

"THE NATURE AND ENGINEERING PROPERTIES
OF
SOME RED SOILS FROM NORTH-EAST BRAZIL"

BY

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"To Ibidunni Akintokun"

SYNOPSIS

Researchers over the years have demonstrated the need for the determination of the nature and fundamental properties of soils as an approach to the understanding of engineering behaviour. This has been shown to be particularly true in the case of tropical soils.

This thesis approaches the analyses of engineering behaviour from a standpoint of the study of the intrinsic properties. The mineralogy, chemistry, structure and strength behaviour of three concretionary red soils from Paraiba State in North-East Brazil, have been determined.

The mineralogy of the red soils were determined using X-ray analysis, differential thermal analysis and thermogravimetric techniques. The microstructure of the red soils has been examined with the optical and scanning electron microscopes. The strength behaviour of compacted specimens was examined in the oedometer, direct shear and triaxial machines.

A phenomenon of breakdown of soil particles under stress is isolated. The influence of the phenomenon on the strength behaviour is demonstrated. The soil elements and properties responsible for the phenomenon are discussed.

LIST OF SYMBOLS

τ	shear stress
τ_m	max. shear stress
σ	normal stress
σ'	effective normal stress
σ_1	major principal stress
σ_2	minor principal stress
u	pore pressure
u_a	pore-air pressure
u_w	pore-water pressure
ϵ	axial strain
h	axial deformation
A B	Skempton's pore pressure parameters
χ	coefficient defining effective stress in partially saturated soils
c	cohesion
c'	effective cohesion
c_u	cohesion in the undrained test
c_{cu}	cohesion in the consolidated-undrained test
c_r	residual cohesion
ϕ	angle of friction
ϕ'	effective angle of friction
ϕ_u	angle of friction in the undrained test
ϕ_{cu}	angle of friction in the consolidated-undrained test
ϕ_r	residual angle of friction

γ_d	dry density
G	specific gravity
e	void ratio
η	porosity
w	moisture content
S_r	degree of saturation
LI	liquid index
LL	liquid limit
PI	plasticity index
PL	plastic limit

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CHAPTER 1

INTRODUCTION

Major construction (particularly dams, roads and airfields) in countries lying within the tropical regions of the world, necessitates the use of local materials. Many of these materials are suitable from an engineering point of view, some are not, but the onus of the differentiation lies with the engineer who, by and large, has been instructed in the methods for materials found elsewhere (temperate regions) which might not be directly applicable to those of these areas.

The main problem with regard to construction with tropically weathered soils is not in non-suitability but lack of adequate knowledge of behaviour under different conditions and at varying states.

In the last few years a number of studies have been carried out covering problems, properties and behaviours encountered with the use of tropical soils. Most of this work largely dealt with characteristics observed during testing and evaluation of these materials, but few have actually gone 'beyond the bridge' to procure reasons for these characteristics.

Gidigas (1970) mentioned that the behaviour of red soils cannot be adequately explained by consideration of physical properties alone, emphasis has to be placed on the intrinsic properties and physico-chemical factors that make for the genesis of the soils. He particularly states that classification of the soils would have to be based on these factors before any reasonable 'sorting' can be achieved.

Lohnes and Demirel (1973) agreed with Gidigas in the use of soil forming factors as a basis for correlation.

These factors have influence on the physico-chemical, chemical and mineralogical properties which, in turn, play a hand in the engineering behaviour and, it is felt that any study of the engineering characteristics must be linked with the mentioned properties for full understanding.

De Graft-Johnson and Bhatia (1970) called attention to the scarcity of information on the engineering characteristics of these soils, for if knowledge about them is to be advanced, information and behaviour of materials from various parts of the world has to be known for any meaningful correlations.

Since this call, a number of studies have been carried out, notably by institutions; Building and Road Research Institute, Ghana, United States Agency for International Development. Lyons Associates (1971) working for U.S. AID have produced reports and papers based on general studies and statistical appraisal of these materials as used in Africa.

Though these contributions are important steps forwards, a great amount of work still needs to be carried out and significant researches are required, for the understanding of these materials. They cover a great part of the tropics and their use in many cases is a question of compulsion, since in some parts no other materials are available.

A number of investigations have been made concerning the nature and the behaviour of red soils in Brazil, mentionable are the work of Vargas (1953, 1975) on physical properties and de Medina (1964) on behaviour and application in highway construction. Some others are found in scattered and non-coherent reports and publications. Thus a considerable amount of information is required, for knowledge of the engineering of red soils in this region.

This work was carried out to determine the nature (intrinsic

parameters) of red soils in the States of Paraiba, N.E. Brazil and to observe the influence of this parameter on the engineering properties. This study is particularly necessary not only because of the lack of knowledge of red soils in these parts, but also for the non-availability of other materials for construction (particularly aggregates) as well as the rashness of the local engineers in using red soils for roads and airfield pavements despite their lack of adequate knowledge of the materials.

Samples from different depths of three occurrences of concretionary red soils were examined. The properties evaluated are the chemical and mineralogical compositions, the physical distribution of the various soil components as seen under the microscope at different levels of magnification and the strength behaviour of the soil when subjected to standard physical tests.

In the following pages of the thesis, the words 'red soils' are used to represent all tropically weathered soils which have been involved in the accumulation of certain components (mainly of iron and/or alumina) and the removal of others (mainly bases).

The word 'laterite' (which may be synonymous) will, however, be found in the descriptions of works that have been done previously. It will also be found where passages are quoted from works in which the term is used.

Arguments for the need to replace the term laterite with the words 'red soils' are presented in 2.1.

CHAPTER 2

HISTORICAL BACKGROUND AND GENERAL REVIEW OF LITERATURE

Since Buchanan's encounter in 1807 in India, of materials used for building, which was recorded in his book 'A Journey from Madras through the Country of Mysore, Canara and Malabar', to which he gave the name 'laterite', controversies have been going on as to definition, description and origin of similar materials found elsewhere. Justification for giving this name to other materials has been based on similarity of colour, composition, occurrence and behaviour. Much of the problem with 'laterites' is not unconnected with the widespread, though differing in concept, usage of the name for different materials.

In contrast to engineering there exists a vast amount of literature (though lacking in concensus) on the geology, genesis and pedology of soils and materials.

Maignien (1966 p.16) considers that 'literature abundance about laterite is because it is not uniquely identified with any particular parent rock, geological age, single way of formation, climate per se, or geographical location. It is rather a response to a set of regionally varying physiochemical conditions, which are still far from being fully known in their interation. Hence untimely generalisations on the basis of too limited experience occurred'. These generalisations mainly served to bring confusion to the scientific study of red soils.

2.1 Definition

What's in a name? That which we call
a rose/By any other name would smell
as sweet.

(Shakespeare - Romeo and Juliet)

When Buchanan encountered the material in India (Buchanan 1807), he made some observations concerning the nature and the properties of the material. He wrote on p.440 of the book:

"...It is full of cavities and pores and contains a very large quantity of iron in the form of red and yellow ochres. In the mass, while excluded from the air, it is so soft that any iron instrument readily cuts it, and is dug up in square masses with a pick axe, and immediately cut into the shape wanted with a trowel or large knife. It very soon after becomes as hard as a brick..."

He continued,

"...As it is usually cut into the form of bricks for building, in several of the native dialects it is called the brickstone (Itica cullu)...The most proper English name would be Laterite, from Lateritis, the appellation that may be given to it in Science."

From the passages the following points clearly emerge, with regard to the nature and properties possessed by the material that Buchanan saw:

- (1) (a) It was red or yellow.
- (b) It contained iron. This is an inference from the colour as a result of the general association of iron with red colour.
- (2) It was soft in situ, but capable of hardening on exposure.
- (3) It was used for making bricks as a result of the hardening property.

A final point concerns the names that should be given to the material:

- (4) Buchanan gave two names. An English one: Laterite, for general use. Another: Lateritis, for scientific use.

It is perhaps an interesting fact that, had the investigators after Buchanan, stuck to his original suggestion of Laterite for general use

and Lateritis for scientific use, the controversies surrounding the definition would have been resolved. These controversies, which are to be discussed consistently deal with the word 'laterite'. It had been used both in general terms and as a scientific term. If the post-Buchanan investigators had used the word lateritis in scientific terms, they would have found, after the chemical analyses were available, that it would contain a wide range of minerals in various proportions. It also had a wide range of properties. The argument for scientific use to characterise a particular nature or property then becomes untenable.

The word Lateritis never became accepted in scientific literature. Laterite was used both as a scientific term and an English word.

Controversies concerning the definition actually started at about the beginning of the present century. This was mainly due to the background of the different workers concerned with 'laterite'. Newbold (1844) had given detailed descriptions of 'laterites' in India and made particular reference to hardness. The property of hardness became accepted by geologists in India as a criteria for defining laterite. Engineers who were concerned with the utilitarian aspect naturally accepted the definition based on physical property. They felt particularly justified since the word laterite is a derivation from 'Later' in Latin meaning bricks. Scrivenor (1909), a geologist, was the voice of the engineers. He considers the term to be an engineer's rather than a geologist's. He suggested the use of the word 'laterite' in a general sense.

Certain other geologists felt that Buchanan had hinted at scientific investigation. Since chemical analyses were available by then, they maintained that the term should be a scientific name (Crook (1909), Evans (1910)).

The background to the controversies and their continuous development over the years were fueled by the different backgrounds of the investigators. Each gave primary concern to his discipline. Bauer in Germany (1898, 1907) analysed the different stages of the product of weathering of granite and diorite from the Seychelle Islands. The final product was found to contain alumina in quantities to give a mineral then known as bauxite (found later to be a mixture of gibbsite and diaspore). As a geologist he considered the possibility of the economic use of the material. He stressed the importance of alumina. Holland (1903), who was then the Director of the Geological Survey of India, made this fact known to the English-speaking world. In addition to publishing Bauer's result, he published some results of chemical analyses carried out on materials from India. These materials were also mostly aluminous.

Warth and Warth in the same year (1903) published detailed chemical analyses of 23 samples from India, all of which they referred to as laterite. They found that the alumina content varied from high (above 60%) to low (6%). They categorically stated that (p.159): "...Laterite is bauxite in various degrees of purity, from the richest wocheinite down to such specimens in which free alumina has entirely disappeared."

The idea that alumina is the main constituent of 'laterite' became rapidly popularised. By 1909 when Harrison wrote a book on the "Geology of the Goldfields of British Guiana", he was chided by a reviewer (Bull. Imp. Inst. 1909, vii, p.133) as using the term "wider than is either used or desirable" (see Scrivenor, 1909). The reviewer stated that: "...only products of weathering containing free alumina in hot, moist climates should be considered as laterite."

Other reports were published using the term laterite not in the strict sense as used by the reviewer Mennel (1909), Harrison (1910).

Fermor (1911) suggested the expansion of the word laterite to cover material containing oxides and hydroxides of iron, with or without titania. These he referred to as 'lateritic constituents'. He proceeded to classify laterite on the basis of their mode of formation and the amount of lateritic constituents. He employed a variety of terminologies which only added to the confusion, e.g. true laterite, lithomargic clay, lithomargic laterite, lake laterite, lateritoid. Strangely enough, it was adopted by some authors (Holmes 1914, 1945) and recently by Gaskin (1975) in a paper published posthumously. On the other hand, it was criticised by Simpson (1912). He suggested the reversion to a classification already used in India: primary laterite and secondary laterite. This is based on the locality of the occurrence in the topographic profile.

Lacroix (1913) agreed with Fermor (1911) on the use of percentages, but considered the total content of hydrated oxides as more important a basis than only iron of Fermor.

Fermor's paper received wide attention and the term 'lateritic' was introduced into terminology. Ultimately any tropical red earth was referred to as laterite or lateritic.

The next group of scientists to come into the discussion were the pedologists. They became interested because 'laterite' was found as a superficial deposit on top of rocks, that sometimes have growing vegetation.

Glinka (1914) was probably the first pedologist to discuss 'laterite'. He considered 'laterite' to be characteristic of soils in the forest zone of the tropics. Martin and Doyné (1927) were the first to carry out chemical analyses of different particle sizes in the soil. They proceeded to classify 'laterites' on the basis of the silica/alumina ratio of the clay fraction. The influence of the professional bias is

expressed on p.545 of their publication. A reason given for using the clay fraction rather than the whole soil is: "...the chemical composition of the highly insoluble (in water) coarse fraction is not so important from an agricultural point of view", as the chemical composition of clay.

Though many of the references quoted by them did not refer to 'laterite' as a soil, they as agriculturalists took it to be so, and referred to it accordingly. Glinka (1914) had however put forward the idea that laterite is a soil. This idea was popularised by Harrasowitz (1930) and in line with the dominant views of the Russian School of Pedology (Dokuchaev's concept of the zonal distribution of soil - see Joffe, 1949, p.8), he associated laterite with a characteristic profile developed under tropical savannah. He suggested four zones:

- (i) a fresh zone;
- (ii) a zone of primary alteration to kaolinite;
- (iii) a lateritic bed proper;
- (iv) a surface zone with ferruginous incrustations.

The zonal distribution implies that accumulation is predominant in particular zones. But a term 'podzol' had existed for soils that involve iron accumulation in a lower horizon. It seemed undesirable to use the word laterite for both the residual soils and the lower horizons of a podzol. Attempts at a distinction was drawn by Marbut (Thorpe and Baldwin, 1940). He suggested that 'tropical podzol soil' should be used for soils in which there is iron enrichment in the lower horizon and 'laterite soil' for soils in which the upper horizon is involved in iron enrichment.

By the third and fourth decades of the twentieth century, it had become difficult to know what the term 'laterite' means. Since then and up until the present date, the bizarre practice of using the term

and proceeding to define the limits is used. The problem that usually arises is that two entirely different materials could be discussed under the same name. Recent examples are Gaskin (1975) who used Fermor's (1911) and McFarlane (1976 Introduction p.vi) who used Sivarajasingham et al's (1962).

When the confusion associated with the term laterite became enormous, investigators advocated the use of the term in a particular sense and suggested it for wide acceptance. Pendleton (1936) strongly urged for the restriction of the term to what he considered to be Buchanan's original sense, one in which a 'laterite' horizon is found in the profile. Pendleton and Sharasuvana (1936) separated laterite and lateritic soils. They considered a laterite soil as one in which a laterite horizon is found in the profile and a lateritic soil to be one (p.434): "...in which it is believed a true laterite horizon will develop if the prevailing conditions persist long enough." The disciplines concerned with the study of 'laterites' have all become exact sciences and articles of faith will obviously not find a place in these areas.

The chemical definition did not provide a solution either for Fox (1936) published the results of analyses carried out on samples from Buchanan's original site. He found little uncombined alumina. Aluminium occurred mainly in the clay mineral kaolinite. According to Fermor (1911), Lacroiz (1913), Martin and Doyne (1927) this would not be a laterite.

As a result of the ambiguity associated with the term 'laterite' certain workers advocated the dropping out of the term altogether in scientific literature, (Scrivenor (1930)). Some other terminologies have been introduced to refer to the material and the process by which it is formed. The word 'ferrallitisation' has been used for the

process, Scrivenor (1930), Vine (1949). Kellogg (1949) introduced the term latosols for sesquioxide-rich soils, soils that were not hard and do not harden on exposure. He retained the term 'laterite' for those that were hard or hardened on exposure. The Soil Survey staff of the United States Department of Agriculture (USDA, 1960) adopted the term 'plinthite' (Greek plinthos, brick) for what Kellogg called 'laterite'. They also introduced oxisols for what had been called laterite and lateritic soils.

No consensus was reached or has been reached in the use of the terms introduced. This is hardly surprising since the precursor 'laterite' has been tossed about for 170 years without any agreement on the use.

Engineers who later came to study the soils scientifically can be practically commended for not adding further complications to the problem of definition. They simply picked on the available ones based on chemical, physical or profile characteristics, Winterkorn and Chandrasekharan (1951), Bawa (1957) used Joachim and Kandiah's (1941) silica-sesquioxide ratio. Remillon (1967) used D'Hoore's (1964) general classification, based on climate and vegetation. Winterkorn and Chandrasekharan's (1951) idea based only on the silica/sesquioxide ratio finds considerable following among Brazilian engineers. It has been recommended by Santana (1970). It is also favoured by Wernicke and de Barros (1974) who have presented arguments for its adoption by civil engineers. Portuguese civil engineers (Laboratorio Nacional de Engenharia Civil (LNEC)) have proposed a definition (Novais-Ferreira (1963)) based on the physical characteristics, chemical composition and mineralogical content. They identified laterite, laterite soil, lateritic soil and lateritic terrain. It can be seen that new definitions employing the term only add to the confusion.

The possibility of restricting the use of the term laterite, laterite soil and lateritic soil to particular groups of soil, with well-defined characteristics, appears extremely difficult. The present writer will agree with the previous workers who suggested the total abandonment of the term. It is no use giving a set of definitions to a term which others will refer to, in an entirely different manner.

McFarlane (1976, Introduction p.vi) in a recent work considers the use of the term laterite in the Kellogg (1949) sense which was adopted by Sivarajasingham et al (1962). This definition which has received some following in the United States of America, (Alexander and Cady (1962), Persons (1970)) specifically excludes material that will not harden on exposure. The property of hardening on exposure is time dependent. A non-laterite of today could be a laterite in a year.

The total abandonment of the term is therefore recommended at least, with reference to engineering. Engineering interest in tropical soil began to develop in the last twenty-five years. Engineers are therefore advised not to get embroiled in the confusion concerning definition. It does not really matter whether a material came to be called a laterite, lateritic soil or laterite soil, what one is interested in is the suitability or non-suitability for particular purposes.

Australian terminologies look promising. Woolnough (1927) introduced the word 'duricrust' to represent all forms of superficial crust. This embraced iron rich crust (ferricrete) silica rich (silcrete) and aluminium rich (alcrete), (Goudie, 1973, p.5). The terms ferricrete and silcrete were introduced by Lamplugh in 1907. Dury's (1969) classification is however, not recommended.

Dury (1960) suggested a classification of duricrust based on the 'essential chemistry' of the bulk content by weight. He distinguishes the following (Dury, 1969, p.80):

Name	'Essential chemistry'
silitic	SiO_2
siallitic	$\text{SiO}_2, \text{Al}_2\text{O}_3$
fersilitic	$\text{Fe}_2\text{O}_3, \text{SiO}_2$
fersiallitic	$\text{Fe}_2\text{O}_3, \text{FeOOH}, \text{SiO}_2, \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}, \text{AlOOH}$
ferrallitic	$\text{Fe}_2\text{O}_3, \text{FeOOH}, \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}, \text{AlOOH}$
ferritic	$\text{Fe}_2\text{O}_3, \text{FeOOH}$
fermangnitic	$\text{Fe}_2\text{O}_3, \text{MnO}_2$
tiallitic	$\text{TiO}_2, \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$
allitic	$\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}, \text{AlOOH}$

Dury specifically mentioned that 'disregard of mineralogy' is one of the principles adopted. The writer cannot see any disregard for mineralogy in the classification shown above. What Dury calls 'essential chemistry' are chemical formulas for minerals. A classification based on 'bulk content' (Dury 1969, p.79) would be expected to give the bulk chemical composition and separate the distribution of the elements into specified minerals. And if the classification is based on mineralogy all the inadequacies found in such definitions as those of Fernor (1911), Lacroix (1913) will apply.

Since engineers would not be expected and could not go into detailed study of chemistry and mineralogy in routine soil analysis, it is suggested that the term 'laterite' should be dropped altogether from scientific use. The words 'red soils' are recommended for use by them. This will include all tropically weathered soil that involves the accumulation of sesquioxides in the soil. Classification can be carried out based on such physical properties that are pertinent to engineering.

2.2 Nature of Red Soils:

Red soils are tropically weathered soils with a high concentration of sesquioxides of iron and/or alumina. They have correspondingly low content of alkalis and alkaline earths. They exist in wide ranges of chemical composition. Silica content varies from low to medium and exists usually as kaolinite, whenever it is found in substantial amounts.

2.2.1 Colour and chemistry:

The presence of iron oxides in various states of hydration gives red soils a range of colours. Shades most frequently encountered are pink, ochre, red and brown. The presence of manganese gives purple colour or shades of it to the soils. No agreement exists on the usefulness of colour in the study of red soils. Maignien (1966, p.19) considers that colour can be a useful parameter in determining the degree of evolution. Coleman et al. (1964) and Coleman (1965) made extensive qualitative and quantitative study of soil mineralogy based on colour and suggests its usefulness. On the other hand, Waegemans and Henry (1954) concluded that except for extremes of colouration corresponding to haematite and to goethite, there does not seem to be any relationship between colour and index of hydration. Colour is a very empirical parameter, since the minerals in soils are known to present different coloration under varying conditions. In addition to this is the masking effect of organic matter on the mineral colour (Mohr and van Baren, 1954, p.425).

The nature of red soils was never much of a controversy as the nature of what should be called 'laterite'.

Controversies are found only in the works before 1900 and in the earlier part of this century (see Harrison, 1910). Arguments on the nature have been long resolved but disagreement still exists when the

term 'laterite' is employed to represent a particular type of red soil.

The first descriptions on the chemical characteristics of tropical red soils were made by Bauer (1898), though Oldham (1893) had published a less comprehensive result of chemical composition of some samples in his book on the Geology of India, p.130. Bauer (1898) analysed samples from the Seychelles and found they contained mostly of aluminium hydroxide with small amounts of iron and combined silica. He compared the composition with that of German bauxites. Warth and Warth (1903) analysed 23 samples from India and found that some contained small amounts of alumina but were rich in iron oxides, while others contained large amounts of alumina and were poor in iron oxides. This showed that the chemical composition of red soils ranges from almost pure alumina to almost pure iron oxide. They, in agreement with Holland (1903) however, considered alumina as the most important oxide.

With the work of Harrison (1910) and Fermor (1911) it became clear that aluminium also exists in the form of alumino-silicates in the soils and these had to be considered. Later, it became generally accepted that products of weathering in the tropics are mixed in various proportions and that they portray the following characteristics (Martin and Doyne, 1927):

- (a) They are usually lacking in alkalis and alkaline earths. It can however be found in quantities where primary mineral particles (e.g. feldspars) are still present.
- (b) Combined silica is usually low, at least when compared with that of the original rock. When found it is usually in the form of clay mineral kaolinite.
- (c) Some red soils contain manganese, sometimes in sufficient quantity to be exploited as a mineral. The same applies to titanium which is frequently identified and to a lesser extent vanadium and

chromium.

- (d) Combine water determined by loss of ignition is always present in appreciable amounts. It is more abundant in aluminous than ferruginous ones.
- (e) Quartz when present, is an important mineral. It is mostly residual but it can be neogenetic.

2.2.2 Structure:

The presence of sesquioxides of iron and alumina in different states and their behavioural tendencies give red soils a characteristic structure, which differs from those found in temperate zone soils.

With reference to the actual physical occurrence, three structural patterns predominate (Maignien, 1966, p.18):

- (i) The indurated elements form a continuous coherent skeleton.
- (ii) The indurated elements are free concretions or nodules in an earth matrix.
- (iii) The indurated elements cement pre-existing material.

Morphological investigations reveal that a number of macrostructural types can be distinguished. These include vesicular - having small bladderlike cavities; vermicular - having small sinuous wormlike tunnels; cellular - having many small cavities which may be connected; oolitic and pisolitic - resembling a pea (Pullan, 1967).

The differences in macrostructures does not necessarily mean differences in microstructure. Alexander and Cady (1962) observed similarity in microstructure, despite differences in macromorphology of indurated red soils from West Africa. Sections studied by these authors revealed spheroidal or slightly elongated elements within a finer mass.

Uehara et al (1962) have suggested that the structure of Hawaiian soils is determined by mineralogy, which in turn is dependent on the

climate. They considered the formation of stable aggregates to be a function of the nature and arrangement of soil minerals with soil pedological units. It seems that the formation of stable aggregates in soil would not only depend on the form of clay but the degree of crystallinity. Baver (1972) mentioned that the aggregate stability of kaolinite soils with more than 5 per cent free iron oxide is generally related to soil anisotropy. Tropical soils which contain amorphous or non-platy clay would be expected to have poorly developed structure.

2.2.3 Mineralogy:

The form of the element can also be as varied as the general chemical compositions. The elements are found mainly as oxides, hydroxides and silicates (Maignien 1966, p.22).

Iron can exist in the form of

Haematite $\alpha - \text{Fe}_2\text{O}_3$

Goethite $\gamma - \text{FeOOH}$

Lepidocrocite $\gamma - \text{FeOOH}$

It also occurs as amorphous hydroxides whose physical constitution is indeterminate.

Aluminium can exist in the form of

Gibbsite $\alpha - \text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$

Boehmite $\gamma - \text{AlOOH}$

Diaspore $\gamma - \text{AlO(OH)}$ (not common)

as well as amorphous alumina and clays.

Silica exists in the form of quartz as well as amorphous silica and clays. The common clays are

Kaolinite

Halloysite

and Montmorillonite to a lesser extent.

An alumino-silicate with non-defined structure can also be present. This is referred to as allophane.

Titanium exists in the form of anatase. It can also occur as a weathering resistant mineral-rutile.

2.3 Engineering Properties:

There is a scarcity of reports on the engineering properties of red soils. The available ones are reviewed here giving consideration to those pertinent to the problems encountered when using red soils as construction materials and particular reference is made to the apparent contradictions in many of the reports. These contradictions again express the problem of using a single name for different materials.

2.3.1 Grain Size:

The grain size analysis, which is a useful classification tool and method of evaluation has been found to be highly dependent on the amount of working and pre-treatment prior to testing. Nixon and Skipp (1957) used a mixture of hydrogen peroxide + hydrochloric acid, and hydrogen peroxide only in the dispersion of some samples from Jinja, Uganda, prior to the determination of the grain sizes.* The particle size distribution curves obtained from the soil without pre-treatment was found to differ from those of the dispersed soils which again differed greatly among themselves.

Terzaghi (1958) recorded an increase of up to 20 per cent in the clay content of Sasuma clay, Kenya, if sodium hexametaphosphate as opposed to sodium oxalate was used as the dispersing agent. He also recorded a decrease in the clay content when the soil was oven dried.

The method of handling and type of pre-treatment has also been found

to affect the consistency limits. Sherwood (1967) reported an increase in liquid limit of Kabete clay, but found no significant change in the value of the plastic limit.

2.3.2 Compaction characteristics:

The compaction characteristics of red soils which are used in engineering evaluation are responsive to a number of factors such as grading, predominant clay mineral, crushing strength of coarse material, degree of weathering and placement condition.

Newill (1961) recorded changes in the compaction characteristics of red clays of Kenya due to drying of the soil, but mentioned that this does not necessarily mean fundamental changes in properties, for he found that the soil contained halloysite (which has some interlayer water within its structure), and if the interlayer water was taken into consideration, little change will be recorded when compared with that of a natural soil.

Tateishi (1967) however, recorded irreversible changes when soils from Hawaii were dried. He found that drying of clayey, plastic soil produces one with granular characteristics with an increase in the maximum dry density and a decrease in the optimum moisture content. Wallace (1973) also recorded similar behaviour for soils formed over similar rocks (volcanics in the continuously wet highlands of Papua, New Guinea. He attributed the irreversible changes to the presence of allophane minerals. It therefore seems that soils developed over volcanics in regions of high rainfall have the propensity for producing significant change, if not irreversibility on drying.

