illite some Si⁴⁺ replaced by Al³⁺

2:1:1 Chlorites Al³⁺ for Si⁴⁺ Al³⁺ for Mg²⁺ in the interlayer

The small charge carried by kaolinite has also been attributed to broken bonds around particle edges which lead to unsatisifed positions of charge. Some work on the position of exchange sites in kaolinite questioned this model, and suggested that (high) charge deficiencies may arise from montmorillonite impurities present in the kaolinite, in addition to isomorphous substitution. (McBride, 1976).

The charge imbalance associated with clay minerals is measured quantitatively by the cation exchange capacity of the mineral.

To maintain charge equilibrium cations are attracted to the surface of a clay mineral and are held in an exchangeable form on, or near, the surface by electrostatic forces. The source of the exchangeable cations is from solution surrounding the clay particles, and as the term "exchangeable" implies the ions associated with the colloidal clay particle are in dynamic equilibrium with the ions of the surrounding solution.

Where the interlayer bond is strong, as for kaolins, exchangeable cations will be adsorbed onto external surfaces. For 2:1 minerals, where successive units are weakly bonded, the cation is adsorbed into the interlayer position. If the ion adsorbed into this interlayer position is potassium a very strong bond between the potassium ion and adjacent 2:1 units is formed - as for the mineral Illite. Chlorites reach charge neutrality through the positive charge deficiency in the 2:1 layers being balanced by a positive charge excess in the interlayer Brucite.

Of the three mineral structures discussed above, it is the weakly bonded 2:1 minerals that produce most problems when subjected to change in environment.

These problems are associated with

- (a) an expanding lattice, and
- (b) particle size.

4.3.4 The Influence of Water on Clay Mineral Behaviour

In a dry clay, adsorbed cations are bound tightly to the soil surface. The cations in excess, and their associated anions are present as a salt precipitate. When this dry clay is placed in water, the salts go into solution and the adsorbed cations are hydrated. The negatively charge mineral surface and the distributed charge in the adjacent phase are together known as the diffuse (or electrical) double layer. Factors which affect the nature and extent of the double layer include:

- (i) composition and concentration of the electrolyte solution,
- (ii) density of charge on the surface of the particle,
- (iii) valence of the cations,
- (iv) dielectric constant of the medium, and
- (v) temperature.

Mathematical theories concerning the double layer which include these factors are well summarised in Lee (1968), and Mitchell (1977).

Previously it has been mentioned that clay particles can be of colloid size. In solution, the particles will move in a random manner. If the attraction forces between particles exceed forces of repulsion, the particles will increase in size - up to a stage where gravitational forces will predominate and the particles will sediment out, i.e. the system flocculates.

The forces controlling attraction and repulsion can be classified as:

- (a) Independent of a system: eg. electrostatic or electromagnetic (Van der Waals)
- or
- (b) System Dependent: eg. electrostatic (electron or ion clouds) or ion hydration.

Electrostatic and electromagnetic forces both contribute to attraction, whilst electron or ion clouds and ion hydration are forces of repulsion, with ion hydration being the major repulsive force. Thus the behaviour of clay minerals in suspension can be controlled by changing the environment. For the Cardinia Creek reservoir turbidity of the water was reduced by introducing gypsum - the thickness of the double layer of the clay particles in suspension was reduced, leading to forces of attraction exceeding repulsion forces. For this case the enlarged particles settled out. (Grant et al, 1976).

Any factor which changes the "thickness" of the double layer will alter the characteristics displayed by a clay soil. Two of the more important system variables are:

- (a) Cation Valence: an increase in valence suppresses the concentration of the solution between adjacent particles, and the potential between particles, leading to a decrease in interparticle repulsion.
- (b) Ion Size: the larger the ion size the thicker the layer required to accommodate the necessary number of cations, hence the greater the repulsion.

Associated with both of these factors is the phenomenon that multi-valent ions are adsorbed preferentially onto the mineral surface. For a given number of exchange sites, the higher the valence the lower the number of ions required to satisfy charge neutrality and hence the double layer is affected accordingly. This feature can be detected through very simple soil tests

eg. Na Kaolinite
$$w_{L} = 53\%, w_{p} = 32\%$$

Ca Kaolinite $w_{L} = 38\%, w_{p} = 27\%$

Since the difference between w_L and w_p is indicative of the compressibility of the soil we can argue that a Na Kaolinite would be more compressible than a Ca Kaolinite.