Matyas (1969) determined the effect of working on Sasumua clay which was described by Terzaghi in 1958. He re-compacted trimmings on materials from preparation of samples of strength tests which had

earlier been compacted and found an increase in the value of the maximum dry density without a change in the optimum water content. This increase in density he attributed to a breakdown in clay aggregates.

Evans (1958) however, did not obtain any differences in compaction characteristics even after the breakdown of coarse fraction of gravels. De Graft-Johnson and Bhatia (1970, p.23) summing up on the apparent contradictions of the reports on the moisture-density characteristics stated that "...studies on the compaction characteristics of Ghana lateritic soils would indicate that the change in the moisture-density relationship is greatly influenced by the initial grading of the material as well as the mechanical strength of the particles." The resulting strength could be increased or decreased during compaction.

The contradiction like any other, is not unconnected with usage of the term 'laterite'. For as it stands, 'laterite' could be clay, sand or gravel that is iron rich. These would normally be expected to behave differently.

2.3.3 Shear strength:

The strength of red soils like that of any other soil depends on the friction between particles and cohesion or bonding between them. However, unlike most clays of the temperate zone, bonding does not only involve charge interaction with electrically charged particles within the system, but also cementation with resultant production of cemented materials. These cemented materials bear little resemblance to original clay to clay picture in soil behaviour. Cementation of materials goes on to a stage where high stability of aggregates, produced by the sesquioxides, result in a near granular material. This near-granular material, though of clay origin, would not be expected to behave like materials with real cohesion. Mixture of these clays (in the true sense of the word) combine

to produce varying strength behaviour of red soils.

De Graft-Johnson et al (1968) carried out tests on compacted lateritic clay from the forest zone of Ghana and found that the angle of friction in terms of total stress was dependent on density and moulding water content as well as structure.

Terzaghi (1958) carried out drained and undrained tests with pore pressure measurements on samples of clay recovered from the compacted fill of a dam at Sasumua in Kenya. He found that the effective angle of friction ϕ' , was independent of the water content (30 - 36 per cent) and that effective cohesion C' could be expressed by an empirical relation:

$$C' = ae^{-bw}$$

where w = water content

e = base of natural log, and

a and b are constants.

Matyas (1969) also carried out triaxial tests on compacted samples of the Sasumua clay and found that ϕ' was not significantly dependent on placement water content but that C' was dependent on placement water content and dry density. He also reported that residual effective cohesion C_r' and residual effective angle of friction ϕ_r' gave consistent values whatever the conditions.

Baldovin (1969) reported that the undrained cohesion C_u and undrained angle of friction ϕ_u , of samples compacted by kneading was influenced by the moulding water content. High values were recorded for unsoaked samples which were reduced sharply on soaking.

It appears that strength behaviour is not only influenced by the mechanical properties such as friction and cohesion but also by physico-chemical factors (Gidigasu 1976, p.344). These factors are imposed by the chemistry and mineralogy. The role of mineralogy is demonstrated

later in the thesis (Chapter 9).

2.4 Genesis of Red Soils:

2.4.1 Historical background:

As red soils were found lying on rock (Oldham, 1893, p.377) and was even referred to as a rock (Newbold, 1844, Oldham, 1893, p.369), it is not surprising that geologists were the first to postulate hypothesis concerning their origin. The study of 'laterite' was rapidly popularised among geologists and in the nineteenth century it was fashionable to postulate about the genesis of red soils (Fermor, 1911).

The accepted theories at the present date (1977) are only modifications of the early theories. Three periods can be distinguished:

- (i) An early period when general observations of the occurrences were carried out. Comparison with occurrences elsewhere allowed postulation of the origin of the material. This was the first half of the nineteenth century.
- (ii) A second period when a closer observation of the morphology was done. Samples from the occurrences were analysed. The operating environmental conditions (tropical) and the physical process were considered before the hypotheses were put forward. This period started at the end of the nineteenth century and continued until recently.
- (iii) Paralleling the later stages of the second period is the last few years, when attention is being given to the physical, physicochemical and chemical conditions. This approach has been assisted to a large extent by the modern instrumental methods of soil analysis. Also is the considerable amount of advancement concerning the knowledge of the nature and properties of materials found in the surface of the earth.

Many hypotheses were suggested in the early period. The excitement generated by one hypothesis invited others. By 1890, Lake was in a position to give a review of a considerable number of theories in a report on the geology of South Malabar.

The early hypotheses summarised by Lake (1890) are:

- (a) Red soils are a residual product of weathering in-place.
- (b) Red soils are detrital
- (c) Red soils are sedimentary
- (d) Red soils are of volcanic origin

The hypotheses concerning the sedimentary (marine) and volcanic origin of red soils, did not receive much attention and were readily dismissed by Oldham (1893), p.386). The other two have persisted to date, though the mechanics suggested for the process have evolved with time.

The idea that a red soil is a residual product of the removal of certain constituents, leaving a residue, was advanced in the early part of the nineteenth century, (Babington, 1821, Clark, 1838). It became widely accepted after Russell's (1889) emphasis on the solvent power of the water percolating through rocks in warm moist climates.

The hypothesis considered that the sesquioxides accumulated are immobile and left behind while mobile constituents such as alkalis, alkaline earths and silica are removed. McFarlane (1976, p.92) has recently identified a short-lived mobility of the 'immobile' constituents. This is prior to reorganisation that takes place in the soil after accumulation.

The idea that red soils develop from detrital deposits on slope is probably the only one that has not survived modification, since it was introduced by nineteenth century geologists Blanford (1859), (Theobald, 1873). From the idea that hardened red soils could result

from cementation of slope deposits came the exposition of the obvious underlying assumption that iron was mobile. This came to be developed to produce the concept of vertical movement of iron, from one horizon to another in a single profile, i.e. non-detrital red soil could result from the precipitation of iron-oxide in a horizon.

Campbell (1917) was the first person to express in clear terms the concept of non-detrital 'laterite' being precipitatory. Though it had been discussed earlier (Maclaren, 1906, Simpson, 1912).

This concept has survived until today, almost in the original form as suggested by Campbell. The concept has survived because it came to agree well with observed field characteristics. For example, it explained the existence of pallid zones (zones that are depleted in iron) beneath accumulation zones in red soils. Also is the wide occurrence of red soils in grasslands (Harraso-witz, 1930). Two hypotheses exist to explain the movement from the base of the profile to the top as it is usually seen:

- (i) Capillary movement of water through pores in the soil.
- (ii) Moving groundwater table that changes along with the rainfall regimes.

These two physical processes which were previously accepted have more recently been criticised. The movement of water through capillary action was found inadequate to explain thick occurrences (in excess of 5 metre) of red soils since capillary movement is limited to a maximum of 2 to 2.5 metres, (Mohr and van Baren, 1954, p.371). On the other hand, the mechanism of enrichment by fluctuating water table cannot totally explain movement of iron. By the Ghyben-Herzberg hypothesis (discussed by Sivarajasingham et al. 1962, p.49) fresh water 'floats' on the underlying solutions, thus presenting a barrier to the rise of these solutions in the profile.

It is now generally accepted that no single physical theory is capable of explaining all the known occurrences of accumulation in red soils. Modern trends, particularly among geomorphologists is to invoke theories based on compromise (de Swardt, 1964, Trendall, 1962).

The problem of the genesis of red soils has moved from one of physical process to the arena of physico-chemistry. The geochemistry of the constituents and the physiochemical conditions for the removal of certain constituents and the accumulation of others have and is still being studied.

Many of the suggestions concerning the mechanism of formation and the formed products were mere generalisations. These were made when the studies of clay mineralogy, mineral equilibria and soil chemistry were still in infancy.

For example, it is general knowledge (now) that both granite and diorite can weather to give similar products depending on the condition of weathering. Harrison (1933) in a paper published posthumously was of the opinion that weathering of basic rocks (e.g. diorite) would lead to the formation of hydrated sesquioxides and the weathering of acidic rocks (e.g. granite) would lead to the formation of aluminosilicate. Fairly recent knowledge has been brought to light by the book of Loughnan (1969) on the chemical weathering of silicate minerals.

Though the theories of physical process have not been individually able to provide explanations for some observed behaviour, the examination of the physico-chemical process have not provided a single solution. The chemical weathering of minerals and the formation of new forms, is a complex process dependent on such factors as climate, topography, parent material (Loughnan, 1969, pp.67-74) to mention a few.

It has become clear that the accumulation of sesquioxides leading to the formation of red soils cannot be due to a single process.

Several phenomena are involved, not all operating at the same level. The similarity in appearance of certain types, particularly the indurated ones, has led to the erroneous conclusion that the problem relates to a single level (Maignien, 1966, p.77).

The writer is of the opinion that the accumulation processes analysed in detail by D'Hoore (1954) adequately explains the genesis of most, if not all forms of red soils. The problem of the source of iron that has been emphasised by Goudie (1973, p.146), McFarlane, 1976, pp.93-94) will not be found in the investigation of physical processes, which does not take into account the systematic analysis of how the different soil components came to be formed and their relationship with the rock on which they lie. In the following sections of this sub-chapter, attention is therefore given to the physico-chemical weathering of primary minerals, the conditions necessary for the mobility of the elements, and the nature of the new minerals formed.

2.4.2 Weathering of minerals:

2.4.2.1 Major rock minerals:

Olivine (Magnesium/Iron silicate $(\text{Mg,Fe})_2(\text{SiO}_4)$) is the first mineral to alter, it weathers very rapidly because it contains cracks (Pecrot et al. 1962). Alteration of olivine even starts during crystallisation of magma (deuteric alteration) due to removal of magnesium from the rim of olivine crystals and collapse of crystal lattice into a chlorite-like structure and iron oxide (Wilshire, 1958). Alteration products consist of yellow isotropic materials which are usually found at cracks and edges. These develop into goethite, it could also go through an intermediate montmorillonite stage (Wilshire, 1958).

Pyroxene: Pyroxenes are noted for cleavages which assist in weathering (Loughnan, 1969, p.80). It alters quickly into ferruginous

substances which turn into goethite; it forms a web around patches of (a) isotropic yellow substance with a few gibbsite crystals, or (b) monocrystalline goethite (Maignien 1966, p.78).

Amphibole: These also have cleavages and are more resistant to weathering than olivine or pyroxene. It turns into amorphous ferruginous material which can develop into goethite (Maignien 1966, p.78).

Feldspar: Present wide range of weathering properties. Cleavages are well marked in plagioclase and this allows penetration by water leading to alteration. Anorthite is the least stable and chemical breakdown is very rapid. Potash and soda feldspars offer great resistance to chemical attack and these minerals tend to be present as detrital grains in sedimentary rocks (Goldich 1938).

Plagioclase: Forms an isotropic rust-coloured substance which settles as an isotropic material or penetrates into cleavages and cracks. This substance usually gives rise to gibbsite - which is the alteration mineral with well defined characteristics. Another type of alteration is the formation of an abundant mass of isotropic ferruginous substance within which an amount of gibbsite appears. This mass then gives rise to goethite and to kaolinite with an accordion type facies. At this stage the matrix is kaolinite and the original structure has collapsed (Maignien 1966, p.78).

Alkali feldspars: These can change into crystalline gibbsite which crystallises first in the cleavages and then takes over the entirety of the original crystals leaving numerous spaces. Alternatively, the feldspars appear to turn into kaolinite but without observable direct replacement of crystal form.

Micas: Micas weather rapidly (except for muscovite). Attack is usually along the cleavage planes. They alter without great change in structure into chlorite and other clay minerals.

Muscovite: Resistant but not impossible to weather. Usually changes into kaolinite (Goldich 1938).

Biotite: Alters rapidly. First turns into chlorite which later turns into kaolinite powder and powdery goethite (Goldich 1938, Pecrot et al. 1962).

Quartz: Resistant and not easily corroded only cracked and jagged (Goldich 1938).

2.4.2.2 Others:

Magnetite: Fe_3O_4 . Very resistant, but residual pieces usually have a border of rust-brown iron-oxide. This turns into haematite and goethite (Craig and Loughnan 1964). Transformation of magnetite into maghemite (Fe_2O_3) is a process considered specific to alteration under tropical conditions.

Ilmenite: Oxide of iron and titanium is moderately stable in the weathering environment and may persist as detrital grains in derived sediments (Harrison 1933).

Chromite: Cr_2O_3 . Resistant to weathering but partial alteration is not impossible.

2.4.3 Secondary minerals formed:

2.4.3.1 Oxides and Hydroxides:

Quartz: Quartz is mostly encountered as residual or detrital material in red soils. It can however be produced by weathering of silicates.

Haematite: ($\alpha\text{-Fe}_2\text{O}_3$). Occurs widely in red soil and in particular ferruginous hardpans. Maignien (1966, p.83) describes various paths by which haematite can be produced in nature:

- (a) From the dehydration of goethite by heat and isolation, although this transformation can be produced in the laboratory

at 320°C. Observations show that haematite is present in the top profiles which contain very little of it or none at all in the subadjacent horizons.

- (b) Within profiles, haematite seems to be derived from alteration of magnetite chromite and ilmenite.
- (c) Haematite can be a contamination product.

Another way of producing haematite is from the conversion of maghemite ($\gamma\text{-Fe}_2\text{O}_3$) which is considered to be metastable with respect to it, at high temperatures.

Goethite: ($\alpha\text{-FeOOH}$) Occurs commonly in red soils. Usually found at brown or red powdery earths, films or concretions - clearly defined crystals are rare. It is the easily obtained product of the ageing of amorphous gels commonly observed at all stages of mineral alteration (olivine, pyroxene, amphibole). Electron microscope observation shows that crystallisation takes place mostly in the voids of the soil structure and this is achieved through the formation of an intermediate hummocky material of poorly crystalline goethite (Malomo, 1974).

In most soils and sedimentary rocks a greater part of the iron-oxide present contains indefinite amounts of water and appears amorphous to X-rays. The term 'limonite' has been applied to this poorly organised material (Loughnan, 1969, p.24).

Aluminium oxides and hydroxides: Unlike iron, aluminium is not usually encountered as the oxide corundum ($\alpha\text{-Al}_2\text{O}_3$) in weathering environments. Diaspore ($\gamma\text{-AlO(OH)}$) has been reported in sedimentary fireclays from Missouri and Pennsylvania in the USA, but has not been observed as weathering product. Pressure - temperature equilibrium curves for the system $\text{Al}_2\text{O}_3\text{-H}_2\text{O}$ show that diaspore is only likely to form at high temperatures. Commonly occurring minerals are gibbsite $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ and boehmite $\gamma\text{-AlOOH}$.

Gibbsite: The most common form of alumina hydroxide in soils. Develops from gels of alumina through crystallisation. Formation of gibbsite from this gel of aluminium hydroxides is aided by (a) high rate of removal anions and cations which, if present in the crystallisation centre, gives considerable distortion and prevents crystallisation, (b) high temperature of percolating water, and (c) high rate of silica removal. Under intensive leaching kaolin (kaolinite and halloysite) has been observed to lose silica with the formation of alumina gel which crystallises into gibbsite (Bates, 1962).

Boehmite: (γ -AlOOH) Sometimes associated with gibbsite in hardpans - can be derived from it through the action of pressure and temperature. Boehmite crystallises only after transport since it usually filled cracks and small diaclasses (Maignen, 1966, p.82). It crystallises from alumina gel at about pH 7 and appears to be derived from gels with a more disordered structure, i.e. one where retention of foreign ions is significant.

2.4.3.2 Aluminium silicates:

Kaolinite: The most common clay mineral in tropical soils. The conditions necessary for the formation of kaolinite from (a) precipitated gels: acid medium, moderately silica-rich but strongly lacking in foreign ions and humid; (b) from ions in solution: the presence of a complex that would maintain Al in 6-fold co-ordination in an acid, humid environment (Maignien 1966, p.86). Product of decomposition of organic matter can assist in the production of such complexes. Attainment of the medium is strongly dependent on the degree of leaching, temperature and drainage.

Montmorillonite: Not very common in red soils. Its occurrence is attributed to weathering of basic rocks in a Mg and Ca rich environment

where removal is so slow that incorporation of these ions into the structure is possible. Forms at high pH, and occurs widely in Africa and India as black cotton soils.

Halloysite: Also not a very widespread mineral. Mostly reported as weathering of volcanics and other extrusive igneous rocks. Bates (1960) found that in Hawaiian Islands, under moderate leaching, halloysite is the preferred alumina to crystallise, but under more intensive conditions, gibbsite and amorphous alumina are the main forms.

Anatase: TiO_2 . A yellow or blue mineral found in products of chemical weathering in the tropics. It is regarded as the form of TiO_2 which is resistant to low temperatures and is consequently the most likely titanium mineral to occur ^{as} products of tropical weathering (Eyles 1952). It can be derived from weathering of titanium containing silicates (sphene, biotite, augite) or ilmenite.

2.4.4 Accumulation theories:

The formation of red soils necessitates the accumulation of sesquioxides in a horizon within a profile. According to D'Hoore (1954) absolute accumulation requires:

- a source of moveable sesquioxides found outside the accumulation zone. This can be referred to as 'zone of origin';
- a mechanism of mobilisation and transport found in the 'zone of translation';
- a way of immobilisation found within the 'zone of accumulation' or reception.

Considerations about absolute accumulation necessitates the analysis of the diverse factors operating in the 'zone of origin', of translation, and of accumulation.

Relative accumulation requires:

- a source of moveable non-sesquioxides, occurring in the zone of accumulation. A mechanism and transport of this is consistently active in this zone.

2.4.4.1 Relative accumulation:

Theories of relative accumulation refer to the removal of relatively soluble material from primary rock originally rich in iron or alumina constituents within the profile. Breakdown of primary silicates with the removal of alkalis, alkaline earths and silica in solution leaves a material more concentrated in iron or alumina. Different opinions exist as regards the mobility and enrichment of materials within the profile.

Aluminous: Primary minerals have been found to weather directly in situ to well-crystallised gibbsite. Such crystals form a coherent skeleton and give rise to an indurated crust. The in situ nature of aluminous materials is supported by the fact within the 'normal' pH range of groundwaters, alumina remain insoluble and cannot be mobilised (Loughnan, 1969, p.33). Also is the existence of gibbsite in contact with fresh rocks whose origin cannot be attributed to leaching. So also are the fillings of gibbsite found as pisoliths which are only observed in the upper part of the profile and whose origin cannot be explained by capillary action or cross channel leaching (Maignien, 1966, p.93).

Ferruginous: This involves in situ transformation as well as vertical transportation of materials, which can be upward or downward. Downward transportation is the leaching of materials from upper part of a profile which accumulates at depths. Upward involves the alternation of the phreatic surface with the establishment of three zones: (i) a zone of non-saturation which is a zone of oxidation; (ii) a zone of intermittent saturation where most of the deposition occurs, and (iii) a zone of permanent saturation which is a zone of reduction. Upward

movement is also possible through capillary rise but this contribution is considered to be very limited and only important at depths near the phreatic surface (D'Hoore, 1954).

For vertical transportation, simultaneous presence of agents of mobility and transportation is required. The different forms of iron in soils are not favourable for its displacement: (a) the ferric ion is almost insoluble at the pH of tropical soils; (b) the ferrous ion is appreciably soluble, but stable only in a reducing environment; (c) iron in its colloid form can be displaced, but it is very sensitive to electrolytes; (d) because of its electropositive charge, iron vigorously attaches to clay and can be leached with it; (e) ferric and ferrous ions are able to associate with certain substances passing through soils, giving electronegative complex which do not attach to clay and are less sensitive to electrolytes, these combinations are essential in the processes of iron, and perhaps of aluminium migration; (f) under certain conditions, iron can migrate in the form of carbonates (D'Hoore 1954).

Movement is achieved through complexing, chelating and reduction. Silicic acid forms complexes with ferric and ferrous ions which are pseudosoluble (Demolon and Bastisse, 1944). Organic anions, mainly polyacids and alcohols, combine to give fairly undissociated and highly stable complexes in particular hydroxy-acids which are present in plants and in their decomposition products, e.g. lactic acid (Bastisse, 1946). Reduction of ferric into ferrous iron thereby promoting solubility can be done through dehydrogenation by bacteria, e.g. 'Eicheria coli', 'Bacillus polymixa', etc. (Bromfield, 1954). Formation of crude organic matter can give rise to products capable of reducing iron, e.g. glucose (Mandal, 1960). Humic and fulvic acids can dissolve ferric ion through complexing and reduction. Chelating agents also exist which interact

with these cations to produce soluble complexes (Schatz et al., 1957).

Precipitation and immobilisation of iron can easily occur when the iron complexing structure is destroyed. The destruction can be produced by change in pH, Eh or by oxidation or destruction of the transporting agent. Immobilisation can also be brought about by attachment to clays, e.g. kaolinite with the formation of stable bonds. This fixation however, depends on soil environment. D'Hoore (1954) arrived at a value of 12 per cent Fe_2O_3 for the saturation of clay surfaces for soils from the Congo.

2.4.4.2 Absolute accumulation:

Allochthonous materials have always been found in deposits of red soils and the total in-situ formation have been doubtful. The mobility, transportation and fixation earlier described, could well apply on a horizontal level and lateral movement with later deposition would result in accumulation. This is particularly dependent on the general relief. Red soils found on slopes probably owe their origin to this mode of formation (Hamilton, 1964).

2.4.5 Summary:

The products of the weathering of rocks in the tropics are many. The processes involved are varied and complex. This fact should be borne in mind when using red soils as engineering materials. Similarity of soils in terms of physical appearance will not necessarily lead to similar engineering properties. Care should therefore be exercised when soils from different localities are compared.

CHAPTER 3

PROBLEMS PECULIAR TO RED SOILS

3.1 Introduction

Tests developed for the appreciation and evaluation of soils of the temperate regions do not appear to be entirely applicable to the soils of the tropical regions. Gidigas (1974) has mentioned that the major difficulty in trying to develop a satisfactory identification system for laterite soils appear to revolve around the so-called 'problem' laterite soils, which may not yield reproducible test results using standard laboratory test procedures and whose engineering behaviour may not be accurately predicted on the basis of standard classification tests.

Identification of the differences between the two soil regions is needed. This is not only as an advancement, in the knowledge of the characteristics presented by these materials, but as a platform for the development of other engineering considerations.

3.2 The peculiar problems

The survey on the physical properties and the use of red soils as engineering materials (see Chapter 2), readily shows that two distinct characteristics separate these soils from those found in the temperate regions. These are found only in red soils and are pertinent to the use of the soils. These are:

- (1) Mechanical Instability
- (2) Thermal Instability

The susceptibility to change as a result of the addition of small quantities of thermal or mechanical energy effect the physical parameters employed in the evaluation of the soils. It also interferes with the

methods used for the measurement of these parameters.

Mechanical instability is manifest in changes that occur on remolding and manipulation. This results in the breakdown of cementation and of structure. It affects such engineering parameters as particle size, Atterberg limits, moisture-density distribution, Townsend et al (1969), Matyas (1969), Newill, (1961).

Thermal instability is manifest in sensitivity to drying and what has been described as elements of 'potential self-stabilisation' Gidigasu (1974). This affects such engineering parameters as Atterberg limits and particle size distribution Moh and Mazhar (1969). It therefore affects the strength of the material.

The parameters used for the evaluation of the soils of the temperate regions are controlled by these two additional characteristics, that are peculiar to red soils.

Mechanical instability essentially results from unstable structure and breakdown of cementation. The structure is controlled by the environment of formation, i.e. geology, climate, topography and vegetation as well as the clay/iron relationships due to charges and the chemical environment imposed by organic matter and alumina-ferrous binding agents.

Thermal instability results from instability of the intrinsic elements of the soil. This is mostly due to the instability of the form of the oxide minerals in different states of oxidation and hydration.

The above discussion can be tabulated.

TABLE 3.1

<u>Characteristics</u>	<u>Causes</u>	<u>Manifestation and Practical Effects</u>
Thermal Instability	Chemistry and mineralogy changes	Sensitivity to drying - affects values obtained for Atterberg limits, grain size, specific gravity, moisture-density.
Mechanical	Structure	Sensitivity to manipulation - affects values obtained for liquid limit, grain size, moisture density.

3.3 Causes of Mechanical Instability:

This can be due to dislocation of one particle with respect to another leading to the failure of the bonds or actual removal of the cementating agent.

Uehara et al. (1962) have described four types of soil structure for Hawaiian latosols that correspond to different rainfall regimes and soil-forming conditions. They observed that aggregate stability of kaolinitic soils with more than 5% free iron-oxide is linkable to the crystalline nature of the kaolinite present. Similar conclusions have been reached by Fripiat et al (1954) who distinguished two forms of kaolinite-iron oxide complexes:

- (a) An ordered complex resulting from the stacking of kaolinite plates along the c-axis all cemented by iron-oxide, and
- (b) a disordered one with no regular pattern for its aggregates formed from less crystallised kaolinite.

Complexes of the type (a) become saturated with iron oxide at about 12% and beyond this individual iron oxide forms while complexes of the type (b) do not have any saturation limit for its clay surfaces.

D'Hoore (1954) has also shown that the maximum amount of iron-oxide that can be associated with a kaolinitic clay without the formation

of iron concretions is 12%.

It thus seems likely that soils with well developed kaolinite under high rainfall regimes will be most resistant to applied mechanical effects.

Mechanical action produces degradation of the cemented particles. This would be brought about by disruption of the network of cemented elements at points of contact, in the case of undisturbed material. It can lead to the reduction in sizes of the aggregates. Finer materials will be produced which can be manifest in the engineering properties.

Townsend et al. (1969, p.76) have observed that '...generally, in situ laterite and lateritic soils possess a granular structure due to the abundance of iron and alumina oxides (sesquioxides) that coat the pore walls, fill the voids and bind the soil particles into agglomerated nodules of various sizes. The granular structure is responsible for the desirable engineering properties displayed by unworked laterite and lateritic soils.'

Similar observations have also been made by De Graft Johnson and Bhatia (1970) who mentioned that red soils have a great potential for degrading under mechanical stress and that this behaviour is responsible for much of the unsatisfactory performance of some existing roads where 'lateritic' gravels have been used.

The strength of the bonds will depend on the state of hydration. Initially the cementation is mainly due to amorphous (non-crystalline) material and this is usually weak. With continued dehydration, the amorphous material would crystallise into more definite forms, e.g. goethite and this would give stronger bonds (Townsend et al, 1969).

Two main factors which influence the materials potential for degradation are the degree of decomposition and the iron-oxide content which defines the maturity. '...Consideration of an effective classification of lateritic soils according to their engineering properties

should take account of these two factors' (De Graft-Johnson and Bhatia 1970, p.19).

As the resistance of the material to degradation, reflects the degree of decomposition as well as maturity of the soil, various tests for assessing the mechanical strength and the durability characteristics of the coarse fractions in red soils have been suggested.

Ackroyd (1959) suggested the use of Aggregate Impact Value while Novais-Ferreira and Correia (1965) recommended Los Angeles Abrasion Test. Vallergera et al. (1968) have suggested a classification system based on the Casagrande Chart that gives consideration to the hardness of the gravels.

3.4 Causes of Thermal Instability:

Thermal instability is due to the chemical and mineralogical changes that accompany drying or are promoted by it. Since this is mainly due to changes in temperature, it can be considered as energy precipitated reactions, i.e. addition of energy to the system leads to higher energy state. The soil can be stable or unstable at this new state depending on the initial conditions.

The possible changes in mineralogy that accompany drying can be considered from the standpoint of the major oxides present.

3.4.1 Iron Oxides and Hydroxides:

In the ferrous state iron can obviously be oxidised and immobilised by heating in air. Though ferrous forms of iron are essential for the mobility of iron and therefore formation of 'laterites', they are hardly found in considerable amount in already formed 'laterites' and laterites above ground water level. Therefore contribution of ferrous form of iron oxide to the susceptibility of red soils to thermal instability is very minimal.

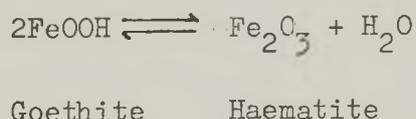
The various forms of hydroxides and hydrated oxides of iron present

in red soils have already been listed p.17). These forms can be changed into anhydrous or less hydrated forms on heating.

All the non-crystalline forms generally referred to as amorphous oxides (includes limonite and ferri-gels) can change into more defined forms on heating. Lepidocrocite or goethite usually results from dehydration depending on the conditions, e.g. pH of the environment, both of which can again lose water to form maghemite or haematite. Change of lepidocrocite via goethite to haematite is also possible (Wefers, 1966).

The possible paths of transformation have been described by Grant (1974) and can be represented for the materials described (based on Oades, 1962), as shown in Fig.3.1.

It is possible for some goethite to be transformed to haematite on oven drying as suggested by the equation:



which is at equilibrium at 70°C and water-vapour saturation. However, most of the changes that occur when red soils are oven-dried cannot be attributed to this transformation. Profound effects have been recorded at relatively low temperature (below 100°C) and slight change in the relative humidity (see Novais-Ferreira and Meireless, 1969). These changes therefore, are more likely to be due to attempts at achieving some form of order from the chaos that exists in an amorphous state. In amorphous material any water attached to the Fe³⁺ ion can be readily lost on increase in temperature since it would not belong to any rigid structure.

3.4.2 Alumina Oxides and Hydroxides:

Unlike iron alumina exist only in one oxidation state but like it, it has more than one hydration state.

It also exists as amorphous hydrated oxide called allophane which usually crystallises into boehmite $AlOOH$ or gibbsite $Al_2O_3 \cdot 3H_2O$. Gibbsite is generally the more favoured form under soil forming conditions.

Both of the crystalline materials can, under specified conditions, give a third hydrate diaspore (Grant, 1974).

The change from one crystalline form to another has not been found to exert much influence on the engineering properties but the transition from amorphous to crystalline material has. Wallace (1973, p.214) has discussed the changes that accompany the drying of soils from the continually wet highlands of Papua, New Guinea. He observed that the 'fabric of microstructure of the volcanic soils (as viewed at x20 mag.) consists of a coarse, open skeleton of rock forming minerals (hornblende, plagioclase and quartz) surrounded by a viscous gel of highly hydrated clay minerals and viscous gel'....'When the present soils are dried, the clay minerals aggregate so that the viscous gel is permanently destroyed'.

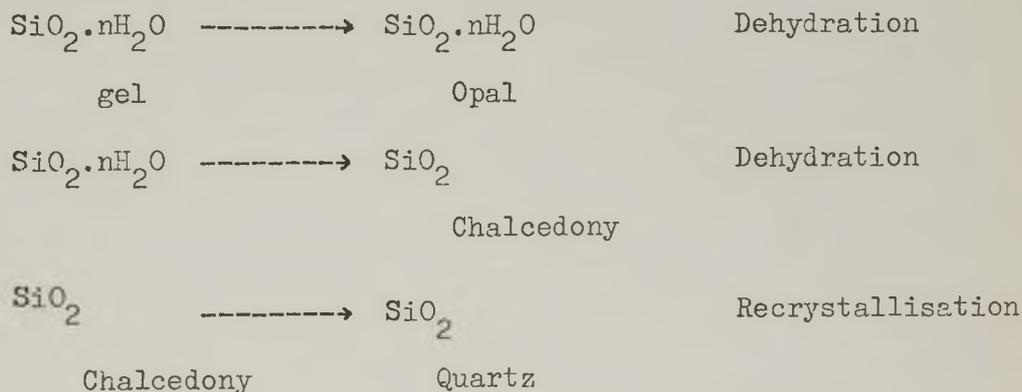
Fieldes (1955) distinguishes between various forms of allophane and indicates that with increasing age the clays contained pass through the sequence allophane B ---- allophane AB ---- allophane A ---- metahalloysite ---- kaolinite. Fieldes (1966) and Fieldes and Furkert (1966) described the structure of allophane and the process by which it forms into hard grains as drying out occurs. According to their description, if no drying out has occurred the allophane consists of gel-like fragments of random alumino-silicate held together by random links at a relatively small number of points. A great deal of water is enclosed in the very open structure. When drying out occurs, the removal of water causes the structure to shrink and the number of linkage points between the

fragments steadily increases leading eventually to the formation of hard grains. The process is irreversible (Wesley, 1973).

3.4.3 Silica Oxides and Hydrated Oxides:

The form of silica present in red soils has been mentioned in Chapter 2.

Changes in drying can be accomplished through the transformation of:



All these involve addition of energy to effect dehydration and recrystallisation. However, only the first



can be effected by the normal increase in temperature within the range being considered. This transformation is not very common. It has not been recorded to bring about considerable changes in the engineering properties of soils when dried.

Another possible factor that can be responsible for change upon the drying of natural soils is the presence of organic matter. This sometimes exists in combination with iron and alumina as complexes and can be easily oxidised. Oxidation would produce changes in the system. It is, however, not found in large amounts in red soils. It therefore cannot be expected to greatly affect the engineering properties of red soils.

3.5 Effects of Mechanical Instability:

The effect of mechanical manipulation and remolding of red soils is to produce structural and cementation breakdown. This generally results in an increase in clay content and exposure of more clay surface (Townsend et al. 1969).

3.5.1 Atterberg limits:

Millard (1962) determined the liquid limit of a red clay which had suffered 15 minutes and then 3 hours of mixing prior to testing. He reported an increase in the value from 49% to 84%. Newill (1961) studied the effect of remolding in the red clays of Kenya for different periods of time. The Sasumua clay (which has been investigated by other authors Terzaghi 1958, Robertsons 1958) and Kabete clay. He found that when Kabete clay was tested with a minimum amount of mixing, values of 56% were obtained for liquid limit and 38% for plastic limit for soils in natural conditions. When mixing was continued for 45 minutes, liquid limit and plastic limit increased to 76% and 39% respectively. After 3 hours liquid limit increased to 84%. Sasumua clay did not present considerable change on mixing.

Sherwood (1967) also reported similar results on the effect of working on a red clay. The liquid ^{limit} was increased by working of the clay. The plastic limit was not influenced significantly by remolding.

The effect of remolding in changing the values of Atterberg limits of red soils can be considered due to the structure of the clay minerals which are in the form of clusters. These are dispersed by vigorous mechanical working and also by the extraction of iron-oxide resulting in increased plasticity (De Graft-Johnson and Bhatia (1970).

3.5.2 Particle Size Distribution:

The effect of manipulation, as already mentioned, is to degrade the cemented particles and result in the production of fines. Such increases have been reported by Newill (1961), Townsend et al. (1969).

3.5.3 Compaction Characteristics:

Evans (1958) used a red soil from Uganda and did not find much change in the moisture density curve in spite of some breakdown of the coarse fraction of the gravel.

Townsend et al. (1969) found that working caused slight decrease in maximum density and little change in optimum moisture of soil from Curundu, Panama Canal zone.

Matyas (1969) determined the effect of the re-use of material on the moisture density characteristics. Re-using the material increased the maximum dry density by about 2lb/cu.ft. whereas the optimum water content did not appear to change.

Studies on the compaction characteristics of red soils from Ghana would indicate that the change in the moisture density relationships is greatly influenced by the initial grading of the material as well as the mechanical strength of the particles and that the resulting density would be increased or reduced during compaction. (De Graft-Johndon and Bhatia, 1969).

3.5.4 California Bearing Ratio (C.B.R.)

For most red soils increasing the compactive effort resulted in an increase of the unsoaked C.B.R. However, for some soils, dependent of the type on clay mineral present, increasing the compactive effort resulted in a reduction of the unsoaked C.B.R. as well as the soaked C.B.R.

L.N.E.C. (1969) reported that over-compaction leading to degradation of aggregates appear to decrease the C.B.R.

De Graft-Johnson et al (1969) have shown that over-compaction can result in great loss of strength and suggested the use of laboratory compaction in distinguishing materials susceptible to mechanical instability from those that are not.

Townsend et al. (1969) have carried out work on the effect of manipulation on the strengths of stabilised red soils. The authors reported that lime increased the unconfined compressive strength of manipulated and non-manipulated soils but values for manipulated soils were found to be significantly higher. The reverse was obtained for materials stabilised with Portland Cement. The unconfined compressive strength of manipulated and non-manipulated were higher for soils stabilised with Portland Cement than with lime. The increase in strength of worked soil probably results from increases in fine particles. In general, fine-textured soils require more cement to achieve a given strength than coarse soils.

With regards to changes observed for lime, they attributed this to be availability of materials for pozzolannic reaction. '...Since most of the free silica has been leached from lateritic soils the silica available for pozzolannic reaction with lime should be found predominantly in clay particles. It appears that pozzolannic action proceeds more slowly in unworked soil than worked soil as there is evidence of only a minor strength gain in the former during the first few weeks of curing.' (Townsend et al, 1969, p.82).

3.6 Effects of Thermal Instability:

3.6.1 Atterberg limits:

As already mentioned, these limits are important parameters used

for the classification of soils as well as the selection and evaluation of soils as an engineering material. They are tests that give an indication of the properties of material in the small range where clay minerals predominate. Addition of thermal energy, i.e. drying, sometimes produces considerable changes in the values obtained for the Atterberg limits.

Terzaghi (1958) obtained values between 58% and 63% for the liquid limit of Sasumua clay, between 37% and 54% for the plastic limit, depending on the amount of drying before the test.

Frost (1967) carried out tests on Mendi soil of Papua, New Guinea and found that oven or air drying reduced the clay content from 36% to 2%.

L.N.E.C. (1969) reported decreases in liquid limit with the drying of soils from Angola. Novais-Ferreira and Meireless (1969) carried out tests on samples previously soaked and natural material. They reported that drying of soils previously soaked causes reduction in liquid limit and for soils that did not undergo previous soaking drying reduces the plasticity up to 80°C but after drying at 105°C the sample became more plastic.

Moh and Mazhar (1969) also obtained a decrease in the liquid limit due to pre-test drying. By oven drying the decrease of liquid limit varied from 14.2% for a soil with high liquid limit to 2.2% for a soil with low limit.

For soils containing the clay mineral halloysite $A1_2Si_4O_8 \cdot 4H_2O$, the decrease in Atterberg limit values have been attributed to the loss of some structural water ($2H_2O$) to give another mineral metahalloysite $A1_2Si_4O_8 \cdot 2H_2O$ which shows different water relationship.

These decreases have also been attributed to decrease in clay size content upon drying (Fruhauf, 1948) with the coating of iron-oxide on

the clay surfaces aggregating the particles into clusters.

Moh and Mazhar (1969) found that measurable changes in clay content were only observed for soils containing enough amount of free iron-oxide to properly coat the particles.

Drying changes the strength of the bond (recorded as low extractable iron oven dried samples) and the clay fraction is partially or completely converted to a non-reactive silt-like fraction with reduction in plasticity. The change of the plastic limit was smaller than those obtained for the liquid limit.

3.6.2 Particle Size Distribution:

The wide range of occurrence of red soils render their particle size to cover wide limits. It extends from material consisting of very coarse > 1 " (as in gravels) to those that are mainly fines (clay-size range).

Terzaghi (1958) recorded a decrease in the clay content of Sasumua clay, Kenya after oven drying. He attributed this to the coating of iron-oxide coagulating the particles into larger clusters during oven drying.

Newill (1961) found that oven drying of Sasumua clay decreases the clay size from 79% - 47% while of another red clay, Kebete, was reduced from 82% to 68%. He confirmed Terzaghi's suggestion for he reported that free iron-oxide was more easily removed from natural Sasumua soil than from oven-dried soil and it was noted that the aggregations of clay particles were more readily dispersed when the soil was in its natural condition. If the difficulty in removing free iron-oxide is associated with the aggregations of clay particles then the greatest difficulty would be expected with the oven-dried Sasumua soil.

Moh and Mahzar (1969) also determined the particle sizes before

and after oven drying of red soils from Thailand and recorded decreases in the clay size content of some while no change was recorded for others. They interpreted the changes as a result of oven drying along lines similar to those of Terzaghi and explained the non-response of some of the samples to oven-drying by the quantity of the iron-oxide present. Soils that do not show changes after oven-drying contain only sufficient iron-oxide to coagulate the soil particles weakly. They concluded that 'the change in clay content due to oven-drying in soils containing insufficient amount of free iron-oxide for coating the soil particles was only temporary and could not be detected by usual method of analysis.

Tateishi (1967) suggested the use of an aggregation index to quantify the susceptibility to oven-drying using particle size as a basis. This index is expressed as a ratio of the sand equivalent value of the oven dried sample to that of the sample in its natural state. An aggregation index of 1 indicates a non-sensitive material whereas higher indices in the range of 2-10 would require modification of the test procedures to eliminate the influence of drying.

3.6.3 Moisture-density Characteristics:

The effects of thermal instability have also been carried out with reference to compaction characteristics. Newill (1961) obtained differences in the compaction characteristics before and after oven drying. He stated that this effect for Sasumua clay for oven drying leads to the transformation of halloysite into metaalloysite and if interlayer water was taken into account the compaction curve obtained did not show any effects.

Frost (1967) recorded an increase in the maximum dry density and a decrease in the optimum moisture content in the drying of Mendi soil

of Papua, New Guinea.

Tateishi (1967), found that drying of Hawaiian soils produces irreversible change to a soil with granular characteristics. Brand and Hongsnoi (1969), also obtained similar results to those of the investigations already mentioned and found that for any given moisture content the dry-density was higher for oven-dried condition for some of the soils and in certain cases, although the maximum dry densities were almost unaffected by drying the optimum moisture contents were considerably changed. They also reported a case where the maximum dry density after oven drying was higher for the dry-side compaction and lower for the wet-side compaction than for the samples prepared by the addition of water to the soil at natural moisture content or the removal from it by drying.

3.6.4. Strength:

The effect of thermal instability on conventional strength measurements, i.e. direct shear and triaxial testing have not been widely investigated and are scant in literature. However, those based on indirect determination, e.g. C.B.R. are readily available.

Brand and Hongsnoi (1960) tested soils from various regions of Thailand to determine the effect of the air and oven drying on the measured C.B.R. of soaked and unsoaked samples. They mentioned that no definite conclusions could be arrived at with regards to strength changes brought about by drying. In some soils the unsoaked C.B.R. values of oven-dried soils were lower than the natural moisture conditions. However, after soaking there was a tendency for the oven-dried samples to give higher C.B.R. values. There were other samples which yielded higher soaked and unsoaked C.B.R.s in the oven-dried condition.

Brand and Hongsnoi concluded that the highest soaked strength of

most of the soils after drying could be related to the swelling characteristics of the soils in the different conditions. The compacted oven dry soil tended to absorb less water and to swell less than those compacted in the natural moisture content condition for equal period of soaking.

L.N.E.C. (1969) reported tests of red soils involving wetting and drying cycles at different temperatures. The increase in C.B.R. which took place as a result of drying in the oven occurred after the first cycle, after which continuation of wetting and drying cycles did not seem to influence the results greatly.

Similar results are reported by Novais-Ferreira and Meireles (1969) who also recorded that the effect of continuous wetting of red soils for periods varying from 4 to 104 days - the C.B.R. values were not altered much by prolonged wetting.

Oven drying generally means drying at 100°C . It has been felt necessary to reduce the drying temperature of red soil susceptible to changes to values somewhat lower. However, Novais-Ferreira and Meireles found that oven-drying at 50°C can produce an increase in the C.B.R. The values of C.B.R. obtained after drying at 50°C were higher than those found after drying at 80°C for some soils.

3.6.5 Swelling and Water Absorption Characteristics:

Brand and Hongsnoi (1969) reported swell values for compacted red soils of Thailand and found that samples previously oven-dried swelled less than samples compacted at their natural moisture content.

Petrification Index - The concept of petrification introduced by L.N.E.C. is a measure of the effect change due to oven drying using the normal channel of measuring the shrinkage limit. Nascimento et al. (1954) define Petrification Index as an attempt to measure the amount

of water resistance that the bonds in a soil have - '...As these water resisting bonds are typical of petrous materials, they could be called "petrous bonds" and the corresponding agglomerating action - "petrification".' (Nascimento et al 1965, p.80).

Define Petrification Index (or degree) as $\frac{Ws}{AL}$

where Ws = Shrinkage limit and AL = Absorption limit. AL is determined by allowing a pat of soil used in the shrinkage limit test to absorb moisture from a porous stone.

For undisturbed samples

Petrification Index = $\frac{Wo}{Wa}$

where Wo = moisture content theoretically corresponding to the undisturbed soil assumed saturated without volume change and Wa = final moisture content of a part cut from the ground and subjected to the absorption test.

3.7 Implications to Classification:

In incorporating red soils within the already established classification system, i.e. Casagrande's, considerations of other parameters have been looked upon. Gidigasu (1971) has evaluated the inadequacies of the present system and has suggested the use of soil forming factors as a basis for classification. Lohnes and Demirel (1973) agreed with him in the use of degree of weathering as a basis and they attempted a parallel between this degree and specific gravity. Vallergera et al. (1969) however, maintains that the Casagrande system is adequate as long as consideration is given to the durability characteristics. They have produced a classification system that incorporates the durability.

As a further step in this argument, two of the specific parameters that should be borne in mind when one is considering grouping of tropical soils is the response to thermal and mechanical effects. The soils that

give considerable response have been referred to by Gidigasu (1974) as 'problem laterite soils' who suggested a number of parameters for their identification.

The immediate division of red soils into problem and non-problem ones give an arm to the already grave polemic of definition since two soils existing within the same classification group in the established system can be on either side of the fence, i.e. problematic or non-problematic side. Obviously there is great need for some criteria for identification and separation but whether this criteria should be part of classification is another matter.

A general expansion of the established test criteria of Atterberg limits and particle size distributions is needed. This should be expanded to incorporate the effects of addition of thermal or mechanical energy.

Such steps can be achieved by introducing a lower level of classification depending on these effects. This will make it easy to pinpoint the response. The user, will have to decide whether the response can be allowed or not. The degradation of material (due to mechanical manipulation) to be used as a central core in an earth dam will reduce permeability and therefore cannot be considered deleterious.

The lower level of identification is just to identify the responses and evaluation is left to the discretion of the engineer. It is therefore desirable to bring the knowledge forward so that decisions can be made in the light of it.

CHAPTER 4COATING AND AGGREGATION OF SOIL PARTICLES4.1 General Background:

The presence of large amounts of iron oxides in the large size fraction of soils has been attributed to the cementing action of iron oxides or the formation of iron concretions (Oades, 1962).

Lutz (1937) found a high positive correlation between the free iron content and the degree of aggregation of silt and clay. McIntyre (1956) measured the macroporosity as used in agriculture (i.e. air space at 100 cm water tension) of a number of soils, that varied widely in free iron-oxide and organic matter. He obtained a significant correlation between macroporosity and free iron oxide in spite of large variation in organic matter. He suggested that the influence of structure of these soils is in the iron acting through the medium of an iron-organic matter complex. He concluded that iron exerts a strong aggregating influence under the soil conditions and postulated that the roles played by free iron-oxide in bringing about aggregation include:

- (a) Cementation due to precipitation of hydrated iron gel and its irreversible dehydration.
- (b) Iron in solution preventing flocculation.
- (c) The formation of organic mineral compounds of humic acids with free sesquioxides.

Fillipovich (1956) found that acid soils rich in hydrated Fe_2O_3 and some organic matter have a stable structure both in the natural state and after washing with NaCl and HCl. From observations of other soils with only organic matter, he concluded that the accumulation of humus

does not improve the structure unless it is associated with colloidal hydrated Fe_2O_3 .

The cementing properties of iron oxide in soils have been observed for some time. Truog et al (1937) described a method of removal of iron-oxide during mechanical analysis to allow for the determination of the actual clay content. The extent of cementation that can be brought about by iron oxide on clay has been shown by Dion (1944) who increased the clay content of Cato soil (Puerto Rico) from 6 - 35% using Jeffries (1944) method. Although the action of iron oxides in this case is that of a cementing agent, the concentration will lead to increasing larger size fractions (Oades, 1962).

Hallsworth and Ahmad (1957) have described the condition of formation of an iron layer found in the zone of alternating wetting and drying, i.e. between winter and summer water tables. Sherman and Kanehiro (1956) studied the ferruginous concretions of Hawaiian latosols and found that they only form in soils with alternate wet and dry periods. They suggest that the iron is deposited as an iron-oxide monohydrate with or without adsorbed water, and on exposure is dehydrated to haematite and probably maghemite as the presence of less than 5% ferrous iron precludes the existence of magnetite and yet some of the concretions were magnetic.

4.2 Mechanism of Coating:

4.2.1 Role of Iron Oxide:

Iron oxides in tropical soils have the capability of existing in a wide range of stable and metastable states. The existence of a number of states has been described by Valetton (1972, p.47). Possible states are monomeric and polymeric. These include Fe^{2+} , $(\text{Fe OH})^+$, $(\text{Fe O}_2\text{H})^-$, Fe^{3+} , $(\text{Fe OH})^{2+}$, $(\text{Fe (OH)}_2)^+$, $(\text{Fe}_2 (\text{OH})_2)^{4+}$ $(\text{Fe}_4 (\text{OH})_4)^-$. There may be precipitation of complex ferriferro hydroxides such as $\text{Fe}_3(\text{OH})_8$ and

$\text{Fe}_4(\text{OH})_{10}$ if Fe^{2+} and Fe^{3+} are in solution. In principle Fe^{3+} may crystallise:

- (a) directly from solution by oxidation of Fe^{2+} , hydrolysis of Fe^{2+} compounds or oxidation of Fe^{3+} organic complexes;
- (b) from amorphous phases.

The factors that determine which of the forms will crystallise include pH, Eh, concentration of solution, the presence of other ions in solution, temperature and ageing environment of the gels.

As is evident from the forms of irons described by Valetton (1972, p.47), most of the forms of iron in solution carry positive charges which are easily transferable to a colloidal system. Crystalline iron minerals are also known to carry charges. Goethite, for example, is amphoteric, depending on the pH. Surface adsorption properties have been shown to exist for it, Parfitt and Atkinson (1976) and for haematite Breuwsma and Lyklema (1972). They can therefore easily adsorb small positive charge-carrying ions even when they are negative.

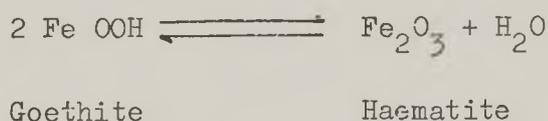
On the other hand, a predominant negative charge is known to exist on clay minerals. This charge results from a variety of reasons which include isomorphous substitution in the clay mineral lattice, broken bonds at the edges etc. (Grim, 1968, p.193). If both clay and iron-oxides are present in a soil, the opposing electromagnetic charges will interact and a coating of iron-oxide over clay will be produced.

Follet (1965) observed that a coating produced by such an interaction on kaolinite is only on one side and concluded that this side alone possesses enough negative charges for interaction. He postulated that this side is the silica tetrahedra side of the kaolinite, that contains silica and gibbsite layers in its crystal structure and that the isomorphous substitution in kaolinite is more significant in the silica layer.

The bond or coating produced by the interaction can be strengthened

as a result of changes that the coating can undergo and the interaction between the surface and other coated surfaces. Ageing and crystallisation will produce more crystalline forms, from the initial coating which in most cases is initially colloidal (Hamilton, 1964). This tends to produce a stronger cementing action as the crystallisation progresses. The crystalline iron minerals that can be produced are goethite, haematite, lepidocrocite, maghemite and magnetite. All these have been found in soils, the concentration of which depends on the condition and the extent of ageing of the original colloidal material. The last three forms are however less commonly found.

The two most common forms, goethite and haematite are commonly found in co-existence with one another and one can easily change into the other. The equation for the transformation



exists in equilibrium at 70°C and water vapour saturation. In general, ageing of a gel under alkaline conditions favours the formation of goethite while under dehydrating conditions will lead to the formation of haematite (see Fi.3.1).

The propagation of cementation into larger sizes and therefore formation of aggregations would not require interaction between clay and iron oxide but between iron coated clay surfaces. The mechanism of continuity of cementation into large sizes is an area which hitherto, has not been given a considerable attention and is still not clear.