When the double layer and mineral particle size are considered together it can be argued that the smaller the particle (and hence the higher the specific surface) the larger the amount of water that (with respect to the mass of dry soil) will be associated with the double layer. This feature is of great importance when the swell of a soil, when wetted from a dry condition, is considered. Of particular significance is the clay mineral montmorillonite: the bonding between successive layers is weak and when an ion associated with this interlayer position hydrates, the clay mineral stack will expand. Expansion associated with moisture can cause structural failure in some lightly loaded structures.

SOIL TO BE TESTED

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TABLE 4.3 - FLOW CHART: TESTS INVOLVED IN IDENTIFICATION OF SOILTYPES AND SOIL MINERALS (AFTER MITCHELL, 1977)

4.4 IDENTIFICATION OF CLAY MINERALS

4.4.1 X-Ray Diffraction

The principle involved with estimating the spacing of crystal planes will not be covered in these notes. It is assumed that the reader is familiar with the concept that, for a given set of parallel planes, spaced d apart, the X-rays emitted from each plane will be in phase when

	nλ	=	2d sin θ
where	λ	=	wavelength of radiation
	θ	=	angle of incidence of radiation to the planes
	n	=	integer

X-Ray diffraction can be utilised to identify the (h, k, l) spacings of clay mineral crystals.^{*} hk spacings correspond to the a and b directions of the unit cell (except triclinic), whilst l gives spacings normal to the plane along c (basal) spacings.

Since clay minerals have mostly layer-lattice structures, the basal spacing can be used as a useful categorisation technique. The l spacing is also most susceptible to changes in environment - a property which can be utilised as an aid to identification.

The most common identification technique involves the preparation of an oriented sample, with detection of intensity of radiation made using a diffractometer - as shown in Fig. 4.5. Orientation of a specimen ensures that the basal plane lies parallel to the bed of the mount - in this way, reflections from planes other than basal planes is suppressed. Fig. 4.5 shows traces for both an un-oriented and oriented sample. For the un-oriented sample reflections from planes other than $(0\ 0\ 1)$ are apparent.

To identify positively certain mineral groups, some pre-treatment is necessary. This pre-treatment commonly includes heating to an elevated temperature and exposure to an ethylene glycol atmosphere. Carroll (1970) recommends the preparation of three oriented slides from the one soil suspension. Three traces can be obtained

^{*} For a discussion on Crystal systems see, for example, Van Vlack, L.H., "Elements of Materials Science", Addison Wesley.



FIGURE 4.5 - DIFFRACTOMETER TRACE

4-21

(a) normal,

- (b) after glycolation (to detect any swelling tendency), and
- (c) after heating (to determine changes in crystal structure).

If equation (1) is simplified to

$$\lambda = 2d \sin \theta$$

it can be seen that X-rays emitted from planes spaced d apart will be in phase. These reflections are known as first-order basal reflections from the (001) plane. Note that for n = 2, an apparent reflection from a plane $\frac{d}{2}$ is obtained. (The (002) plane). The intensity of reflection from these planes, and their spacings can be used to characterise the clay mineral. Table 4.4 summarises X-ray characteristics for certain clay minerals.

Having obtained a diffraction pattern (diffractogram), as shown in Fig. 4.5, the diffractogram can be compared with a set of "in-house" standards, obtained from minerals with a known composition or structure, or the d spacings obtained can be compared with d spacings of standard clay minerals as recorded in the ASTM Power Diffraction File. In this way, some indication can be gained of the minerals predominant in the unknown sample.

Example: Consider the trace for the oriented specimen (Fig. 4.5). The three most intense peaks occur at

$$\begin{split} I_1 &: \ 2\theta \ = \ 12.4^\circ \\ I_2 &: \ 2\theta \ = \ 25.0^\circ \\ I_3 &: \ 2\theta \ = \ 37.8^\circ \end{split}$$

Converting these 2θ values to d spacings via

$$d = \frac{\lambda}{2d \sin \theta} = \frac{1.54}{2 \sin \theta}$$
 [°]A

we have:

$$I_1 : d = 7.13^{\circ}A$$

 $I_2 : d = 3.56^{\circ}A$
 $I_3 : d = 2.38^{\circ}A$

Within experimental tolerances, these correspond to the 7.15°A (001), 3.57°A (002) for Kaolinite (Carroll, 1970).

TABLE 4.4

X-Ray Identification of Selected Clay Minerals (2µm) Oriented Mount

(After Carroll, 1970)

Mineral	Basal Spacings (001)	Glycolation Effect (1 hr, 60_C)	Heating Effect - 1 hr
Kaolinite	7.15°A (001) : 3.57°A (002)	No change	Becomes amorphous 550-600°C
Kaolinite- disordered	7.15°A (001) Broad: 3.75°A (002) Broad	No change	As above - at lower temperature
Halloysite 4H ₂ 0 2H ₂ 0	10°A (001) Broad 7.2°A (001) Broad	No change No change	Dehydrates to $2H_20$ form at 110°C Dehydrates at 125- 150°C
Illite	10°A (002) Broad	No change	Amorphous 560-590°C (001) Enhanced on heating
Smectite (Montmorillonite)	15°A (001) and integral series of basal spacings	(001) expands to 17°A	At 300°C (001) Becomes 9A
Mg Chlorite	14°A (001) and integral series of basal spacings	No change	(001) increases in intensity : < 800°C Mass Loss - No Structural change

We are further justified in predicting Kaolinite because of the very intense (001) and (002) reflections as compared with the (003) and (004). (004) at $2\theta = 51.1^{\circ}$)

Estimating the relative proportions of various clay minerals present in an unknown soil sample using X-ray diffraction is extremely difficult. There can be differences in the crystallinity of each component, varying degrees of hydration, variations in the distribution of the components in the mix, and other factors which act against accurate assessment. Where conditions are favourable, measurements of the area under the peaks at the principal spacing of each component can be compared to standards and some estimation of the relative proportions made. Carroll (1970) discussed procedures adopted, and limitations, in some detail.

X-ray diffraction is an extremely powerful, and most definitive method for identifying clay minerals. However, a complete identification will usually require additional testing. The range of testing is discussed in subsequent sections.

4.4.2 Thermal Analysis

(a) Differential Thermal Analysis

The application of Differential Thermal Analysis (DTA) to the identification of soils used in engineering applications was reported extensively by Lambe (1952). The technique has as its greatest advantage simplicity and rapidity. It is claimed that the order of accuracy for the determination of constituent minerals is of the same order as X-ray diffraction i.e. 5-10%. A schematic arrangement of the equipment involved is as shown in Fig. 4.6.

As a sample is heated, reactions will occur. If the heat absorbed or emitted during these reactions is compared to characteristics displayed by an inert material (usually calcined alumina) undergoing the same temperature change, a trace is obtained of differential thermal response versus temperature. (see Fig. 4.7).

If the sample absorbs heat with respect to the standard, a trough occurs in the trace (an endothermic reaction). Where the sample evolves heat compared to the standard, a peak occurs (an exothermic reaction). A heating rate of 10° C/min is usually adopted through a range up to 1000° C.

Important reactions include:

- (a) Dehydration (Endothermic)
 - (i) adsorbed water (driven off between 100-300°C)
 - (ii) crystal lattice water (the (OH) ions) termed dehydroxylation.Dehydroxylation causes a complete destruction of the mineral structure.



Figure 4.6 Essential Features- D.T.A. Apparatus



FIGURE 4.7 - THERMOGRAMS OF VARIOUS CLAY MINERALS (AFTER LAMBE, 1952)

- (b) Exothermic Reactions
 - (i) Crystallisation: new crystals may form from amorphous materials, or from old crystals destroyed at lower temperatures,
 - (ii) Oxidation: e.g. or organic matter between 250-450_C.

Lambe (1952) published a large variety of DTA traces performed on soils drawn from many parts of the world.

MacKenzie (1957) includes detailed discussion on the interpretation of traces and the quantitative estimation of proportions of components present in a sample.

Fig. 4.7 shows traces for four soils, taken from Lambe (1952). Note that the Halloysite is distinguished from the Kaolinite principally by the relatively large endotherm between 100-200°C, corresponding to the loss of adsorbed water.

(b) <u>Thermogravimetric Analysis</u>

Thermogravimetric Analysis (TGA) involves a measurement of the change in mass of a sample as it is heated.