It is possible that the intergrowing and the interlacing role of iron oxide encounters in sedimentary rocks, e.g. sandstone, takes over and this is responsible for the propagation. D'Hoore and Croegart (1954) have suggested that oxide coated clays are bound by alumino-ferruginous

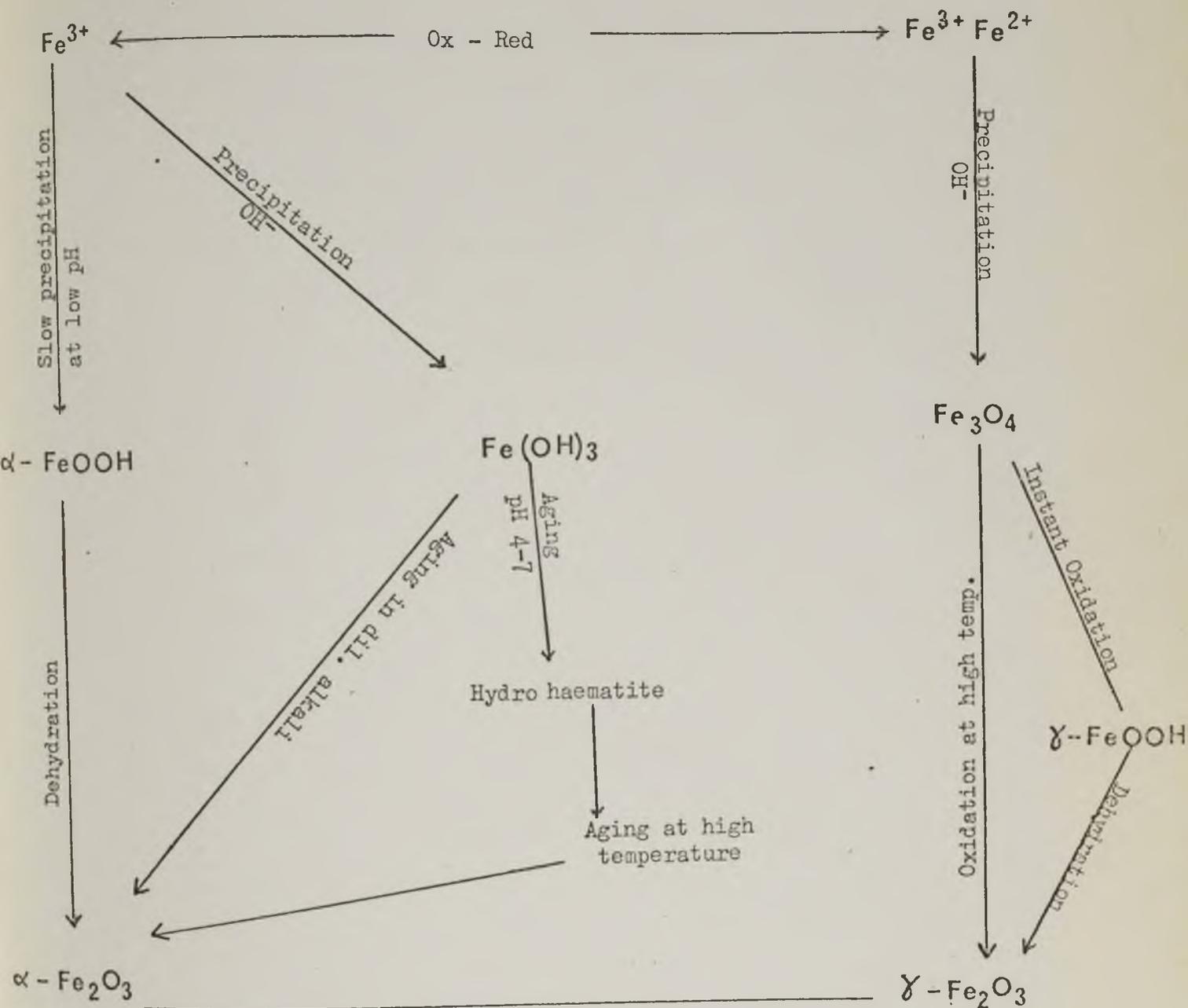


Fig. 3.1 Paths of transformation of various forms of iron oxides (based on Oades, 1962).

(ferri argillan) binding agents to form stable aggregates of silts which are also cemented to form larger size particles.

A mechanism for the formation of aggregation in soils is proposed in Chapter 8. This is based on the results and observations of some soil properties investigated in this research.

Maignien (1958) was of the opinion that the role of sesquioxides (mainly of iron) in the induration of soils can be separated into three, all being related to the introduction of iron oxide from an outside source - absolute accumulation:

- (a) They cement the textural particles.
- (b) They impregnate the formations in-situ.
- (c) They concentrate and form concretions.

He considers that the cementations of textural particles occurs most frequently in coarse elements, and is rarely found when the mean diameter of the soil particles is less than 20 μm . It is found in fine grained soils (clays) only when the fines have undergone some form of cementation or concretion formation.

Impregnation, which is the filling of voids in-situ, occurs in fine grained materials, e.g. silt, clayey sand, sandy clay. This process is generally diffuse and takes place along the lines of least resistance to the circulation of water. Certain regions become indurated and this generally consists of particles that are usually coarser than the surrounding finer materials.

The formation of concretions results from the cementation, precipitation or deposition of oxide films around various nuclei, quartz grain or mineral debris. The concretions are a product of cementation and impregnation, but since the phenomenon is highly concentrated, it results in fairly rounded products which are segregated from each other. Formation of concretions may also be due to the induration of sesquioxides,

segregated from clayey materials.

4.2.2 Possible role of other oxides and properties:

The stability of initial aggregation will obviously determine whether a concretionary profile will form or not. Though iron oxide has to be present for any formation of concretions or nodules to occur (Fripiat et al. 1954), it appears that a considerable amount is not required and that no linear relation exists between iron oxide content and aggregate stability or the strength of the material for that matter, (Uehara et al. 1962). Also is the fact that the simple picture given above of interaction between clay and iron-oxide to produce iron-coated clay surfaces have not been found to agree with field and experimental observation in certain cases (Greenland et al. 1968).

Alexander and Cady (1962) observed that despite the similarity in chemical and mineralogical composition including iron content of some soils of West Africa, some hardened while others did not. They concluded that other properties were involved and wrote that "...hardness or the ability to harden, is as much a matter of arrangement of components as of kind of components."

Uehara et al. (1962) found that despite the evidence supporting the idea that free iron oxide enhanced aggregate stability (e.g. McIntyre, 1956), correlation between the aggregate stability and amount of free iron oxide extends to 5% of the free iron-oxide. The aggregate stability of soils with more than 5% free iron-oxide was found to be related to soil anisotropy and they suggested that the kaolinitic plates in soils are oriented and cemented together through hydrogen bonding. Tropical soils containing amorphous or non platy clay usually have poorly developed structure (Baver, 1972).

Some authors are of the opinion that iron-oxide in soils exists as discrete crystals and do not produce any bonding, at least not on the molecular level. Deshpande et al. (1964) have mentioned that "... the presence of significant amounts of iron-oxide in soils is generally considered to exert important effects on their physical properties, but no satisfactory experimental evidence exists to substantiate this view." From analysis of permeability, wet sieving, and other soil physical properties, these authors concluded that iron-oxide in the soils examined by them are present as discrete crystals and do not cement soil particles. They suggest that the belief that iron oxides are important in the aggregation of soils have arisen because:

- (1) methods for the removal of iron oxide are not specific as they do remove appreciable amounts of aluminium oxides and suitable controls have not been used to distinguish the effects of the different materials removed.
- (2) The large amounts of iron oxides in many 'lateritic soils', suggest obvious correlation with their physical properties.

Further arguments along this line have been brought forward by Deshpande et al. (1968), Greenland et al. (1968), Greenland (1975).

Grant (1974) boiled different concretions in Na OH, in which aluminium oxide is soluble and iron oxide is insoluble. He found that most of the concretions disintegrated and concluded that the continuous bonding in some of these soils is due to aluminium oxide. Observation of the chemistry and mineralogy of the nodules in the present investigation seems to indicate that some other element in addition to iron, is sometimes involved in binding (see Chapter 8).

4.3 Concluding remarks:

As a conclusion to this Chapter, it can be said that aggregation

and hardening in tropical soils is a natural phenomenon which occurs in the soil concomitant with soil formation and is promoted by the weathering conditions as well as the presence of certain oxides. This helps to give good physical properties to them when removed from the point of natural occurrence and translocated to other places for use as a construction material. A change from the physical property possessed in-situ can be brought about by:

- (1) Thermal effects which generally enhance most physical properties.
- (2) Mechanical effects (e.g. working) which are usually deleterious to most physical properties.

Obviously, for small quantities or low values of these effects an 'elastic' behaviour is produced, i.e. no permanent change is brought about by the addition of these effects whether thermal or mechanical, but for higher values a permanent change can be effected.

The inherent nature of the soil which is governed by a number of soil forming parameters such as clay minerals, distribution of elements, climate, vegetation, stage of oxidation or dehydration of the oxides, structure etc., thus comes into play to determine the extent of the response of the soil to the mentioned effects. The investigation of the nature, as an approach to the explanation of engineering behaviour is carried out in this thesis.

The response in the case of thermal effects has been correlated with a parameter referred to as the degree of laterisation (Gidigas, 1971), which is essentially qualitative. In general, for a low degree of accumulation of sesquioxides the response will be high and the change brought about by drying is semi-permanent; for a high

degree of accumulation, little change is effected as a result of drying. In the case of mechanical effects, the response can be linked to the extent of arrangement of the solid particles and voids (structure) which is not actually quantifiable.

CHAPTER 5

GEOLOGY AND SETTING

5.1 Location, Physiography and Drainage:

The localities from which the samples for the study were taken can be found in Paraiba State. This is one of the smaller states in the large North-East region of Brazil. The state has an area of 56,372 km² and is bounded to the north, west and south by the states of Rio Grande do Norte, Ceara and Pernambuco. It is bounded to the east by the Atlantic ocean (see Figure 5.1).

The Brazilian National Geographic Council has divided the state into nine physiographic regions:

Litoral e Mata)	
Agreste e Caatinga Litoranea)	Coastal
Serido)	
Brejo)	
East Borborema)	Central
Central Borborema)	Hinterland
Sertao Alto)	
Sertao do Piranhas)	Interior
Sertao do Oeste)	

The elevation is low at the coast, this increases gently inland up to a fairly large plateau. The plateau changes to a low depression far hinterland towards the western frontier, immediately adjacent to which is a mountainous range in its southern border. The following geomorphological units have been distinguished (Sudene, 1972):



N.E. Brazil

Paraiba State

Fig. 5.1 Map of Brazil showing the location of the North-east and of Paraiba State.

1. Coastal lowlands and low coastal plateau.
2. Subcoastal lowlands.
3. Borborema plateau.
4. Sertaneja depression.

Numbers 1 and 2 are low areas on and near the coast that increase in height very gradually inland reaching a maximum of about 200 m.

The Borborema plateau covers the central and the northern portion of the state and is punctuated in many areas by inselbergs and residual hills. The top of many of the residual hills is flat and this gives the 'plateau within a plateau' impression. The general height of the tabular areas is 200 m but this can increase up to 600 m where the residual hills persist.

The Sertaneja depression occurs far inland towards the western frontier. It is a low, flat, undulating pediplane surface with very little inselbergs. It is the site of old sedimentary deposition, part of which can be found near the west of the town Teixeira, immediately adjacent to the depression. The depression is at an elevation of 250 m which increases rapidly to 770 m within 14 km towards the mountain range.

The area belongs to the dry, semi-desert of the North-East and is consequently fairly well-drained. Most of the rivers have temporary regimes with a few perennial ones near the coast, all flowing in an east-west direction towards the coast. The important ones are River Piranhas, River Paraiba, River Curtiba, River Mamanguape. They all have intermittent regimes inland near their source, that become perennial towards the coast.

5.2 Geology:

The geology of Paraiba is dominated by the Basement Complex. An undifferentiated jumble of Cambrian or older granites, quartzite, marbles and schists. The complex covers all the area of the state of Paraiba,

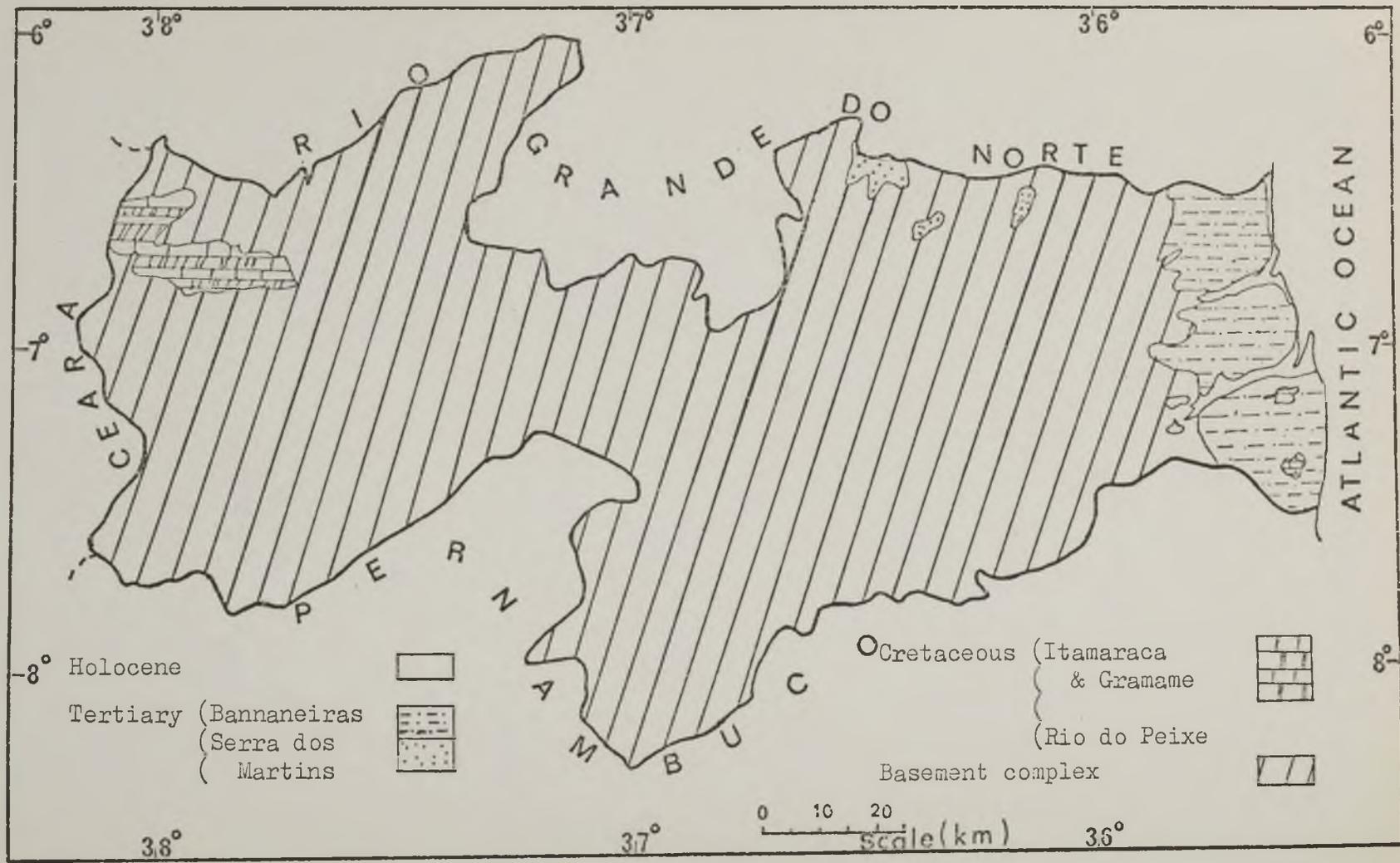


Fig. 5.2 Geological map of Paraiba State

except a coastal belt about 40 km in width trending north-south and an infolded cretaceous area deep in the hinterland towards the western frontier in the Sertaneja depression. Isolated patches of tertiary deposits, which are mainly erosion remnants, can be found in a few places around Cuite and Bobacadinho on the Borborema plateau near the border with Rio Grande do Norte (see Figure 5.2). Modern studies have resulted in the partial separation of the basement complex into:

Pre-Cambrian (B) - Schists, Phyllites, Quartzites with muscovites.

Pre-Cambrian (CD) - Gneisses and Migmatites.

Acidic Plutons - Granites.

The cretaceous and tertiary belt along the coast is usually limited to 50 km in width and those found within the hinterland are mostly extensions of a fairly large sedimentary deposit. The geological sequence within the state can be summarised as follows:

Holocene - alluvials, dunes and mangrove swamp

Tertiary - Sierra dos Martins series

- Barreiras Group (includes Bananeiras formation)

Cretaceous - Paraiba group - includes Itamaraca and Gramame formation

- Rio do Peixe series

Pre Cambrian B - complex

Pre Cambrian CD - complex

Acidic Plutons - complex

Oldest sedimentary rocks are of the Rio do Peixe series which is found near the western frontier of the state near Souza. It extends outward from Paraiba beyond the frontier into a large section of the adjoining state, Ceara. They are found as remaining patches of a fairly large sedimentary area a few metres in thickness. Thickness

at the type locality in Ceara is about 260 m. The sedimentary sequence is initiated by conglomerates followed by sand and silts with clay intercalations. General colour is dark red. Environment of deposition was predominately continental.

The Paraiba group includes Itamaraca and Gramame formations and extend beyond the state into adjoining Pernambuco. The two are contemporaneous at least in parts. Thickness of Itamaraca is about 330 m and of Gramame about 40 m. Itamaraca is a calcareous sandstone and was deposited in partly filled waters near the coast, while Gramame, a fairly argillaceous limestone was deposited in calm waters further inland. Both represent the coastal deposits and are equivalents of the Rio do Peixe in their lower parts.

A number of basaltic and diabasic intrusions can be found cutting across the lower cretaceous sedimentary deposits. These have been dated as post Maestrichtian (late cretaceous to early tertiary (Kegel, 1957)). The deposition of the Paraiba Group can be placed between Campanian to Maestrichtian.

The Serra dos Martin series of the tertiary deposits occurs as non-fossiliferous sandstone found in a few spots on residual hills in Rio Grande do Norte and to a lesser extent in Paraiba. They are generally found as caps on top of these hills. According to Moraes (1924), they are the remains of an old sedimentary ^{deposit} that continued with the coastal Barreiras Group. A projection of the basal plane of the sandstone passes over the cretaceous. The sandstone is loose, 'unconsolidated' and clayey in parts with the lower facies kaolinised.

The Barreiras Group is a fairly well documented extensive sedimentary body. It is found all over the north and north-east of Brazil. The type locality is in the Amazon basin where they are found as non-fossiliferous medium grained sandstones, with a few clayey bands. In a number

of places in the coastal belt of Paraiba, it is encountered as kaolinised clayey sandstone rich in iron oxide.

5.3 Materials for the Study:

The soil samples for the study were taken from three known sites of concretionary red soils, all occurring within the state of Paraiba. The localities are found near the towns of Sape, Cuite and Nova Floresta. The soil samples are hereafter given the name of the town.

The samples were collected from the B horizon of the profiles, in polythene bags, capable of holding 20 kilograms of material and about 10 bags were collected for each site. Different depths were sampled within the profile, in an attempt to cover all the levels represented within the profile.

Figure 5.3 shows the samples sites within the state of Paraiba and by comparison with Figure 5.2, the basic geology of the areas can be discerned.

5.3.1 Sape:

The locality occurs in the coastal plains of Agreste/Caatinga - one of the local physiographic divisions. The plains are on top of the tertiary sedimentary rocks of the Barreiras group which has been described.

The region is reasonably flat with a general altitude of about 125 metres. The sample was taken from a vertical cut 30 metres from the right side of the road on the Sape-Guarabira stretch, 16 kilometres from Sape (see Figure 5.3). The base coarse of the stretch, an asphalt pavement, is made with materials won from the cut.

The profile occurs at the break of slope of the valley of a rejuvenated stream. A schematic diagram of the soil profile at the cut is shown in Figure 5.4. This consists of a dark grey humus horizon,

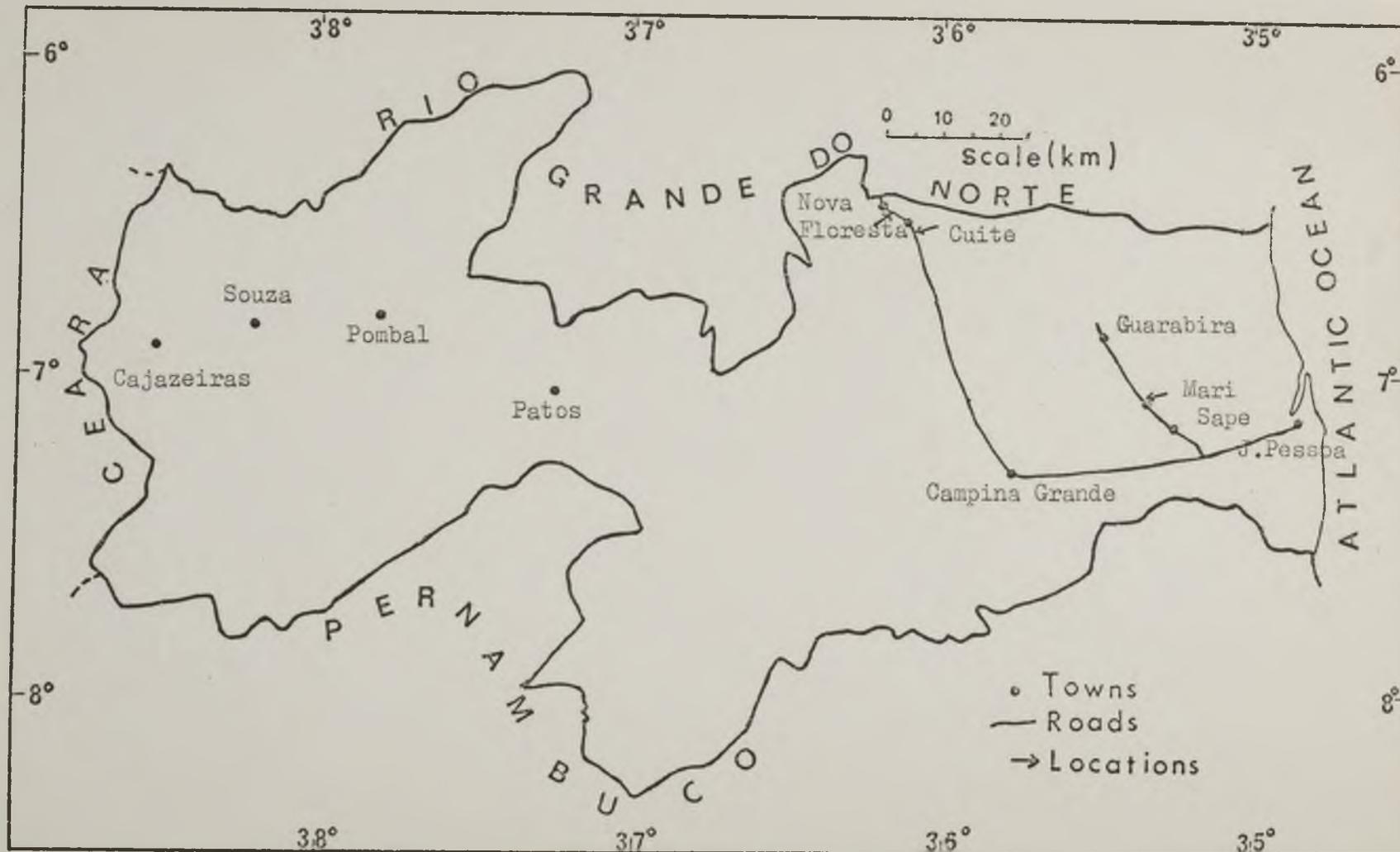


Fig. 5.3 Map of Paraíba showing the sample locations.

Thickness

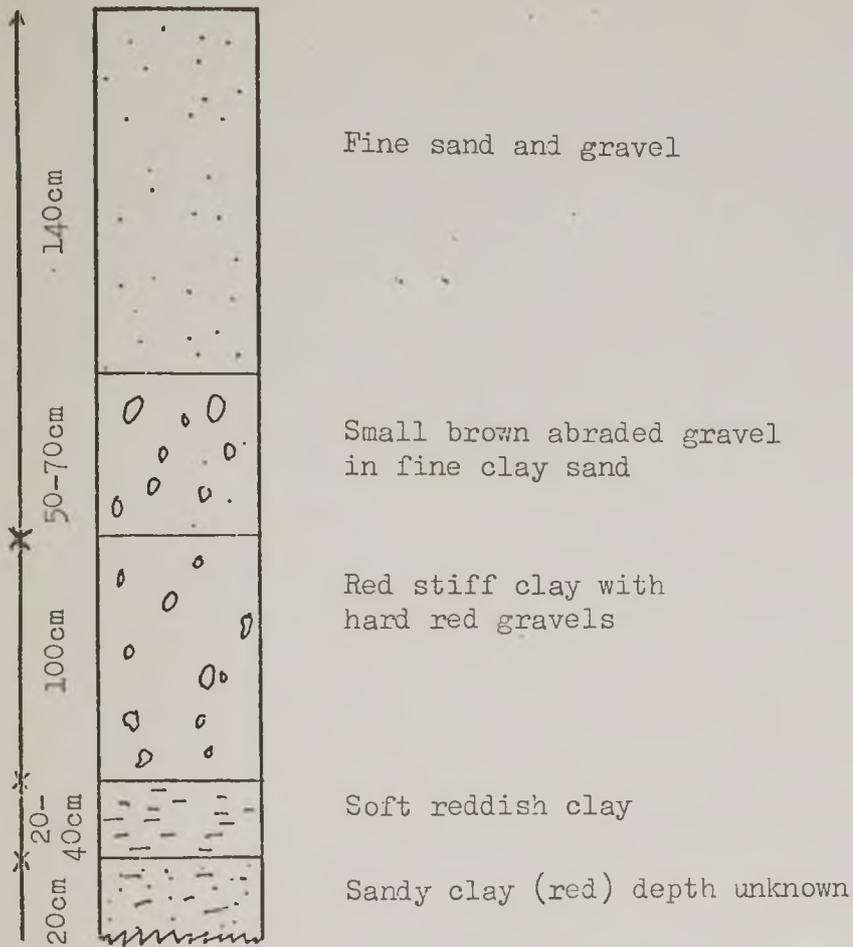


Fig. 5.4 Schematic diagram of Sape profile at the locality where samples were taken for analyses.

forming the A horizon. This is fairly thick, reaching up to 2 metres in places.

The red concretionary soil is found within the B horizon. Three layers can be distinguished on the basis of colour, shape and sizes of its constituents.

- (i) An upper layer of red-brown to purple-brown, small, hard, irregular (shape) concretions mixed with light brown rounded (abraded) gravels or previously hardened concretion. They are surrounded by a matrix of red brown silty clay.
- (ii) An intermediate layer of similar red brown concretions, lacking in light brown abraded gravels. The concretions are of irregular shape as above, but considerably larger. The concretions are set within a matrix of red, stiff, sandy clay.
- (iii) The lowest exposed layer consisting of smaller, softer and fewer concretions than the intermediate layer. The matrix of red, stiff, sandy clay mentioned above is softer.

Soil samples for the investigation were taken from the three separate layers.

5.3.2 Cuite and Nova Floresta:

Cuite and Nova Floresta can be found 7 km of each other in the local physiographic zone of Borborema East, on the Borborema Plateau. They occur on the highest of the many hills punctuating the undulating surface of Borborema Plateau. The hill has an altitude of 700 metres which is the highest in Paraiba except for the mountainous regions near the western border.

The occurrence at Cuite is a cut, 10 metres from the road site, on the Barra de Santa Rosa-Cuite road 2 kilometres from Cuite. The occurrence at Nova Floresta is a road cut, 1 km from Nova Floresta on the Cuite-Nova Floresta Road. This cut was made during the recent

reconstruction of the road. The two occurrences are grouped together because they are considered to be essentially the same.

They occur within 7 km of each other and little change, if any, would be expected at such distances, save for the inherent heterogeneity of the soil itself. There is also similarity in the general expression of the profiles (colour, layer, shape and size constituents) and the occurrence of pieces of concretionary gravels in the top soil layer found between the two localities. Similar profiles probably exist beneath these pieces and suggest some form of continuity between the two localities.

The profiles also occur on tertiary (Eocene) sedimentary rocks, the upper part of which corresponds to the Serra dos Mertins series (Moraes, 1924). The series, which has been described consist of a basal sequence of kaolin, a product of chemical weathering of the crystalline basement. The clayey kaolin passes into a more sandy sequence towards the top. The sandy clay forms the top of the hill.

Schematic diagrams of the profiles are shown in Figures 5.5 and 5.6.