Changes in mass of a sample can result from:

- (i) Loss of adsorbed water ($t < 200^{\circ}C$)
- (ii) Removal of lattice water ($t < 800^{\circ}C$)
- (iii) Loss of CO₂ from carbonates
- (iv) Removal of volatile components from non-clay mineral constituents in the sample
- (v) Oxidation, eg. Ferrous to Ferric Iron can produce an increase in mass of the sample.

Where it is important to minimise the mass change caused by non-clay components, pre-treatment of the sample is often performed, eg. removal of carbonates can be affected by repeated treatment with hydrochloric acid.

Typical dehydration curves for various clay minerals are shown in Fig. 4.8. An extensive tabulation of dehydration data can be found in Mielenz et. al. (1954).



FIGURE 4.8 - DEHYDRATION CURVES OF TYPICAL CLAY MINERALS (FROM WEBBER, 1972)

4.4.3 Specific Surface

The assumption underlying the determination of specific surface is that when exposed to an environment of glycol, the clay layer will adsorb a monomoledular layer of the glycol. Under some conditions this assumption is not true.

Clay layers will adsorb polar molecules from the liquid as well as the gaseous phase. Either ethylene glycol or glycerol is used as the environment. The test procedure involves determining the dry mass of sample, exposure in a glycol atmosphere and determination of mass of sample plus glycol retailed.

Specific Surface $(m^2/g) = \frac{Mg}{(Ms \ge 0.00031)}$ Mg = mass of glycol retained Ms = mass of dried sample 0.00031 = mass of glycol (in grams) required to form a monomoledular layer on a surface area of $1m^2$.

4.4.4 Cation Exchange Capacity

Cation exchange capacity (CEC) measures those ions bound loosely to the mineral surface by charge deficiency in the particular crystal lattice. One procedure used to determine CEC involves pre-heating the sample to remove soluble salts and organic matter, and then saturating potential sites with CaC1₂. Sodium acetate is used to replace the Ca²⁺ which is then determined in the washing solution by titration. For a more complete description see Jackson (1958).

4.4.5 <u>Chemical Analysis</u>

Chemical analysis gives composition in terms of elements - generally expressed as oxides on a percentage basis. As well as providing information on composition, a chemical analysis can be used for predicting performance at high temperature, (for burned clay products). Where K_20 is present in a clay mineral sample, the proportion can be used to determine the percentage illite present.

Table 4.5 gives typical values for glycol retention (specific surface), CEC, and K_{20} for selected clay minerals.

MINERAL	CLYCOL	CEC (meg/100g)	K ₂ 0 (%)
	RETENTION (mg/g)		
Kaolinite	16	3	0
Halloysite 4H ₂ 0	60	12	0
Halloysite 2H ₂ 0	35	12	0
Illite	60	25	8-10
Montmorillonite	300	85	0
Chlorite	30	40	0

TABLE 4.5 SELECTED CHARACTERISTICS OF CERTAIN CLAY MINERALS (from Mitchell, J.K., "Fundamentals of Soil Behaviour" Wiley, 1976)

4.4.6 <u>Electron Microscopy</u>

An electron beam performs a similar function to a light beam, but because the wavelength of the electron beam <<< the wavelength of visible light the potential resolving power of an electron microscope is much greater than that for a light microscope. Magnifications of 100 000+ can be achieved readily.

The transmission electron microscope (TEM), as its name implies relies on transmitting an electron beam through the sample. Since the penetrating power of an electron beam is low at the potential differences commonly employed, very thin specimens must be used.

For TEM work a replica is often made of the specimen surface. A replica is a thin film which is transparent to electrons. The replica, which can be used to great effect in textural studies will produce a negative impression of the soil surface - to overcome this a second replica is often taken, using the original as a base.

A scanning electron microscope (SEM) utilises a reflection geometry. An electron beam is scanned over the soil surface and the secondary electrons emitted are collected and displayed on a cathode ray tube. Whilst the resolution of the SEM is not as good as the TEM a greater depth of field is obtained - particularly useful for morphological studies.

Sample preparation for the SEM is relatively simple (although special techniques have to be used to dry the specimen and prepare a "clean" surface). Since a clay surface is non-conducting a thin layer of gold or gold alloy is plated onto the surface prior to examination.





Electron microscopy has been used for textural and morphological studies of clay minerals, examination of sample disturbance and examination of sheared zones. (See for example "Proceedings of the International Symposium on Soil Structures", Gothenburg, 1973, Morgenstern and Tchalenko (1967).

4.4.7 Field Identification

In some cases the presence of a soil type that may cause problems can be detected during a field inspection. Depending on the magnitude of the project, a field identification may provide an adequate categorisation of clay mineral type. Ingles and Metcalf (1972) have produced a simple identification table (Table 1.6) which utilised features such as colour of retained water, surface relief, rock type, erosion and soil profile as an aid to mineral identification. Emerson (1967) has devised a simple field (or laboratory test) which can be used to aid further the identification of predominant clay minerals.

4.5 <u>APPLICATIONS TO ENGINEERING PRACTICE</u>

An understanding of the mineralogy associated with clay soils can be useful as an aid to the complete explanation of observed behaviour. The following section gives examples of such behaviour.

4.5.1 Residential (Lightly Loaded) Slabs-on-ground

When a clay suffers a seasonal moisture change, movement associated with soil swell and shrinkage will occur. This movement will vary with depth and climate. Factors which influence seasonal movement of clay soils are:

- (i) Clay mineralogy
- (ii) Soil profile,
- (iii) Site drainage,
- (iv) Climate,
- (v) Superimposed loading, and
- (vi) Location and type of trees.

TABLE 4.6 FIELD IDENTIFICATION OF SOIL MINERALOGY (After Ingles & Metcalf (1972)

Turbid Water - Yellow Brown/Red Brown:	Soil Profile
Montmorillonites Illites	- Extensively cracked; wide, deep and
	closely spaced (50-60mm): Ca Rich
Clear Water: Ca, Mg, Fe Rich soils, highly	Illites Montmorillonites
acid soils	
	- open textured, appreciable clay:
Clear Water with Bluish Cast: Non-Saline	carbonates or kaolin (Never
Kaolinites	Montmorillonite seldom illite)
Frosion Gullies and/or field tunnelling:	- "wormy" appearance on exposed
Montmorillonites	weathered profile: Montmorillonite + soil
	salinity
(Mild gullying): Kaolinite	
	- relatively thin strongly bleached horizon
Landslips: Kaolinites, Chlorites	near soil surface (up to 600mm from top)
Country rock type - granitic: Kaolinites,	above bleach: fine silt
Mica	
	below bleach: dispersive soil
- poorly drained basaltic:	
Montmornionites	
- well drained basaltic: Kaolinites	
- Mudstones: Montmorillonites, Illite	
Soil profile - Mottled	
Red/orange/white: Kaolinite	
- Yellow/orange/grey or MedDark grey,	
Black: Montmorillonites	
- Brown and Red-brown clays: illite, some	
Montmormonte	
- white and lt-grey clays: Kaolinites	

For engineering applications, the predominant clay mineral type is rarely determined directly - mineralogy is inferred from other classification tests eg. Linear shrinkage and plasticity index. Clays where kaolinite is present as the predominant mineral will yield low values of Linear shrinkage and Atterberg Limits, whilst the expansive montmorillonite will result in correspondingly much higher values.

Frequently physical characteristics will minimise any tendency for soil movement. A layer of non-expansive soil over an expansive layer will minimise any swell. Excluding climate, other factors which reduce swelling include the thickness of the expansive layer and the drainage conditions of the site.

Extensive damage to lightly loaded structures has been caused by moisture loss in the soil beneath foundations caused by trees taking water in through their root systems.

Providing that the moisture conditions beneath a lightly loaded structure are maintained constant, no movement, swelling or shrinkage, of the soil will occur.

4.5.2 <u>Dispersive Soils</u>

It has been mentioned earlier that where forces of attraction between clay mineral particles exceed repulsion forces, a flocculated structure exists. If any change of environment occurs to change this relationship the clay mineral stacks can disintegrate and individual particles will go into suspension. A soil which follows this behaviour is known as dispersive. Where individual particles are very small, low seepage velocities will carry away particles in suspension and a loss of material will ensue. Such a loss can lead to failure of earthern structures eg. earthern channels or dams.

The mechanism which controls the deflocculation of a clay soil is associated with the ionic equilibrium between the counter ions (cations) associated with the clay surface and the concentration of ions in the bulk water phase. A disequilibrium can lead to structural failure.

Equilibrium can be upset when the ionic concentration of impounded water (as in the case of a dam) is significantly different to that for the "natural" soil water. Since rain water has a relatively low ionic concentration a soil prone to deflocculation can cause major problems in earth dams.