Cuite (a) - This is the right side of the cut, where materials for road construction are being won. It has a yellowish ochre B horizon consisting of cobbles and gravels, cemented by a matrix of softer material. The gravels extend right up to the surface into the A horizon. It can be subdivided into a brownish grey, top humus layer with plant roots - 8 to 20 cm in thickness. This is followed by 30 cm - 80 cm of brownish gravelly material. The lowest exposed part of the soil profile consists of reddish brown concretions in red sandy clay. About 80 cm of this is exposed.

Cuite (b) - This is on the left side of cut and is similar to (a) in many respects. It has 30 cm of brownish grey humus layer, with concretions, which is followed by 1 metre of gravels and cobbles with

Thickness

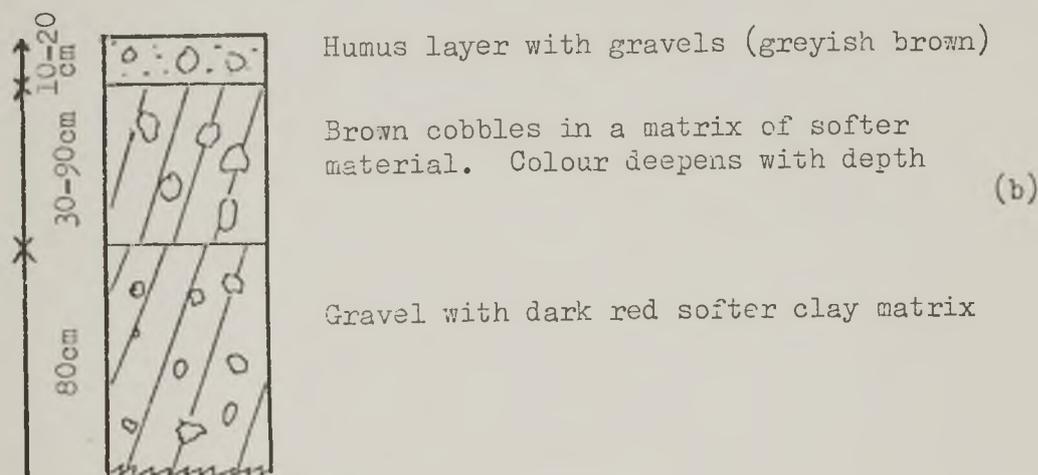
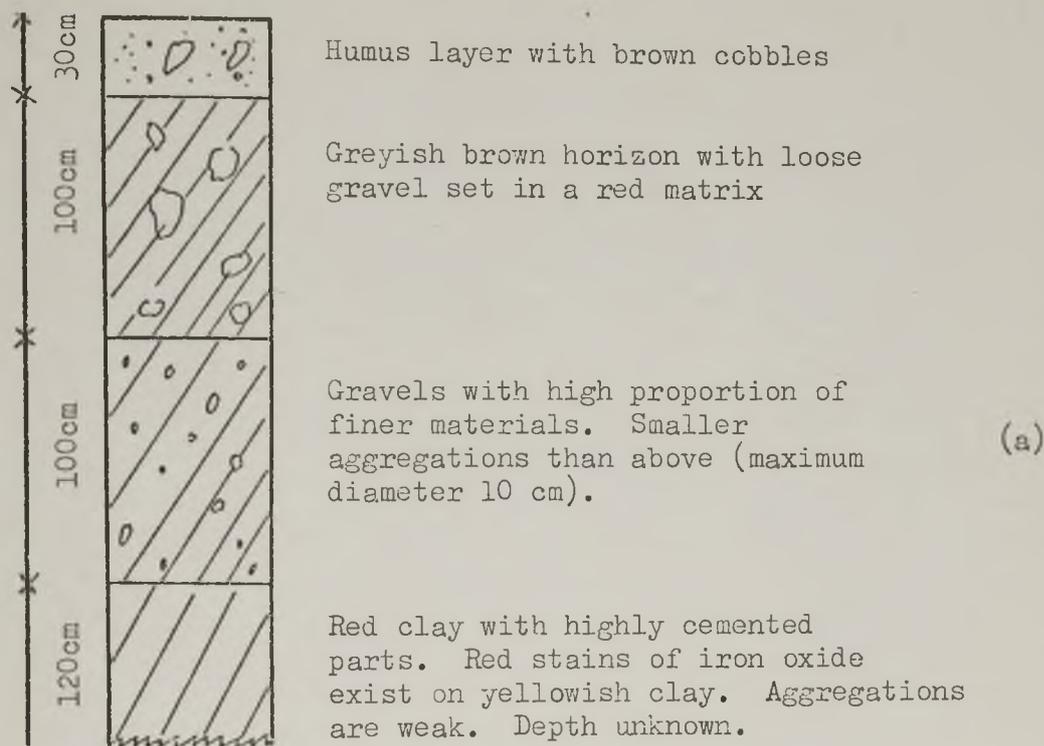


Fig. 5.5 Schematic diagram of Cuite profile at the locality where samples were taken for analyses

(a) Right side of cut

(b) Left side of cut

Thickness

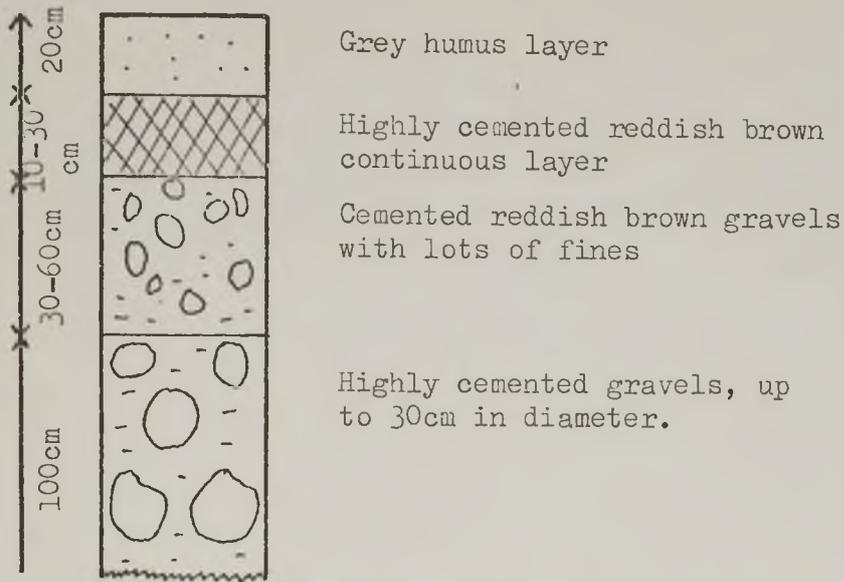


Fig. 5.6 Schematic diagram of Nova Floresta - profile at the locality where samples were taken for analyses.

diameters up to 25 cm. At the bottom of this is a concretionary layer similar to the previous in colour (reddish brown) with a higher proportion of finer materials and containing concretions of maximum diameter 10 cm. Thickness of this layer is about 1 metre. The gravels gradually disappear with depth and there is a transition to a sandy clay layer with iron mottles. The depth of this mottled layer is unknown but 80 cm of it is exposed.

Nova Floresta - similar to Cuite (a) except for differences in thickness of the layers and the presence of a hard continuous cap at the top (see Figure 5.5).

5.4 Sample collection:

The following general procedure was followed during the collection of materials for analysis:

- (a) Observation of profile and selection of layers of interest.
- (b) Removal of loose material affected by present day weathering.
- (c) Removal of representative samples and collection in polyethylene bags to safeguard against contamination and loss of water.
- (d) Transport to the laboratory and air drying.

5.4.1 Mineralogy and chemical investigations:

Cuite - Three representative samples were taken from the profiles at Cuite. For identification purposes the following code will be used for representation:

- (1) Samples taken at 1.0 m depth from the surface of the profile at the left side of the locality.
- (2) Samples taken from 2.0 m depth at the left side.
- (3) Samples taken from 2.0 m depth at the right side.

Nova Floresta - One sample was taken from the profile. The code here is:

(4) Sample taken at 1.2 m depth from the surface of the profile.

Sape - Three samples were taken from the profile and the codes for these are:

(5) Sample taken from 2 m depth

(6) Samples taken from 3 m depth

(7) Samples taken from 4 m depth

Specimen preparation in the laboratory:

The heterogenous nature of the samples and the fact that the diameter of the concretions is not a constant—large aggregation are dependent on the small ones - made some consideration necessary as to the sizes to be used in the analysis. Separation was made to observe the differences in composition of the different fractions of the indurated elements and the surrounding matrix.

Samples were initially divided into two fractions:

(a) Fraction of material greater than 9.5 mm

(b) Fraction of material less than 9.5 mm

This arbitrary division is based on the fact that most samples exist either below 2 mm or above 10 mm which makes the visual separation easy to carry out. The indurated elements are generally above 10 mm and the surrounding loose matrix usually below 2 mm.

(a) - is hereafter referred to as concretions.

(b) - is hereafter referred to as fines.

Both were divided into subfractions.

(b) was divided into four subfractions using standard grain size analysis - B.S.1377:

(i) 9.5 - 2.0 mm

(ii) 2.0 - 0.75 mm

(iii) 0.075 - 0.002 mm

(iv) less than 0.002 mm

For the division of (a) into subfractions the following procedure was adopted:- Since it is known that the degradation of the aggregation can be affected by immersion, the aggregates were therefore immersed in water for up to 4 days. These were made to undergo light mechanical treatment by receiving blows from a hammer while kept in polythene bags. The broken materials were later washed into sieves and two fractions obtained, a fraction between 0.075 and 0.002 mm and the fraction below 0.002 mm. The drying of all the fractionated specimens was limited to 60°C to avoid fundamental alterations that can occur when drying is carried out at higher temperatures.

Second digits were introduced and used along with the first to identify the subfractions. These are:

Fines:

- 0 - 9.5 - 2.0 mm
- 1 - 2.0 - 0.075 mm
- 2 - 0.075 - 0.002 mm
- 3 - less than 0.002 mm

Concretions:

- 4 - 0.075 - 0.002 mm
- 5 - less than 0.002 mm

e.g. 21 is Cuite at 2 m depth from the left side with sizes between 2.0 - 0.075 mm.

5.4.2 Compressibility and Shear Strength:

All representative layers from each of the deposits were taken and mixed for the determination of compressibility and shear strength characteristics. The samples were air-dried and pulverised using a jaw crusher - this method was preferred because it reduces all sizes

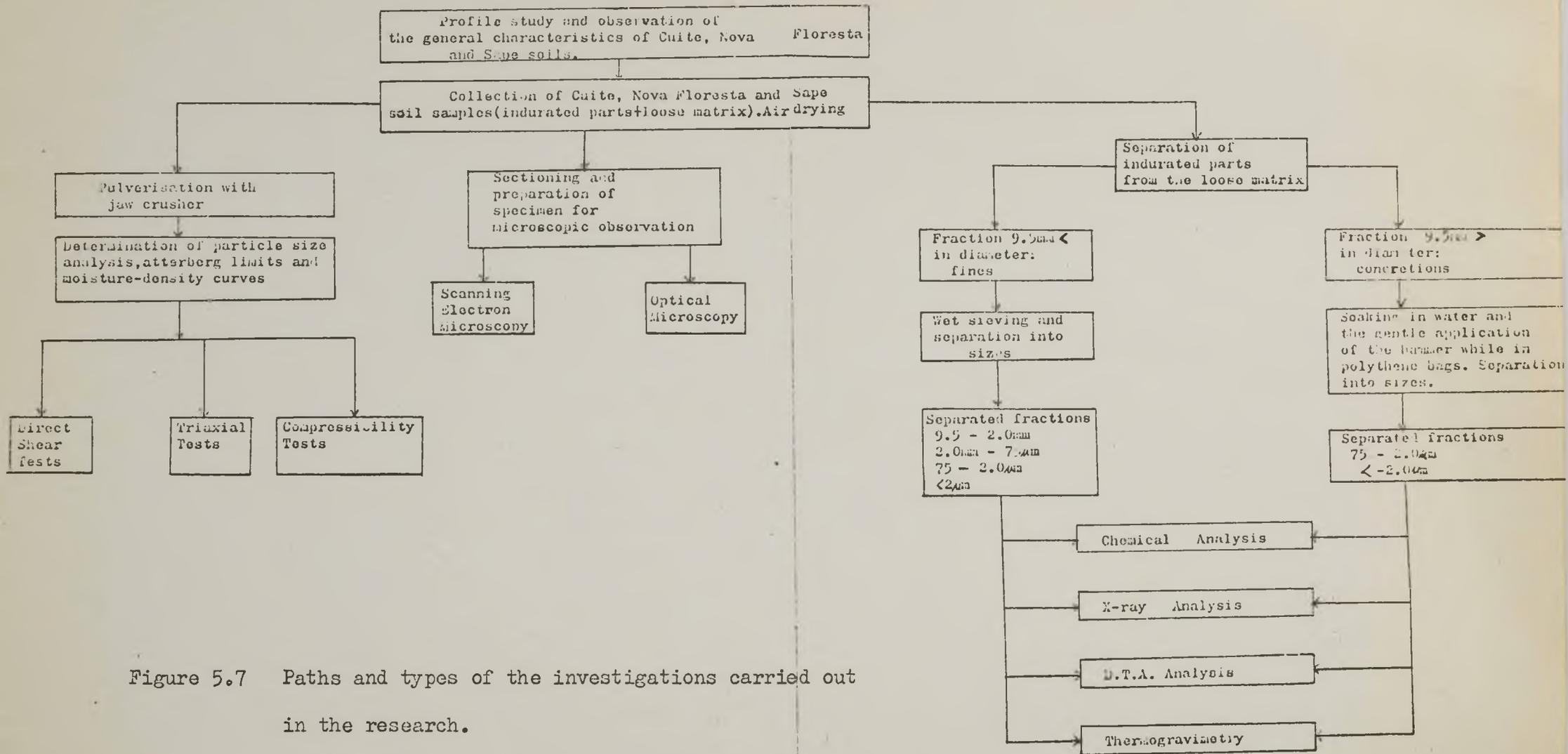


Figure 5.7 Paths and types of the investigations carried out in the research.

greater than 12.7 mm (1/2 in) to sizes below or equal to this while having little effect on the materials which originally had sizes below - since the larger sizes are essentially aggregates of smaller ones, disaggregation as a result of pulverisation was reduced to a minimum. This is the attempt to simulate as much as possible the field use conditions, particularly the use of the materials in road construction.

CHAPTER 6

MINERALOGY AND CHEMISTRY

5.1 Methods

The identification of mineral components in soils and clays is carried out by a number of methods based on the response of the materials to imposed physical and chemical environments.

Mitchell (1976, p.85) has listed the methods and techniques that may be employed for the determination of soil composition and the study of soil grains

1. Particle size analysis and separation.
2. Various pre-treatments prior to mineralogical analysis.
3. Chemical analysis for determination of free oxides, hydroxides, amorphous constituents and organic matter.
4. Petrographic microscope study of silt and sand grains.
5. Electron microscope study of the clay phase.
6. X-ray diffraction for identification of crystalline minerals.
7. Thermal analysis for identification of mineral components.
8. Determination of specific surface.
9. Chemical analysis for cation exchange capacity.
10. Staining test for identification of clays.

Many of the methods listed above will be encountered throughout this work, only the ones that concern the chemistry and mineralogy will be dealt with in this Chapter.

6.1.1 X-ray diffraction

6.1.1.1 General Background

The X-ray diffraction technique is now a routine method in the

identification of minerals in soils and clays, it is a very powerful tool employed by geologists, material scientists and metallurgists.

It is based on the fact that most materials are crystalline and hence show some similarity and regularity of structure. The orderly atomic arrangement in the crystals causes them to behave with respect to X-rays and electron beams in much the same way as does a diffraction grating with respect to visible light.

When the surface of a mineral is bombarded by an X-ray beam of a single, well-defined wave length, e.g. 1.5\AA (commonly used in diffraction work) the electrons in the atoms near the surface are made to vibrate synchronously and they re-emit X-rays which behave as if their source were the atom itself.

The emitted waves form electromagnetic wave fronts which expand outwards from each atom. The waves from all the atoms in the surface layer combine to re-form a plane wave front moving away from the surface at the same angle as the incident radiation, this process is called reflection and will occur at all angles of incidence. Due to the high penetrating power of X-ray the incident wave front encounters deeper layers imparting a portion of its energy to each of them (depth of penetration is about $10,000\text{\AA}$). Each of the atoms in the deeper layers can be considered as reflectors.

At some angles of incidence, the reflections from successive layers reinforce one another to give a diffracted beam which is unaffected by changes in the horizontal offset of the lower layers.

The geometric condition for the reinforcement is governed by Bragg's law. This can be expressed as:

$$n \lambda = 2d \sin \theta \text{ (Bragg's equation)}$$

$$\lambda = \text{known wavelength of X-rays}$$

- d = spacing between each of the diffracting planes
 θ = angle of incidence of the X-rays

All the possible sets of diffracting planes of atoms in a mineral can be obtained by rotating its crystals in an X-ray beam. A diffracted beam will emerge as the Bragg's equation is momentarily satisfied by each set of planes.

A simpler way of obtaining the same information is to grind the crystals to a fine powder which is really thousands of small crystals. The random orientation of the small crystals in the specimen ensures that at least a few would be in the correct position for any particular set of planes. The complete diffraction pattern is thus generated.

In a practical X-ray diffractometer, the angle θ is commonly increased from a low value and the intensity of the diffracted beam is continuously monitored. A diffraction maximum indicates that the sample contains a mineral with atomic planes spaces at the distance d , corresponding to the value of θ at which the maximum occurs.

The basic principle necessitates a source of X-rays, sample to be irradiated and a detection mechanism.

X-ray generation is usually achieved by using a tube in which electrons are caused to stream from a filament to a target material across a voltage drop of about 50,000V. Filters are used to produce required wavelengths (made as monochromatic as possible).

The detection of a diffracted beam can be accomplished electronically or by a photographic film. In the powder camera, the X-rays are detected by wrapping a photographic film around the inside of a cylinder. The powder is glued into a small, glass fibre at the centre of the cylinder.

In the electronic goniometer, an electric X-ray detector moves in

an arc around the powdered specimen. The output is recorded on a paper chart. Each diffracted beam is only recorded for a short time but this effect is compensated by mounting the powder as a film spread out over the surface of a glass plate or in a rectangular hole within a specimen holder, so that the irradiation is over an area rather than a point. The powder is illuminated by a convergent beam of high intensity. The diffracted beam will be convergent if the plate is held at the angle of diffraction to both the incident beam and to the detector. The plate must therefore rotate at half the speed of the detector and the detector must be mounted at the point of convergence of the rays. This arrangement gives an intense beam without serious loss of precision in the measurement of the diffracting angle.

The basic principle is that the planes of similar atoms in adjacent stacked units have a basal spacing which characterises the spacing between these layers. Thus it is possible to directly examine the structure of a given clay mineral. Moreover, changes in the state of interlayer complexes can be monitored and correlated with changes in external conditions producing the interlayer change (ASTM 1970).

6.1.1.2 Limitations to the use of X-ray analysis in the investigation of soil mineralogy:

X-ray diffraction as applied to the study of minerals is a very powerful tool. Its use in the investigation of soil mineralogy is, however, hampered by its fundamental principle.

The diffraction of X-rays by materials requires that the materials possess regularity of internal structure i.e. crystalline. Materials that are not crystalline i.e. amorphous will not diffract X-rays. X-ray diffraction is therefore only applicable to soils in which the minerals are crystalline (MacKenzie 1957 (a), Oades (1962)). Amorphous materials are known to exist in soils MacKenzie (1957 (b)). Although

not much attention has been given to them in the past. Alexander and Cady (1962), their role is now being re-examined. Amorphous constituents have been demonstrated to influence the properties (Townsend and Reed 1971) of red soils. Its presence in sensitive clays (quickclay) has also been demonstrated (McKeys et al. 1977, Bentley (1977, p.271).

To detect the presence of amorphous materials in soils, other methods are employed e.g. Thermal.

As a result of isomorphous substitution and interference due to the presence of other minerals, X-ray analysis is not adequate for accurate quantitative work. Carroll (1970, p.5) mentioned that the presence of accessory minerals e.g. anatase, gibbsite, quartz, can be found by X-ray diffraction '...only if they occur in excess of about 5 per cent by volume or weight'.

X-ray diffraction analysis of Brazilian red soils.

The identification of the mineral components of the different fractions of the Cuite, Nova, Floresta and Sape soils was carried out using this technique. A Phillips diffractometer equipped with a goniometer was used.

Sample Treatment: To produce the complete diffraction pattern of the mineral present, the sample had to be in powdered form. The specimen was used as powder when available below 75 μm or had to be ground to pass sieve No. 200 B.S. Grinding was carried out in a mortar. The process was interrupted frequently with sieving so that the below 75 μm fraction is not subjected to any more grinding than is necessary. Natural and glycol treated specimens were used.

Initially, in line with previous investigations of red soils (Correia et al. 1969, Moh and Mazhar, 1969) the soil specimens were

q - Quartz g - Goethite
 k - Kaolinite h - Haematite

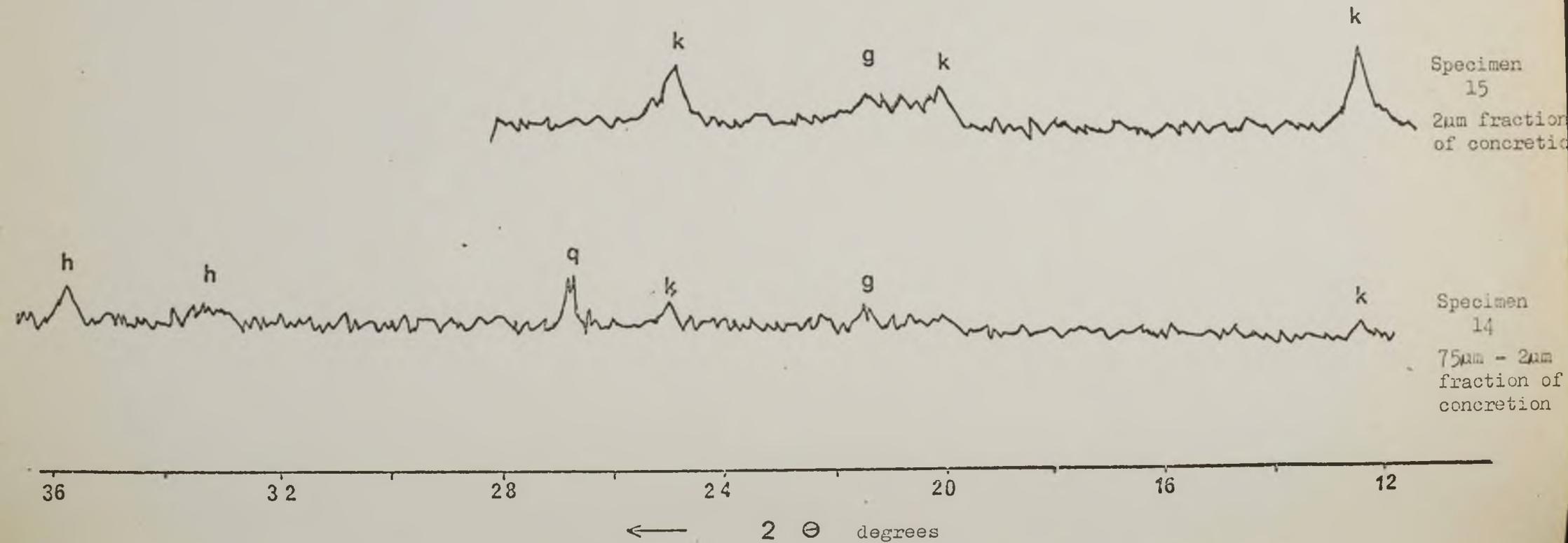


Fig. 6.1(a) X-ray Diffractogram obtained with Cu α radiation. Cuite soil. 1.0 m depth - left side.

analysed in a diffractometer with Copper $K\alpha$ radiation. The resulting diffractogram (Fig.6.1a) does not have very distinct peaks. This diffractogram shows that a high amount of absorption exists. The absorption which is mostly due to the presence of iron (Carroll 1970, p.46) is in agreement with the observation of Townsend and Reed (1971). Townsend and Reed demonstrated the existence of iron oxide coatings on soil particles of a Panamian red soil. The presence of iron oxide coatings on soil particles will lower the mineral peaks in the diffractogram, as a result of the absorption of incoming radiation by the coatings. The identification of the mineralogy of red soils with Copper $K\alpha$ (Cu $K\alpha$) radiation is therefore limited by the reason of absorption. ✕

A search was made for a radiation that will be suitable for red soils. This led to the preparation of Table 6.1(a). A table of the mass absorption coefficient of minerals that can be found in red soils. Examination of the table shows that the mass absorption coefficient of clays for Cu $K\alpha$ radiation ranges between 30 - 50. Its value for iron oxide minerals however, is in excess of 200. Since these soils were expected to contain iron (albeit from the red colour), Fe $K\alpha$ would be the preferred radiation.

An iron tube that produces Fe $K\alpha$ was, however, not available. The only available alternative was a Molybdenum tube. This was used and the resulting diffractogram (Fig.6.1b) has more distinct peaks. (compare with Fig.6.1a). The common soil minerals have a low absorption for Mo $K\alpha$ radiation (see Table 6.1b).

It should be mentioned that the higher energy of Mo $K\alpha$ radiation (shorter wavelength) could cause it to produce a high level of fluorescent radiation (secondary X-rays produced from the material under bombardment, when the energy of the incoming radiation is high enough

to eject electrons). It is therefore not strongly recommended for this reason. It was used therefore in conjunction with diffractogram produced with Cu K α radiation, before any mineral identification was carried out. Co K α ($\lambda = 1.7902$ and Fe K α ($\lambda = 1.9373$ are most suitable and should be used when available. The references for iron minerals in the ASTM file of index cards used Co K α radiation.