It is not intended to discuss the failure mechanism in detail. For further information on theory and applications see Ingles and Metcalf (1972).

4.5.3 Aggregates

To assist in the interpretation of the long-term behaviour of basalt aggregates used in concrete and road pavements, research projects have been conducted.

The basalt aggregate from some quarries includes material in which the primary rock forming minerals have been altered by weathering. The resulting secondary minerals can include expanding clay minerals.

To avoid any potential disruption to cured concrete or road bases a limit is set on the proportion of secondary minerals in the aggregate: in the case of concrete aggregate, 25%, whilst for road aggregate 20%.

REFERENCES

Bohor, B.F. and Hughes, R.E., "Scanning Electron Microscopy of Clays and Clay Minerals" Clays and Clay Minerals, Vol. 19, No. 1, 1971.

Brown, G. (Ed), The X-ray Identification and Crystal Structures of Clay Minerals", The mineralogical Society of London, 1961.

Carroll, D., "Clay Minerals: A Guide to Their X-ray Identification, The Geol. Soc. of America, Special paper 126, 1970.

Cole, W.F. and Beresford, F.D., "Evaluation of Basalt ... as an Aggregate for Concrete", Proc. ARRB., Vol. B, Pt. 3, Perth 1976.

Cole, W.F., Lancucki, C.J., and Nickson, N.M., "Ceramic Clays and Shales from the Melbourne Area" CSIRO, Div. Bldg. Research Tech. Paper no. 22. 1968.

Currey, D. "Extension course on Engineering Geology" Australian Geomechanics Society, Vic. Group, Melb. 1968.

Emerson, W.W. "A Classification of Soil Aggregates Based on their Coherence in Water". Aust. Journal of Soil Research Vol. 5, 1967.

Gillot, J.E., "Clay in Engineering Geology, Elsevier, Amsterdam 296p, 1968.

Grant, K., Ingles, O.G., Lawrence, I.A. and Sommerville, P.J., "Protecting a Dispersive Soil by Water Treatment", ASTM, STP, 623, 1976.

Grim, R.E., "Clay Mineralogy", 2nd Ed, McGraw Hill, NY, 596p 1968.

Holland, J.E., Washusen, J. and Cameron, D., "Ground Movement Information for Melbourne Soils...", Australian Geomechanics Society, Vic. Group, Symposium on Insitu Testing Testing for Design Parameters, Melb. Nov. 1975.

Ingles, O.G. and Metcalf, J.B., "Soil stabilization", Butterworths, Sydney, 366p, 1972.

Jackson, J.L., "Soil Chemical Analysis", Prentice hall, Englewood Cliffs New Jersey. 498 pp. 1958.

Kirkpatrick, W.M. and Rennie, I.A., "Clay Structure in Laboratory Prepared Samples", Proc. International Symposium on Soil Structure, pp. 103-112, Gothenburg, 1973.

Lambe, T.W., "Differential Thermal Analysis", Proc. H.R.B. Vol. 31, pp. 620-641, 1952.

Lambe, T.W. and Whitman, R.V., "Soil Mechanics", Wiley, New York, 553p, 1969.

Lee, I.K. (Ed) "Soil Mechanics - Selected Topics" (In particular Ch. 1) Butterworths, London, 1968.

McBride, M.B., "Origin and Position of Exchange Sites in Kaolinite", Clays and Clay Minerals, Vol. 24, No. 2, 1976.

MacKenzie, R.D. (Ed) "The Differential Thermal Investigation of Clays" Min. Soc., London, 456 pp. 1957.

Mielenz, R.C., Schieltz, N.C., and King, M.E., "Thermogravimetric Analysis of Clay and Clay-Like Minerals. Proc. Nat. Conf. Clays and Clay Minerals, 1953.

Mitchell, J.K., "Fundamentals of Soil Behaviour", Wiley, New York, 422p, 1976.

Morgenstern, N.R. and Tchalenko, J.S., "Microscopic Studies in kaolin Subjected to Direct Shear", geotechnique, Vol. 17, No. 4, 1967.

Neilson, J.L., "The Silvrian Rocks of the Melbourne Region...", Australian Geomechanics Society, Vic. Group, Workshop on Engineering Properties of Melbourne Mudstone, Melb., April 1977.

Webber, J.R., "A Comparison of the Results of the Washington Degradation Test...", Country Roads Board of Victoria Research Memorandum No. 16, 1972.