Table 6.1 (a)

Mass absorption coefficient of minerals commonly found in red soils, for Cu K α and Fe K α radiations.*

Mineral	Cu K α ($\lambda = 1.5418\text{\AA}$)	Fe K α ($\lambda = 1.9373\text{\AA}$)
Quartz SiO ₂	34.41	66.54
Kaolinite Al ₂ Si ₂ O ₅ (OH) ₄	29.67	62.04
Halloysite Al ₂ Si ₂ O ₅ (OH) ₄ · 2H ₂ O	29.35	52.94
Gibbsite Al ₂ O ₃ · 3H ₂ O	24.06	43.64
Boehmite AlOOH	47.79	92.34
Haematite Fe ₂ O ₃	218.55	53.18
Goethite FeOOH	240.33	56.22
Magnetite Fe ₃ O ₄	226.17	54.46

* Calculated from International Tables for X-ray Crystallography (1962), using the chemical formulas beneath the mineral names.

q - Quartz g - Goethite

k - Kaolinite h - Haematite

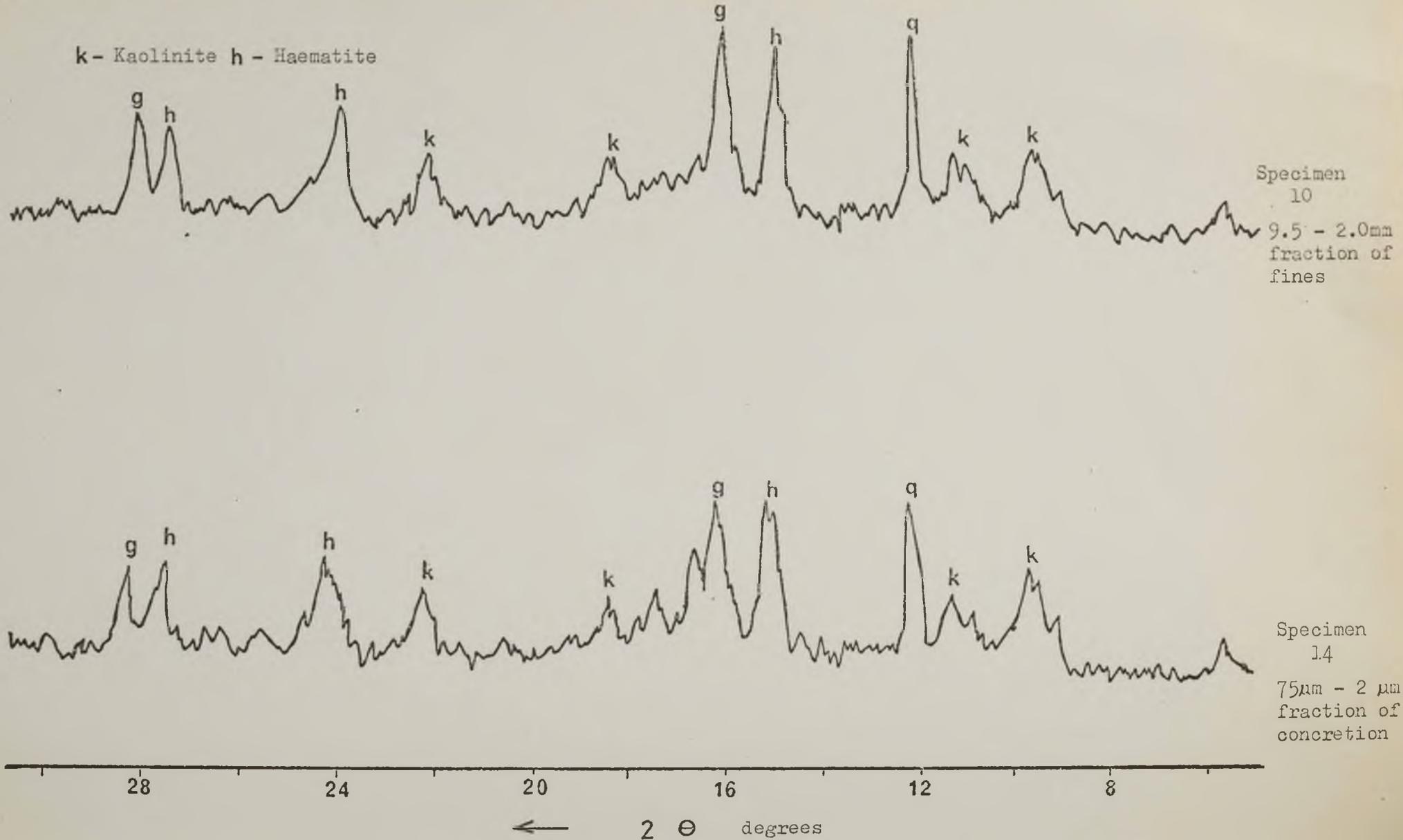


Fig. 6.1(b) X-ray Diffractogram obtained with Mo α radiation. Cuite soil. 1.0 m depth - left side.

Table 6.1 (b)

Mass absorption coefficients of minerals commonly found in red soils for Mo $K\alpha$ radiation.

Mineral	Mo $K\alpha$ ($\lambda = 0.7107\text{\AA}$)
Quartz SiO_2	3.69
Kaolinite $\text{Al}_2 \text{Si}_2 \text{O}_5 (\text{OH})_4$	3.78
Halloysite $\text{Al}_2 \text{Si}_2 \text{O}_5 (\text{OH})_4 \cdot 2\text{H}_2\text{O}$	2.96
Gibbsite $\text{Al}_2 \text{O}_3 \cdot 3\text{H}_2\text{O}$	2.74
Boehmite AlOOH	5.07
Haematite $\text{Fe}_2 \text{O}_3$	27.34
Goethite FeOOH	30.02
Magnetite $\text{Fe}_3 \text{O}_4$	28.24

Test conditions in X-ray analysis

Tubes - Cu $K\alpha$ and Mo $K\alpha$

Operating conditions - 40 kV, 15 mA for Cu $K\alpha$ and

42 kV, 14 mA for Mo $K\alpha$

Scanning rate - $\frac{1}{2}^\circ$ of 2θ per minute

Chart speed - 10 mm per minute

The results and the logic of identification can be found later in the Chapter.

6.1.2 Thermal Analysis:

6.1.2.1 Introduction

The technique of thermal analysis is another of the mineralogical techniques employed in the investigation of clay, when a material is subjected to a physical environment (heat in this case) and the response to the environment is determined.

Thermal analysis has been defined by Wendlandt (1964, p.4) as "techniques in which some physical parameter of a system is determined as a function of temperature". The dependence of a great number of physical properties of substances on temperature has long been recognised but the application of the changes that occur to characterise the material has only been of great importance in the last fifty years.

The common physical parameters measured in thermal investigations have been described by MacKenzie (1974). They include:

Mass changes - Thermogravimetry, derivative thermogravimetry,
isobaric and isothermal weight curves.

Energy changes - Differential thermal analysis,
differential scanning calorimeter,
heating curves.

Dimensional changes

Mechanical strength

Optical changes

Electrical changes

Magnetic changes

Emitted sound

Evolved volatiles

Reactions in solution.

The measurements can be carried out under static, quasistatic, or dynamic temperature conditions depending on the change in the temperature of the heat source with time. In general, the measured physical parameter is recorded as a dynamic function of temperature (Wendlandt, 1964, p.9).

The most widely used techniques are those that depend on change in mass energy, dimensions and on evolved volatile products as the temperature is increased and out of these, the ones that have been mostly applied in the analysis of soil composition are:

Thermogravimetry: depends on changes in mass.

Differential Thermal Analysis: depends on changes in energy.

Evolved Gas Analysis: analysis of evolved volatiles most applicable in non-clastic sedimentological studies.

The thermal analytical techniques employed presently, to ascertain the mineralogy of these samples are the ones mentioned above, namely:

Thermogravimetry, TG, with Derivative thermogravimetry DTG and Differential thermal analysis, DTA.

These are based on the reactions and transformations that occur within a sample as its temperature is raised from room temperature to say 1000°C. The responses to temperature increase are then measured in terms of the type of changes earlier described. Important common changes that occur on heating a clay or soil sample are:

(a) Dehydration: - This is the loss of water as a consequence of heating.

Water in soil samples are usually of three kinds:

- (1) Free pore water: - this is the water normally present in the pores or the hygroscopic moisture content. Usually lost at about 105°C.
- (2) Absorbed water: - this is the water absorbed by the clay mineral surfaces due to hydrogen bonding between the clay mineral and water molecules. Also included are absorbed water at the

surfaces of iron oxide minerals: - Goethite Haematite:

Usually lost up to 240°C.

- (3) (OH) ions in the crystal lattice susceptible to transformation to H₂O on disruption of any O - H bonds. This is referred to as dehydroxylation. Dehydroxylation causes a complete destruction of mineral structures whereas a removal of absorbed water is a reversible process. The temperature at which major amount of crystal lattice water is lost is the most indicative property of identification of minerals.
- (b) Crystallisation: - New crystals can form from amorphous materials or from components formed at lower temperature, e.g. decomposed crystals.
- (c) Phase changes: - Some crystal structures change from one form to another at a specific temperature.
- (d) Oxidation: - Combustion of organic matter or the transformation of oxides from lower oxidation states to higher ones, e.g. Fe²⁺ Fe³⁺.
- (e) Reduction: - Reversal of oxidation usually takes place in a controlled atmosphere.
- (f) Decomposition: - Carbonate materials decompose when the temperature is high enough to yield carbon dioxide CO₂.
- (g) Reaction with atmosphere.

These are changes utilised in general mineralogical studies, many others exist and some are enforced on the system, e.g. reduction in a controlled atmosphere. Gordon (1963) has separated reactions detectable by thermal analysis into those produced by physical changes and those which involve a chemical change as well.

Physical Changes: - These reactions involving physical changes that are detectable by thermal analysis include - Crystalline transition, Fusion, Vaporisation, Sublimation, Adsorption, Desorption, Curie-point

transition, Liquid crystal transition, Heat capacity transition.

Chemical Changes: - These include Chemisorption, Desolution, Dehydration, Oxidative degradation, Oxidation in gaseous atmosphere. Redox reactions, Solid state reactions, Combustion, Polymerisation, Pre-curing (Resins) and Catalytic reactions.

6.1.2.2 Thermogravimetry:

6.1.2.2.1 General Background:

This involves the record of continual weight loss as a specimen is heated. Thermogravimetric analysis has enjoyed a considerable popularity, as an approach to material analysis, with chemists and metallurgists for some length of time. The interest of the clay mineralogist and engineers in its use for the investigation of soils and clays is only recent.

The first scientific thermobalance was designed by Honda (Keattch and Dollimore 1975, p.5) who in fact was the first person to use the word "Thermobalance". It is a beam type of deflection thermobalance, which incorporated a system whereby a conversion into a null point type was possible. The specimen was placed in a platinum container which hangs down from the lower end of a thin porcelain tube fixed vertically to one end of a beam that rests on an agate knife-edge. At the other end of the beam a weak spiral spring is stretched vertically and attached to the bottom of a Dewar-flask filled with oil. The Dewar flask can be raised or lowered by means of a calibrated screw system. Weighing is achieved by determination of the beam deflection by adding a known weight to the pan or by adjusting the Dewar flask by means of a calibrated screw system (a null-point method).

Heating was accomplished by means of a platinum wire furnace wound non-inductively on a porcelain tube while temperature measurement was

by a platinum-rhodium thermocouple.

6.1.2.2.2 Factors affecting results:

The factors affecting results obtained from such a balance, in fact any thermobalance, have been described by Duval (1963, p.1-77). Wendlandt (1964, p.6) has divided the factors that can influence the mass change curve of a sample into two categories:

1. Instrumental (Thermobalance) factors
 - (a) Furnace heating rate
 - (b) Recording or chart speed
 - (c) Furnace atmosphere
 - (d) Geometry of sample holder
 - (e) Sensitivity of recording mechanism
 - (f) Composition of sample container.
2. Sample characteristics
 - (a) Amount of the sample
 - (b) Solubility of evolved gas
 - (c) Particle size
 - (d) Heat of reaction
 - (e) Sample packing
 - (f) Nature of sample
 - (g) Thermal conductivity.

All these factors have already been given some amount of attention elsewhere and will not be dealt with at length in this study. An important study of these is found in the book of Duval (1963).

Some errors result from these factors and interplay between a number of them. These include:

- Sample container air buoyancy
- Furnace convection current

Random fluctuation in the recording mechanism
Furnace induction effects
Electrostatic effects on balance mechanism
Environment of the thermobalance
Condensation of sample support
Temperature measurements and calibration
Weight calibration of recording balance
Chart-paper ruling
Reaction of the sample with sample container
Temperature fluctuation.

A number of these errors do not apply to the present study for they are reduced to negligible values in the apparatus design, while some can be avoided if cautionary steps are taken during experimentation. However, some are inherent in the design and corrections have to be made. An appraisal of the important ones is considered useful.

Sample container buoyancy:

The density of the gas phase decreases with increase in temperature, the convection within the air above the sample increases with temperature. As a result of this as well as the effect of heat on certain parts of the balance mechanism, a thermally inert crucible, when heated, gains weight.

Simons, Newkirk and Alferis (1957) have studied the effect of temperature increase on a Chevenard thermobalance and presented a table of apparent mass gain versus temperature for difference weight on the balance.

They suggested the application of empirical equations to the thermogravimetry (TG) curve. Other workers have suggested the use of vents at the top opening of the furnace as a means of determining the correction factor. Keattch and Dollimore (1975, p.26) however, considered that the best method is the preparation of a correction curve, with an empty

crucible or a crucible filled with a thermally inert material, up to a temperature of at least 100°C above the maximum working temperature, under conditions identical to those of the actual experiment.

In the investigations of the mineralogy of the red soils, the 'vitreosil' silica specimen holders were heated empty to determine the gain in weight with temperature and a correction of the results was carried out accordingly.

Temperature measurement:

Measurement of actual sample temperature is a difficult task if not an impossible one. The experimenter has to be satisfied with the temperature values measured near the sample. If the sample temperature is taken as the temperature measured by a thermocouple just above the sample container, then the temperature of the sample will either lead or lag behind the furnace temperature. The magnitude of this difference depends upon the nature of the reaction (whether it is endothermic or exothermic), the heating rate, the sample thermal conductivity and the geometry of the sample holder.

6.1.2.2.3 Thermogravimetry in the investigations of soils and clay:

The use of thermogravimetry in the investigation of soils and clays has not received as much attention as other methods such as X-ray and DTA. The lack of attention is, in part, due to the problems encountered in the use of the method as a basis for qualitative determination, since the peak temperature of a reaction which is characteristically used in TG depends greatly on such factors as the particle size and the rate of heating. The use of the method as a means for quantitative analysis has been achieved with some degree of accuracy, (Smalley et al. 1977). It is an apparently untapped powerful method for the quantitative analysis of clay minerals.

Schnitzer et al (1959) employed TG in the quantitative analysis of soils and clays. They carried out TG analysis of soils, relatively pure clays, crystalline carbonates and soils to which known amounts of carbonates were added. Close agreement between TG and chemical methods of quantitative analysis was achieved in the determination of organic content, particularly for soils with up to 40% clay. They present a thermogram of a 1 : 1 mixture of Fithian Illite and H-bentonite. They were able to distinguish the two components of the mixture based on the standard materials of the two initial components.

Mitchell et al. (1966) have used TG to study quantitatively the amount of organic material from limestone. Smalley et al. (1975) have used the method to quantitatively study the mineralogy of quickclay of St. Jean Vianney. They concluded that the quickclay consist mostly of primary mineral particles. A conclusion earlier reached by Gillot (1971) based on X-ray analysis and differential thermal analysis. The agreement between the results from the different methods show that thermogravimetry deserves more attention than it is being presently given.

6.1.2.2.4 Thermogravimetric Analysis of Red Soils:

The thermogravimetric analysis of the different fractions of the red soils was carried out in a Stanton TR O₂ thermobalance to determine the amount (albeit semi-quantitative) of mineral contents identified with X-ray analysis and DTA.

The equipment is a beam form of the deflection type instrument consisting of a capacitance-follower plate which measures the beam movement and records the change in weight for a pre-determined beam movement; it includes an automatic weight loading arrangement which activates a microswitch that adds or subtracts a particular weight when the beam movement reaches the end of a deflection.

A side-by-side record of the furnace wall temperature, weight and time is given by the recorder.

A temperature programmer is enclosed which gives linear heating rates for about three different rate values. Temperature measurement is done by a platinum rhodium-platinum thermocouple with the hot-junction on the furnace wall.

The furnace itself is a vertical 50 mm bore, with nichrome bifilar windings that reaches a maximum temperature of 1000°C . The bifilar configuration allows the study of magnetic materials (such as iron-oxides) and ensure no magnetic interaction between the furnace and the sample. The furnace is linked to a transformer assembly, which automatically adjusts the voltage supply to the furnace to acceptable values, over the full range of temperatures encountered during a thermogravimetric run.

The sample was heated in static air using silica crucibles at the rate of $300^{\circ}\text{C}/\text{hour}$. Temperature measurement was via the furnace thermocouple and the furnace wall temperature was taken as the specimen temperature. 1.0 g was the usual weight of the sample taken, but trial runs of 0.5 g were carried out to see the effect of specimen size on the curves. The results and observations based on these are presented later in this Chapter.

The DTG curves were obtained from TG curves by plotting weight variation in five-minute intervals against the temperature. The period five-minutes was chosen for convenience since a time marker pen is incorporated in the recording mechanism. This marks off five-minute periods in the normal TG curve.

6.1.2.3 Differential Thermal Analysis:

6.1.2.3.1 Technique:

The heating of a material produces energy changes in the material. If the increase in energy is sufficient enough to produce a reaction or a transformation (see p.92) then during the course of the reaction, a non-linear condition is imposed on the temperature increase with time, e.g. for an endothermic reaction; energy is added to the system without changes in temperature from the initiation to completion, the reverse for an exothermic reaction.

If a specimen capable of undergoing one of the changes listed in p.92 is heated, with one which is incapable (i.e. inert), assuming they have similar thermal properties, the temperature increase in the inert material will continue linearly while that of the specimen will suffer breaks where non-linearity exists. If the temperature difference between the two is measured, such difference will be registered only when the non-linearity is existent and this is the basis of the DTA.

The DTA apparatus usually consists of a sample holder, usually ceramic nickel or platinum and a furnace as a source of heat. A temperature programmer to provide constant rate of heating, thermocouples for measurement of temperature as well as the temperature difference between the sample and the inert reference material, and a recorder for the output from the thermocouples. Sample weight is of the order of 1 gram.

Le Chatelier (Wendlandt 1964, p.1) was described as the first person to use thermal analysis, his actual method was to obtain a heating-analysis curve rather than a DTA. His work involves embedding a thermocouple in a clay sample heated at about $10^{\circ}\text{C}/\text{min}$. Response from the thermocouple connected to a galvanometer, was measured by reflection of flashes from an induction coil on the galvanometer mirror, to a photographic plate. The temperature of the sample was then displayed on

the developed photographic plate, as a series of lines, each of which corresponded to a spark discharge. An endothermic reaction was indicated by closely spaced lines, while wider spacing was indicative of an exothermic reaction.

Roberts Austen (1899) has been credited as the discoverer of the DTA as we know it. He suggested the use of a double thermocouple, where one is placed in the sample and the other in a reference block in the furnace.

In this method of analysis, material scientists and clay mineralogists provided the lead, in the application of the method to the study of thermally-aided reactions. A good historical review can be found in the works of MacKenzie (1957) and Wendlandt (1974).

6.1.2.3.2 Factors affecting results:

As for thermogravimetry, the results obtained from DTA depend on a variety of factors which are influenced by the instrument and sample characteristics. Instrument factors include - Furnace atmosphere, furnace size and shape, sample holder material, sample holder geometry, wire and bead size of thermocouple junction and heating rate. Factors dependent on sample characteristics include particle size, thermal conductivity, heat capacity, packing density, swelling or shrinkage of the sample, amount of the sample, affect of diluent and degree of crystallinity.

The DTA like X-ray diffraction analysis is almost routine in the investigation of soils and clays and no further elaboration in connection with general problems and previous investigations. Excellent books and book chapters exist on these, MacKenzie (1957), Garn (1965), Blazek (1974) are among a few.

However, a few general observations as is pertinent to the present work are worth mentioning, particularly in the relationship of the DTA

to other methods of mineralogical analysis.

6.1.2.3.3 Limitations to the DTA:

Many of the limitations to the DTA apply to thermal analysis in general, but some are peculiar to it, all these will be discussed in this section.

MacKenzie (1957a) has discussed the limitation of the use of differential thermal analysis in the investigations of clays. The limitations to the use of the method in qualitative terms can be divided into:

- (a) Those resulting from the apparatus
- (b) Those resulting from the technique
- (c) Those resulting from mineralogy

(a) - This is due to such problems as furnace induction, heating rate etc.

(b) - Results from the empirical value of the method which gives rise to many difficulties originating from small differences in technique.

Rigid standardization of technique is essential, before results obtained from one apparatus can be compared with those obtained from another.

(c) - Mineralogy - The use of DTA in clay mineral identification is beset with problems due to the very fact that the peak temperature which is used for reporting, is a variable and depends highly on the degree of crystallinity. MacKenzie (1957a) concluded that the identification of minerals from a normal differential thermal curve is very difficult and frequently impossible.

Limitations to the use in quantitative determinations - Quantitative work is based on the determination of the area under a peak and a reference base line. For this it is necessary to know the area under the peak of a pure mineral identical to the sample under investigation, a solution, as noted by MacKenzie (1957a) which is not always practicable. A

further problem is that of overlapping peaks which might not lend themselves to easy resolution.

6.1.2.3.4 Differential thermal analysis of red soils:

The results obtained by this investigation are beset by the problem listed in the discussions of the limitations. However, such problems as the standardisation of techniques and to a lesser extent the variation in the crystallinity of minerals have been to a considerable extent resolved at the present date, since increased communication has allowed the standardisation of equipment. The DTA results were employed as an aid in the qualitative determinations to supplement X-ray diffraction results.

The limitations to quantitative determinations mentioned, are easily overcome in this work, since fairly simple mineral systems are encountered and luckily enough they show changes at widely spaced temperature ranges, thus removing the problem of overlapping peaks, in this respect, the DTA result could serve as a sound enough basis for quantitative analysis. However, due to the greater ease of thermogravimetry which provide nearly the same amount of accuracy, it was employed in quantitative work.

The DTA analysis of the samples were carried out using Technical Equipment Corp. "Delta Therm" model D6000 DTA apparatus specimen holders and block were of ceramic and temperature measured by platinum thermocouples. Operating conditions were:

Heating rate $10^{\circ}\text{C}/\text{min}$.

Maximum temperature $1,000^{\circ}\text{C}$

Sensitivity $10^{\circ}\text{C}/25.4 \text{ min}$.

Chart speed $7.6 \text{ mm}/\text{min}$.

The results and observations are presented later in this chapter.

6.1.3 An appraisal of the different methods:

The X-ray diffraction appears to be the most powerful tool employed in mineralogical analysis. It is the most satisfactory single procedure for the identification of complex mixtures of clay minerals (Brindley 1951). A considerable advantage enjoyed by thermal methods of analysis is that unlike X-ray diffraction, they are not only applicable to crystalline minerals but to amorphous material too.

The relative merits of the different methods of thermal analysis have been considered by various workers. Newkirk and Simons (1966), Erdey et al. (1954).

The advantages of the DTG have been summarised by Erdey et al:

1. The curves may be obtained in conjunction with TG and DTA measurement.
2. The curves for DTA and DTG are comparable but the results of the former indicate, even those changes of state that are not accompanied by loss in mass. The curves of the latter method are more reproducible.
3. While the curves for the DTA extend over a wider temperature interval, due to subsequent warming of the material after reaction, the DTG measurements indicate exactly the temperature of the beginning, the maximum rate and the end of the change.
4. On the TG curves, changes following each other very closely cannot be distinguished, as the corresponding stages coincide. The DTG curves of the same change indicate by sharp maxima that the thermogravimetric stages can be divided into two parts.
5. The DTG can be used for the investigation of materials which for some reason or another cannot be analysed by DTA, e.g. some organic compounds melt during heating even so, DTG method yields fairly good results.

Newkirk and Simons, (1966) have criticised the relative importance given to the DTG and have sounded a note of warning that the DTG curve, whether derived mathematically or recorded directly, contain no more information than does an integral TG curve, obtained under the same experimental conditions, it simply displays it differently.

6.2 Results and Observations:

6.2.1 X-ray analysis:

The X-ray diffractograms of the different fractions are presented in Figure 6.2 - 6.4 and Appendix B. The d spacing of the peak calculated from Bragg's formulae were compared with a d spacing of standard minerals as recorded by the American Society for Testing Materials (ASTM 1970b) on their sets of file cards.

The minerals identified in the samples are kaolinite (usually disordered), Goethite, Haematite, Quartz, Anatase. The relative proportions of these, based on the intensities of major peaks are shown in Tables 6.2 - 6.4.

Kaolinite was identified on the basis of the reflections given at 7.14 \AA (001) and 3.75 \AA (002) in the absence of Chlorite which give a 7.13 \AA reflections as well as a 14.25 \AA one. The absence of ~~metahalloysite~~ halloysite was assumed because the diffractograms of the sample of natural material that have not suffered air-drying did not show any peak at 10 \AA characteristic for halloysite.

Haematite and Goethite - The identification of one in the presence of the other posed a problem for just X-ray analysis and the resolution is indicative of the use of these techniques to complement one another.

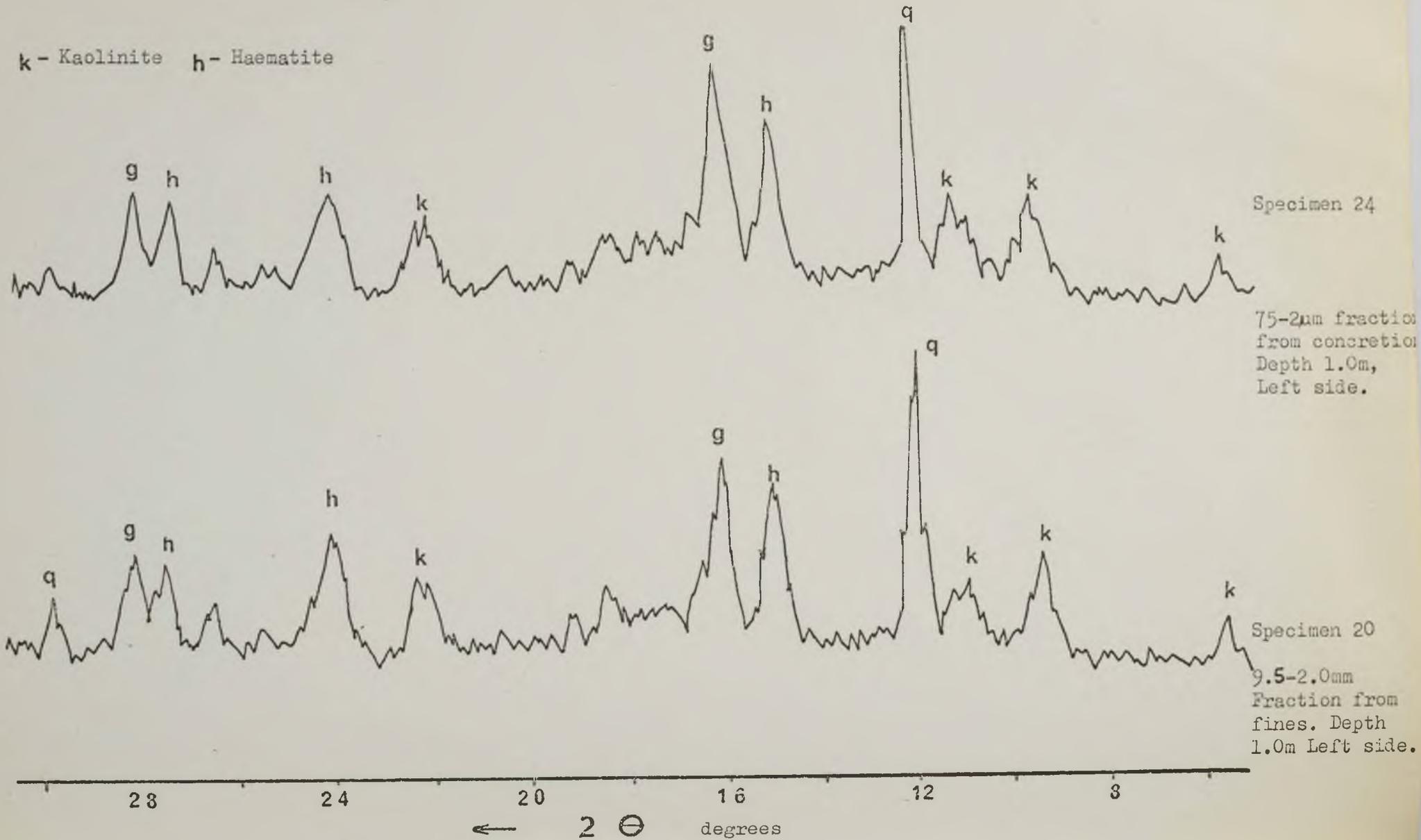
Most previous works, e.g. Coleman et al. (1964) Moh and Mahzar (1969), have identified iron-oxide in their sample but failed to draw any distinction between different forms of iron-oxide despite the knowledge

q - Quartz

g - Goethite

k - Kaolinite

h - Haematite

Fig. 6.2 Typical X-ray Diffractogram of Cuite soil (Mo K α radiation)

q - Quartz g - Goethite
 k - Kaolinite h - Haematite

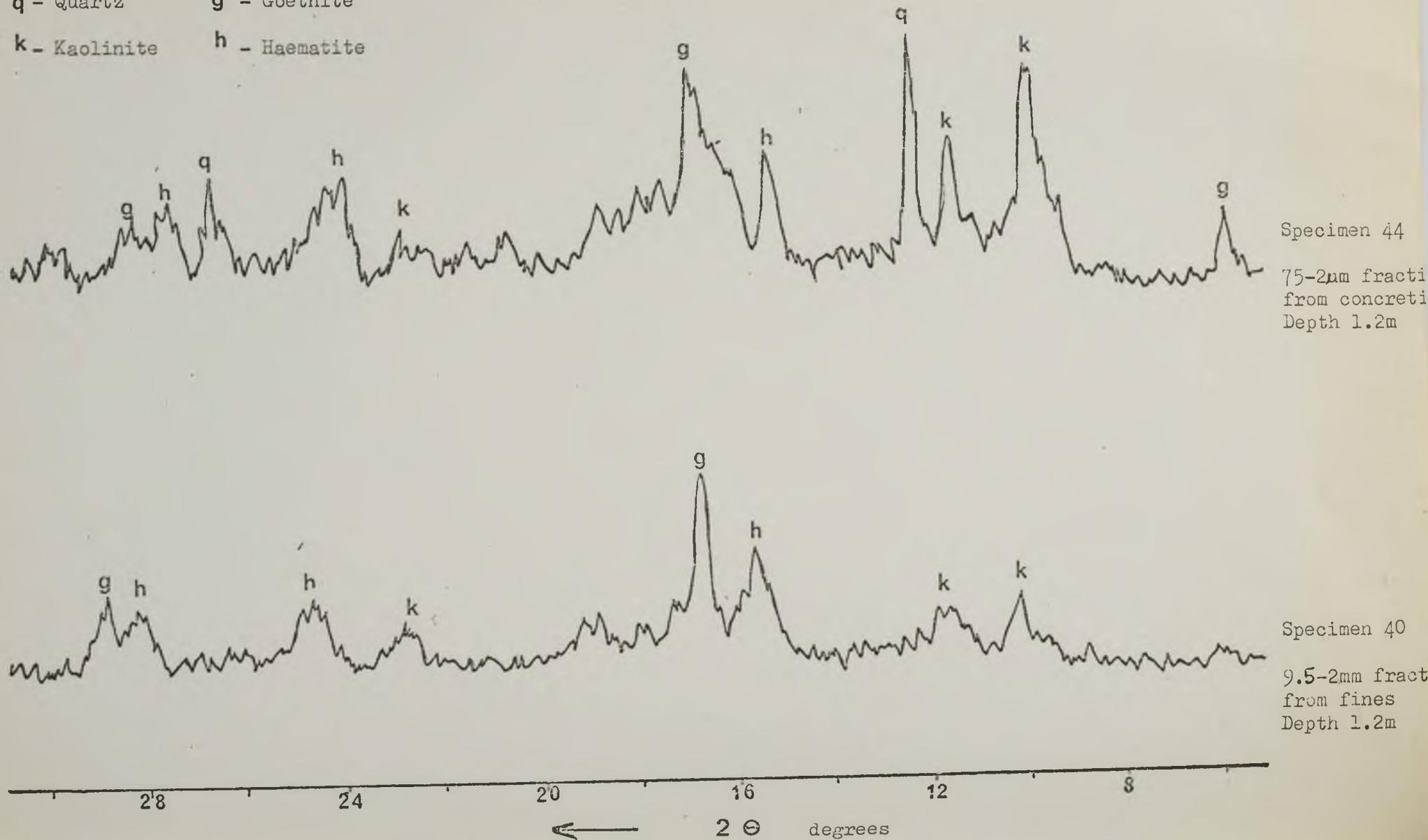


Fig. 6.3 Typical X-ray Diffractogram of Nova Floresta soil (Mo K α radiation)

q - Quartz g - Goethite

k - Kaolinite h - Haematite

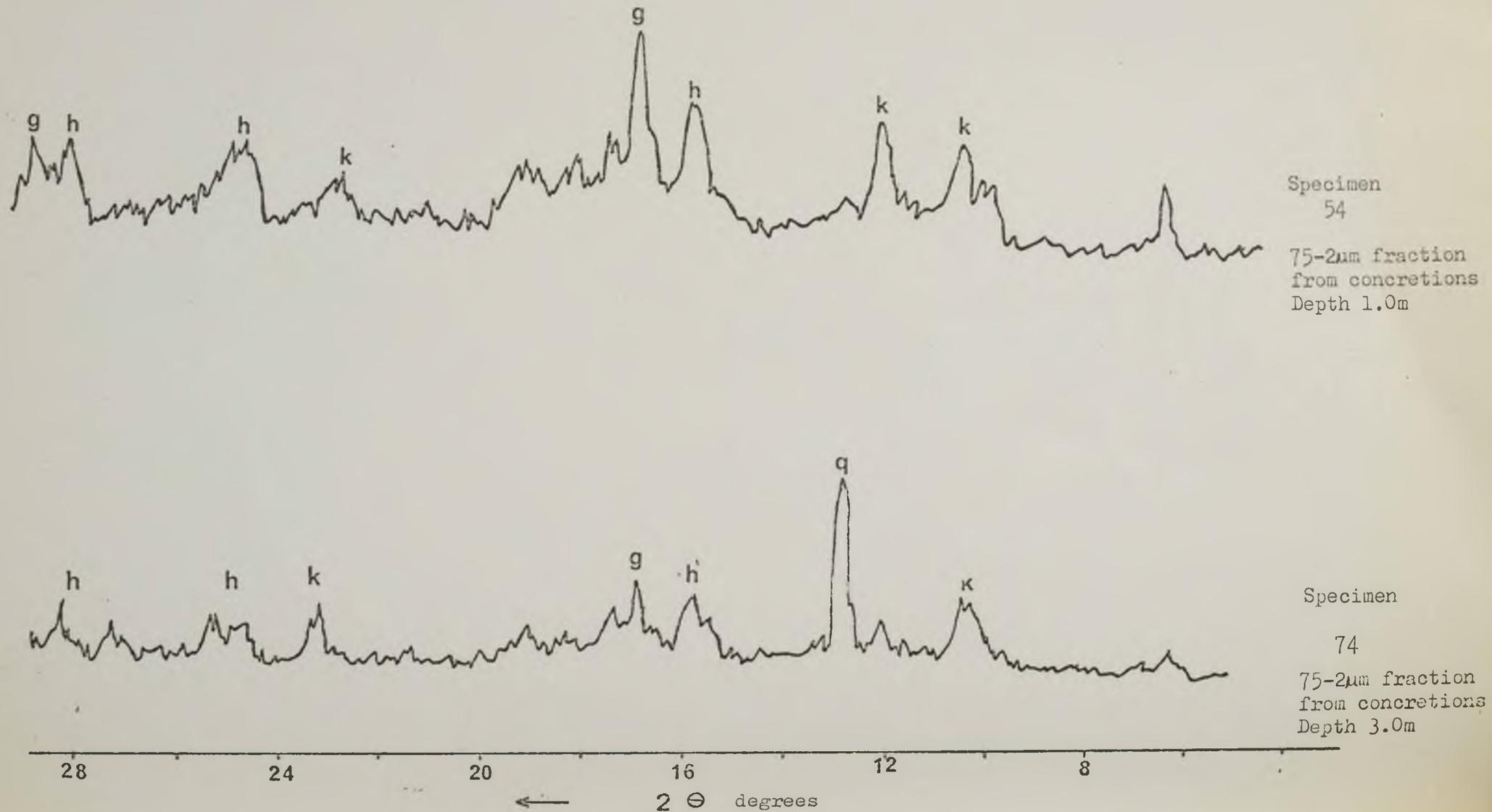


Fig. 6.4 Typical X-ray Diffractogram of Sane soil (Mo K α radiation)

and conclusions that the iron-oxides considerably influence the engineering properties of these materials.

Only the first few reflections are of any use in the identification of a multi-mineral system in particular for those in which varying degrees of crystallinity exist, resulting in considerable background. The highest peak intensity for goethite and the second highest for goethite occur at d spacing of 2.69\AA .

The highest for goethite at 4.2 is close to the 4.26 (100) reflections for quartz. Owing to the high degree of crystallinity of quartz in these samples, considerable amounts tend to manifest in the masking of peaks of minerals (Malomo, 1974).

The third highest peak intensities for both haematite and goethite also occur too close together for easy resolution: 2.45\AA for goethite and 2.25\AA for haematite. Though goethite was indicated by the diffractograms, confirmation had to be achieved by DTA analysis.

Haematite was confirmed by the 1.69\AA peak. Quartz was identified by its (101) reflections at a d spacing of 3.34 which was confirmatory in the absence of mica.

A problem which could not be resolved is the presence or absence of anatase. In the chemical analysis, certain specimens were found to contain up to 4.35% of titanium oxide. The highest intensity for anatase (which is the form of titanium in weathered soils) at 3.15\AA is very near that of kaolinite at 3.58\AA and precise identification is thus a problem at small concentration. The minerals identified in the fractions below 2u m are kaolinite, haematite, goethite and anatase. The relative concentrations are shown in Tables 6.2 - 6.4. Minerals identified in the fractions above 2u m are quartz, kaolinite, haematite, goethite, anatase.

TABLE 6.2

CUITE - RELATIVE ABUNDANCE OF MINERALS IDENTIFIED WITH
X-RAY DIFFRACTION

Specimen	Kaolinite	Haematite	Goethite	Anatase	Quartz	
10	++++	++++	+++	tr	++++	
11	++++	+++	++	tr	+++	
12	++++	+++	++	tr	++	
13	++++	++	+	tr	-	
14	++++	++++	+++	tr	++++	
15	++++	++	+++	tr	-	
20	++++	++	+	tr	+++	
21	++++	++	+	tr	++++	
22	++++	++		tr	++	
23	++++	++	+	tr	-	
24	++++	++++	+++	tr	++++	
25	++++	++	+++	tr	-	
30	++++	++++	+	tr	++	
31	++++	+++	++	tr	++++	
32	++++	++	++	tr	-	
33	++++	++	+	tr	++++	
34	++++	++++	+	tr	-	
35	++++	++++	+			
++++	very abundant		+	little		
+++	abundant		tr	trace		
++	some		-	none		
	very abundant	abundant	some	little	trace	none

TABLE 6.3

NOVA FLORESTA - RELATIVE ABUNDANCE OF MINERALS IDENTIFIED WITHX-RAY

Specimen	Kaolinite	Haematite	Goethite	Anatase	Quartz
40	++++	++	++	tr	+++
41	++++	++	+++	tr	+++
42	++++	++	++	tr	++++
43	+++	++	+++	tr	-
44	+++	++++	++++	tr	++
45	+++	+++	++++	tr	-

++++ very abundant + little

+++ abundant tr trace

++ some - none

very abundant abundant some little trace none

TABLE 6.4

SAPE-MARI - RELATIVE ABUNDANCE OF MINERALS IDENTIFIED WITH
X-RAY DIFFRACTION

Specimen	Kaolinite	Haematite	Goethite	Anatase	Quartz	
51	++++	++	++	+	++	
52	++++	++	++	+	++	
53	+++	++	+++	tr	-	
54	++	+++	++++	+	tr	
55	++	+++	++++	tr	-	
61	++++	+	+	++	+	
62	++++	+	+	+	+	
63	++++	+	++	+	-	
64	++	++++	+++	+	tr	
65	++	++++	+++	tr	-	
71	++++	++	+	++	+	
72	++++	++	++	++	+	
73	++++	++	+++	+	-	
74	++	++++	++++	++	+	
75	++	++++	++++	tr	-	
	++++	very abundant	+	little		
	+++	abundant	tr	trace		
	++	some	-	none		
	very abundant	abundant	some	little	trace	none

6.2.2 Differential Thermal Analysis:

The DTA thermograms were characterised by a maximum of six thermal events in most cases, all denoted by peaks, some of which reduced to second-order transitions i.e. one in which only a change in the horizontal base-line is detected. Standard graphs from an atlas of thermo-analytical curves (1971-74) by Liptay (1971) and observations from books by MacKenzie (1957) and Garn (1965) helped in the identification. The events can be numbered i - vi.

✓ (i) 100 - 110°C

This is an endothermic peak which is due to loss of hygroscopic moisture of the material.

✓ (ii) 180 - 220°C

This is an endothermic peak which reduces to second order transition in certain cases. When it occurs at lower temperature it sometimes merges with the enhanced peak of loss of combined water at 105°C resulting in a larger but ill-defined peak. Two possible interpretations for this peak are:

- (1) The presence of organic matter which is known to give endothermic peaks in this region (see Grimshaw and Roberts, 1957).
- (2) The presence of poorly crystalline oxides and hydroxides of iron or silica which will dehydrate at a high temperature.

Chemical analysis for the organic matter show that it is present but in amounts less than 3%. Analysis of a sample tested with H₂O₂ failed to remove this peak.

The peak was therefore interpreted as mainly due to poorly crystalline oxides.

✓ (iii) 320°C - 360°C

An endothermic peak more prominent in the specimen from the

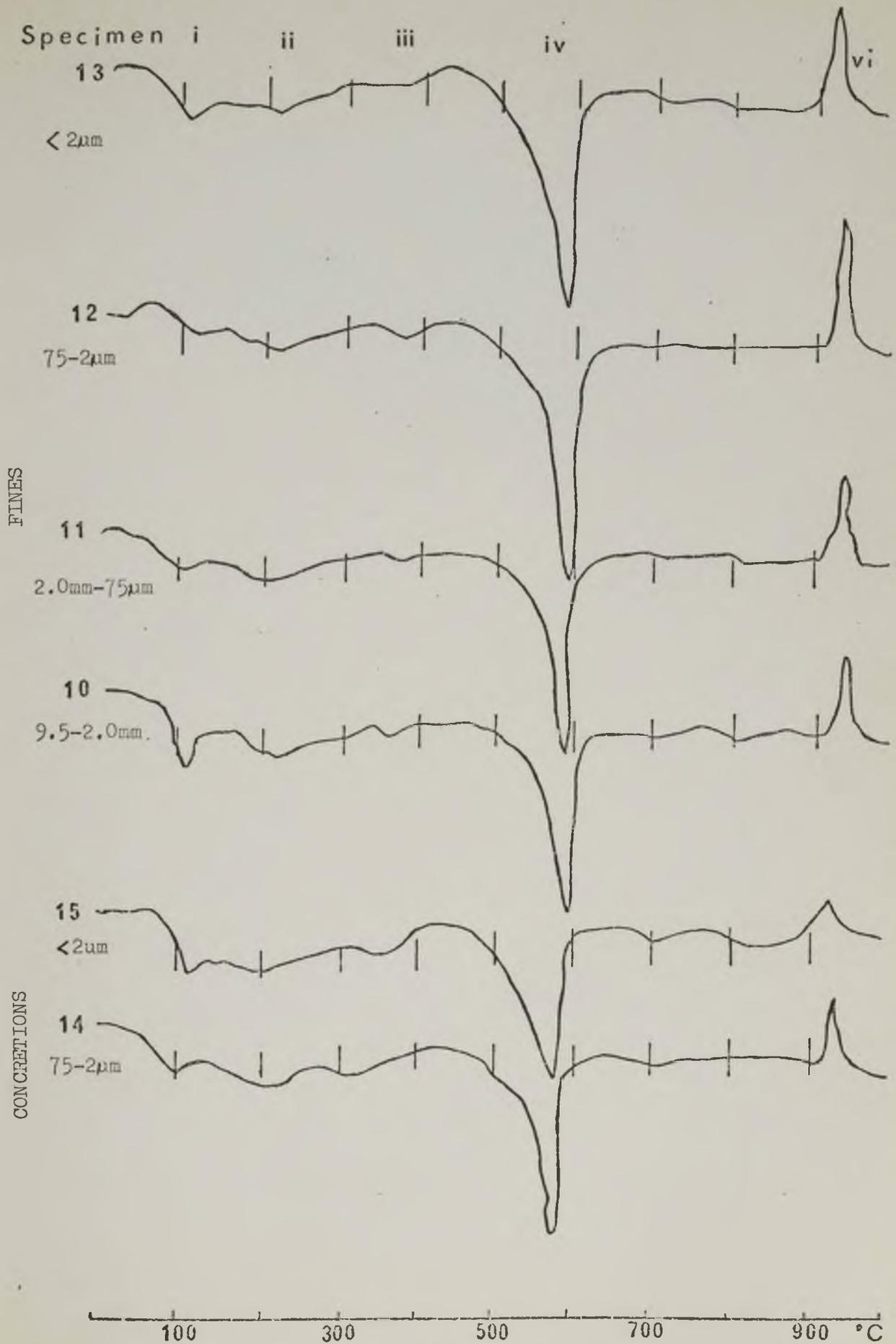


Fig. 6.5 Typical D.T.A. of subfractions from the same horizon in Cuite soil. Depth 1.0m, left side.

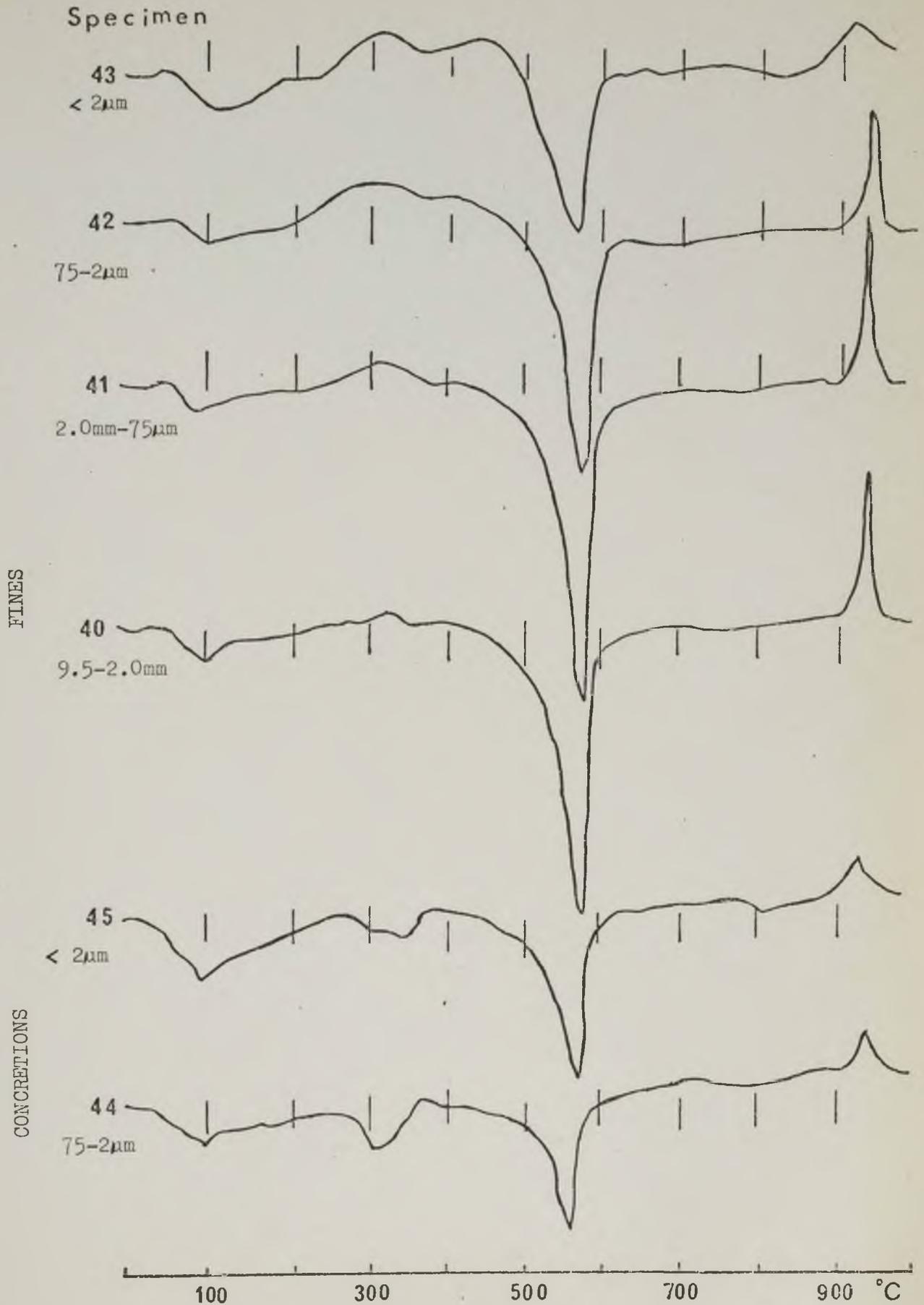


Fig. 6.6 Typical D.T.A. of subfractions from the same horizon Nova Floresta soil. Depth 1.2m.

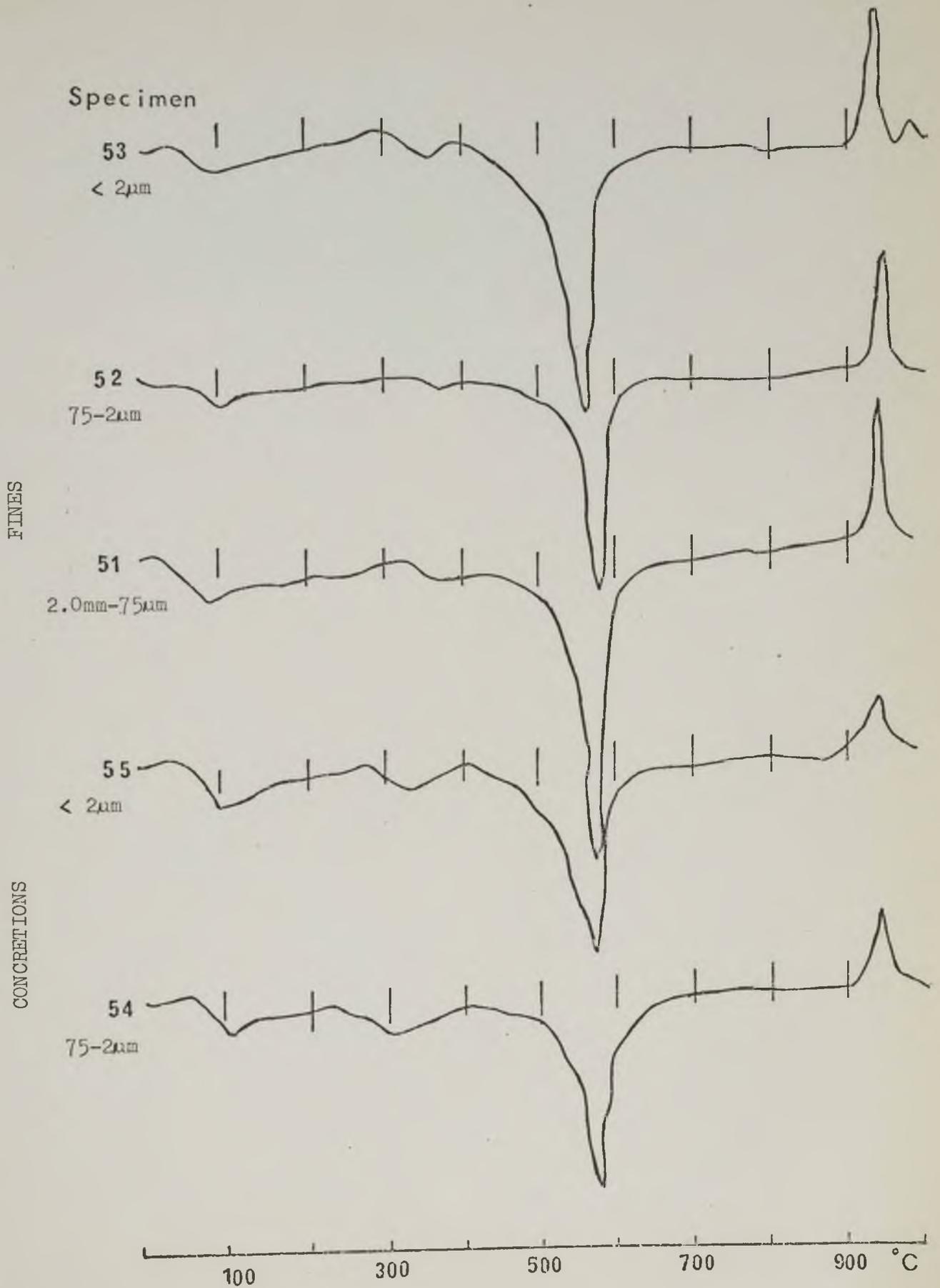


Fig. 6.7 Typical D.T.A. of subfractions from the same horizon in Sape soil. Depth 1.0m

concretions than from the fines indicative of the dehydration of goethite to haematite.

✓ (iv) 550°C

A strong, endothermic peak attributed to the dehydroxylation of clays (kaolinite in this case). It is the most diagnostic peak for kaolinites in samples and represents the collapse of the structure and formation of amorphous material.

(v) 930°C

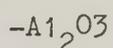
A third endothermic peak at 930°C just before a major exothermic peak occurs in most of the samples. This peak has been attributed by Brindley and Hunter (1955) to the retention of some form of order in dehydrated kaolinite and on this basis Holdridge and Vaughan (1957) suggest that it is reasonable to suppose that a trace of water may also be retained and that its removal accounts for limited endothermic reaction.

An alternative explanation also given by Holdridge and Vaughan is that of a poorly ordered compound which decomposes to amorphous alumina and silica, thermochemical considerations show that this would involve a very small energy absorption to give an endothermic peak.

✓ (vi) 945°C

The only exothermic peak within the temperature range of the experiment. Opinions differ as to the significance of this reaction but all varied observations can be narrowed down into two:

(1) Recrystallisation of amorphous alumina and the formation of



(2) Crystallisation of mullite $3Al_2O_3 \cdot 2SiO_2$ from amorphous alumina and silica.

The presence of these peaks was used as an identification procedure and no attempt is therefore made to go into the controversies surrounding their significance.

6.2.2 Thermogravimetry and Derivative Thermogravimetry:

The derivative thermogravimetry curves were obtained from TG curves by plotting weight changes in 5 minute intervals. This appears to be a simple and satisfactory way of obtaining a derivative curve from the TG result produced by a Stanton balance (Smalley et al. 1977). Identification was made by comparison with the DTA curves. It was recognised that the peak temperatures are not coincident but the region and shape are considered as useful aids to identification. Three weight loss peaks that can be numbered A-C are present in all the samples studied, though in different quantities (Figures 6.8 to 6.10).

Peak A - this occurs at 100-110°C and is due to loss of combined water in the specimen.

Peak B - 320°C due to the dehydration of goethite and the formation of haematite. It is more pronounced (therefore contains more material) in the specimens from the concretions.

Peak C - 480°C. This is due to the dehydroxylation of kaolinite in the sample.

The simplicity of the determination of amount of goethite and kaolinite is the main reason for using this method. Table 6.5 shows some results of determinations on the Cuite and Nova Floresta soils.

TABLE 6.5

Quantitative determinations of the kaolinite and goethite contents of some specimens from Cuite and Nova Floresta soils.

- (i) Determined from Thermogravimetry
- (ii) Determined from Chemical analysis

	Kaolinite (i)	Goethite (i)	Kaolinite (ii)
10	37.24	24.92	46.55
20	37.67	15.55	75.90
30	51.79	15.63	51.06
40	37.26	30.91	72.10

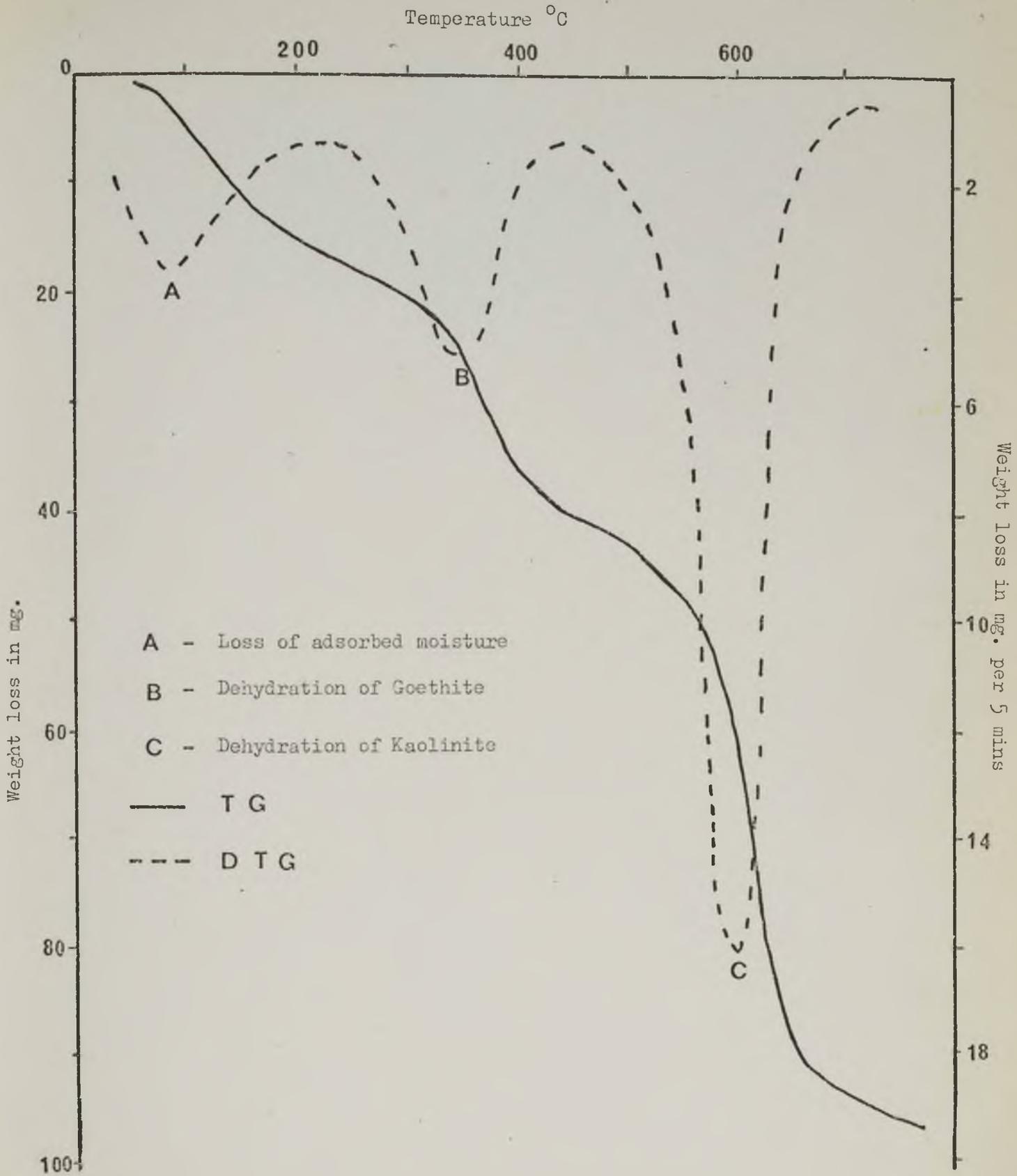


Fig. 6.8 Typical T.G. and D.T.G. curves from Cuite soil.
 (Specimen 15 - <math> < 2\mu\text{m}</math> fraction from concretion Depth 1.0m,
 left side)

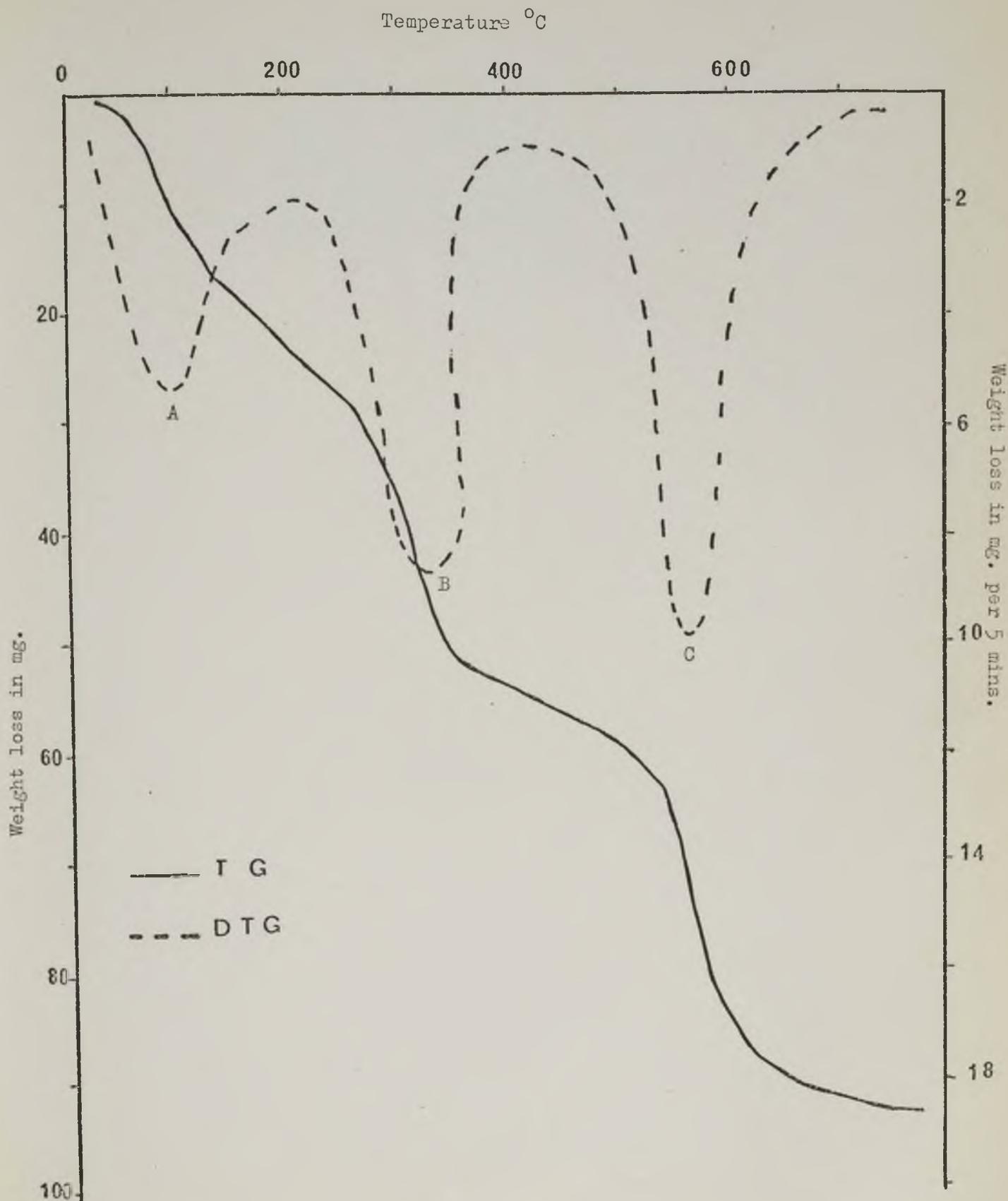


Fig. 6.9 Typical T.G. and D.T.G. curves from Nova Floresta soil.
(Specimen 45 $<2\mu\text{m}$ fraction from concretion Depth 1.2m)

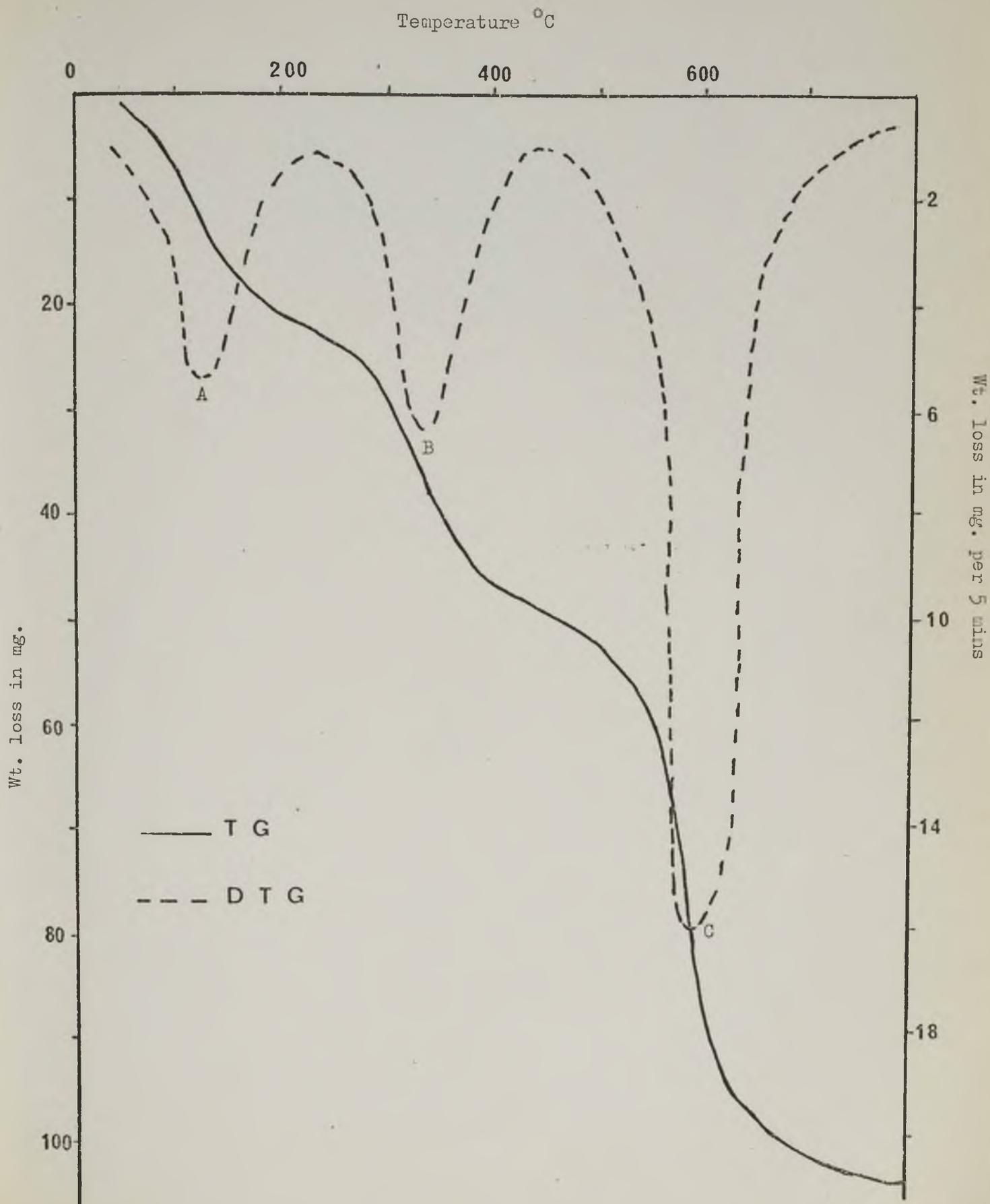


Fig. 6.10 Typical T.G. and D.T.G. curves from Sape soil.
(Specimen 55 - <2mm fraction from concretion, Depth 2m)

Buoyancy correction was made by heating the sample in air. It should, however, be noted that after these corrections, the error in the experiment still was about 8%, as such results given here cannot be giving anything but a semi-quantitative interpretation.

6.2.4 Chemical Composition:

Chemical analyses, using wet chemical methods, were carried out on all the subfractions from the concretions as well as the fines of the three localities.

The percentages of silica, iron and alumina as the oxides are given in Table 6.6 - 6.8. Also given are the moisture losses on heating to 110°C and 1000°C.

Generally the chemical composition of the subfractions of the fines are in the following order:

silica > alumina > iron

and for the subfractions in the concretions

iron > silica > alumina

The descriptions of the variations in the chemical compositions of the sub-fractions from the different levels in the profiles of Cuite, Nova-Floresta and Sape are now considered.

6.2.4.1 Cuite and Nova-Floresta - Plot of the chemical composition versus the average diameter of the sub-fractions show that the content of silica is a maximum at the silt size range, for the fines. However, for the subfraction from the concretions little change is observed with increase in size and in one case a notable decrease occurs. These plots are shown in Figures 6.11, 6.13 and the values are presented in Tables 6.6 and 6.7. One would normally expect the highest silica content to correspond to the materials in fine sand or coarse sand range since

TABLE 6.6

SILICA, ALUMINA AND IRON COMPOSITION OF CUITE SOIL

Speci- men	Side	Depth (m)	Size (mm)	Material	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	R [*]	S/A [*]	S/R [*]
10	+	1.00	9.5-2.0mm	Fines	35.0	32.4	18.4	3.86	3.17	1.51
11	+	1.00	2.0-76μm	Fines	47.5	15.2	22.2	3.13	3.64	2.53
12	+	1.00	76μm-2μm	Fines	46.4	10.2	27.4	3.32	2.88	2.32
13	+	1.00	2μm	Fines	32.5	7.5	32.1	3.62	1.72	1.50
14	+	1.00	76μm-2μm	Conc.	26.0	49.7	16.0	4.67	2.76	0.93
15	+	1.00	2μm	Conc.	39.0	11.0	28.5	3.48	2.33	1.87
20	+	2.00	9.5-2.0mm	Fines	40.0	11.0	30.0	3.63	2.43	1.84
21	+	2.00	2.0-76μm	Fines	40.0	8.0	32.0	3.64	2.12	1.83
22	+	2.00	76μm-2μm	Fines	44.0	9.0	28.0	3.31	2.67	2.22
23	+	2.00	2μm	Fines	21.0	6.3	19.0	2.26	1.88	1.55
24	+	2.00	76μm-2μm	Conc.	40.4	32.2	15.0	3.48	4.58	1.93
25	+	2.00	2μm	Conc.	34.0	10.7	28.0	3.41	2.07	1.66
30	++	2.00	9.5-2.0mm	Fines	36.0	25.8	20.2	3.59	3.03	1.67
31	++	2.00	2.0-76μm	Fines	43.0	15.4	24.6	3.38	2.97	2.12
32	++	2.00	76μm-2μm	Fines	47.0	11.8	23.5	3.04	3.40	2.57
33	++	2.00	2μm	Fines	30.4	8.6	26.0	3.08	1.99	1.64
34	++	2.00	76μm-2μm	Conc.	32.0	44.3	12.8	4.02	4.27	1.33
35	++	2.00	2μm	Conc.	30.4	28.4	23.2	4.05	2.23	1.25

+ Left Side, ++ Right Side.

* S - Molecular composition of silica

A - Molecular composition of alumina

R - Molecular composition of alumina + total iron oxide

TABLE 6.7

SILICA, ALUMINA & IRON COMPOSITION OF NOVA FLORESTA SOIL

Specimen	Depth (m)	Size (mm)	Material	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	R	S/A	S/R
40	1.20	9.5-2.0mm	Fines	34.0	15.0	28.5	3.73	2.03	1.52
41	1.20	2.0-76 μ m	Fines	40.0	8.5	32.5	3.72	2.09	1.79
42	1.20	76 μ m-2 μ m	Fines	42.0	10.0	28.0	3.37	2.55	2.08
43	1.20	2 μ m	Fines	28.0	7.0	27.0	3.08	1.76	1.51
44	1.20	76 μ m-2 μ m	Conc.	20.0	52.7	12.3	4.50	2.76	0.74
45	1.20	2 μ m	Conc.	22.0	29.5	21.7	4.00	1.72	0.92

S - Molecular composition of silica

A - Molecular composition of alumina

R - Molecular composition of alumina + total iron oxide

TABLE 6.8

SILICA, ALUMINA AND IRON COMPOSITION OF SAPE SOIL

Specimen	Depth (m)	Size (mm)	Material	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	R	S/A	S/R
50	2.00	9.5-20mm	Fines	No material		2mm			
51	2.00	2.0-76 μ m	Fines	44.0	8.0	20.5	2.51	3.64	2.92
52	2.00	76 μ m-2 μ m	Fines	44.0	9.0	28.0	3.31	2.67	2.21
53	2.00	2 μ m	Fines	32.0	8.3	28.7	3.33	1.90	1.60
54	2.00	76 μ m-2 μ m	Conc.	20.0	45.5	18.0	4.61	1.89	0.72
55	2.00	2 μ m	Conc.	25.8	31.0	21.0	4.00	2.09	1.08
60	3.00	9.5-2.0mm		No material		2mm			
61	3.00	2.0-76 μ m	Fines	43.5	3.5	33.1	3.46	2.24	2.10
62	3.00	76 μ m-2 μ m	Fines	41.8	5.0	33.1	3.56	2.15	1.96
63	3.00	2 μ m	Fines	36.0	5.0	32.1	3.46	1.90	1.73
64	3.00	76 μ m-2 μ m	Conc.	20.0	42.0	20.0	4.59	1.70	0.73
65	3.00	2 μ m	Conc.	24.0	28.6	20.3	3.77	2.01	1.06
70	4.00	9.5-2.0mm	Fines	No material					
71	4.00	2.0-76 μ m	Fines	45.0	10.2	26.0	3.18	2.95	2.36
72	4.00	76 μ m-2 μ m	Fines	36.8	14.8	28.5	3.72	2.20	1.65
73	4.00	2 μ m	Fines	35.1	10.7	30.7	3.68	1.94	1.59
74	4.00	76 μ m-2 μ m	Conc.	20.0	48.5	15.2	4.52	2.24	0.74
75	4.00	2 μ m	Conc.	19.0	39.7	18.0	4.25	1.80	0.75

S - Molecular composition of silica

A - Molecular composition of alumina

R - Molecular composition of alumina + total iron oxide

visual as well as the microscopical examination shows the presence of quartz as the main identifiable single mineral in these ranges.

Figures 6.11, 6.13 present the plots of alumina content versus average sizes. This shows that the amount of alumina is little affected by increase in sizes and is fairly constant for the fines. However, for the sub-fractions from the concretions there is a notable decrease on going from clay to silt sizes. The constancy of the alumina content for the fines for all size ranges indicates that the aggregation process only involves the cementation of clay minerals to produce materials of larger sizes and that no segregation of alumina is involved. ✕

The plot of iron content versus sizes is shown in Figures 6.11, 6.13. It can be easily seen that for the smaller sizes the iron content is constant. It does not change until large sizes are reached and then the values can be fairly high. An increase is recorded on going from clay to silt sizes of the specimen from the concretions, and the values are higher than for the fines. It appears the segregation and concentration of iron are only significant in the concretions.

The plot of the silica sesquioxide ratios versus sizes are shown in Figures 6.12, 6.14. This ratio has been linked to the degree of laterisation and has been employed in the classification of red soils. The silica-sesquioxide ratio is lower than 2 which is the parameter used in classification. It is lower than this value for all the subfractions of the concretion. However, for the fines, it attains values higher than this and reaches a maximum at silt sizes.

The silica-alumina ratios versus size are given in Figures 6.12, 6.14 and show that the clay sizes of the fine materials generally have values below or close to 2 and that from the concretions generally have values considerably higher than 2. The silt size ranges from both subfractions also have values notably higher than 2.

— Fines
 - - - Concretions
 △ ▲ 1.0m depth - Left side
 □ ▣ 2.0m " " "
 ○ ● 2.0m " Right side

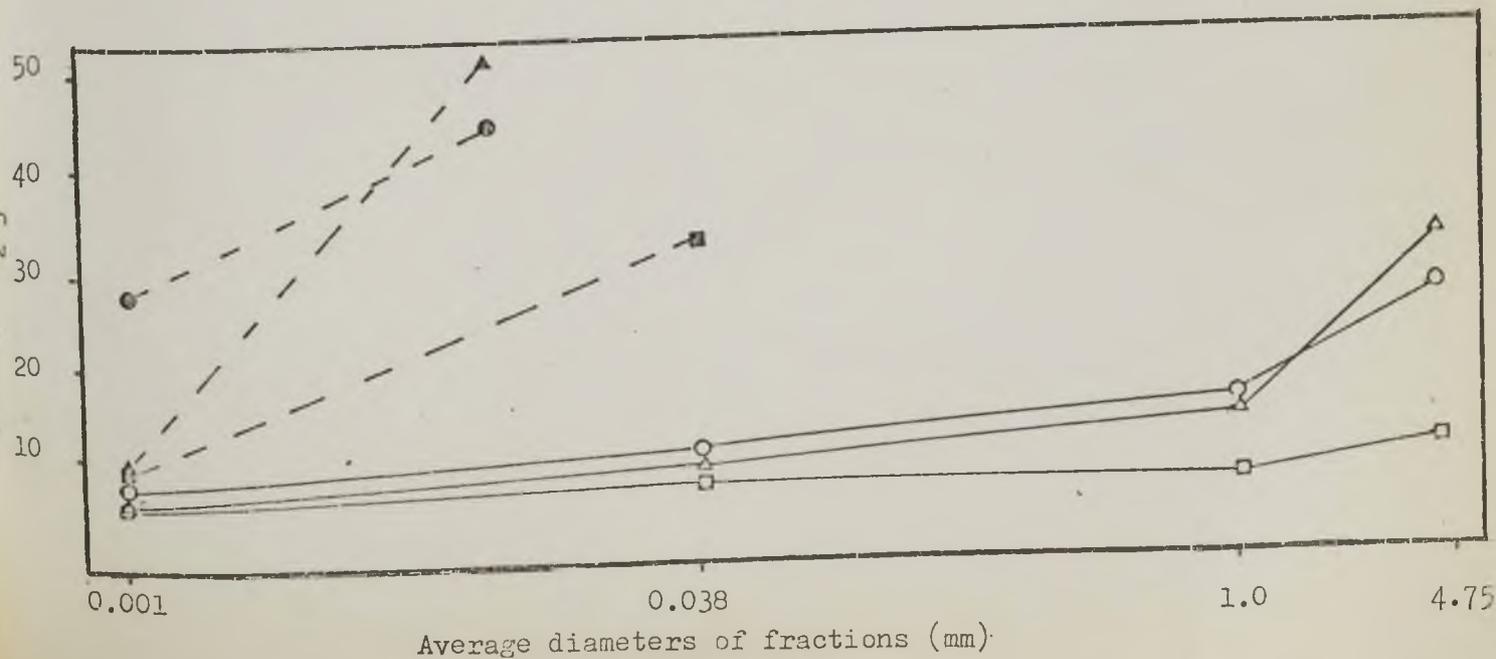
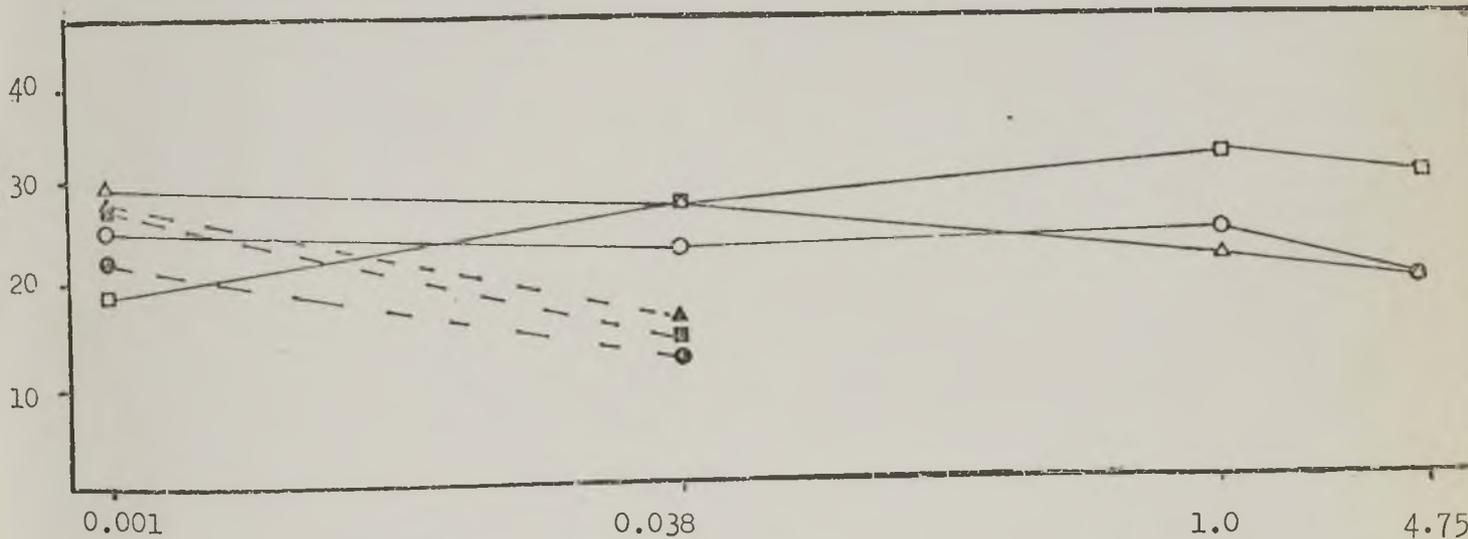
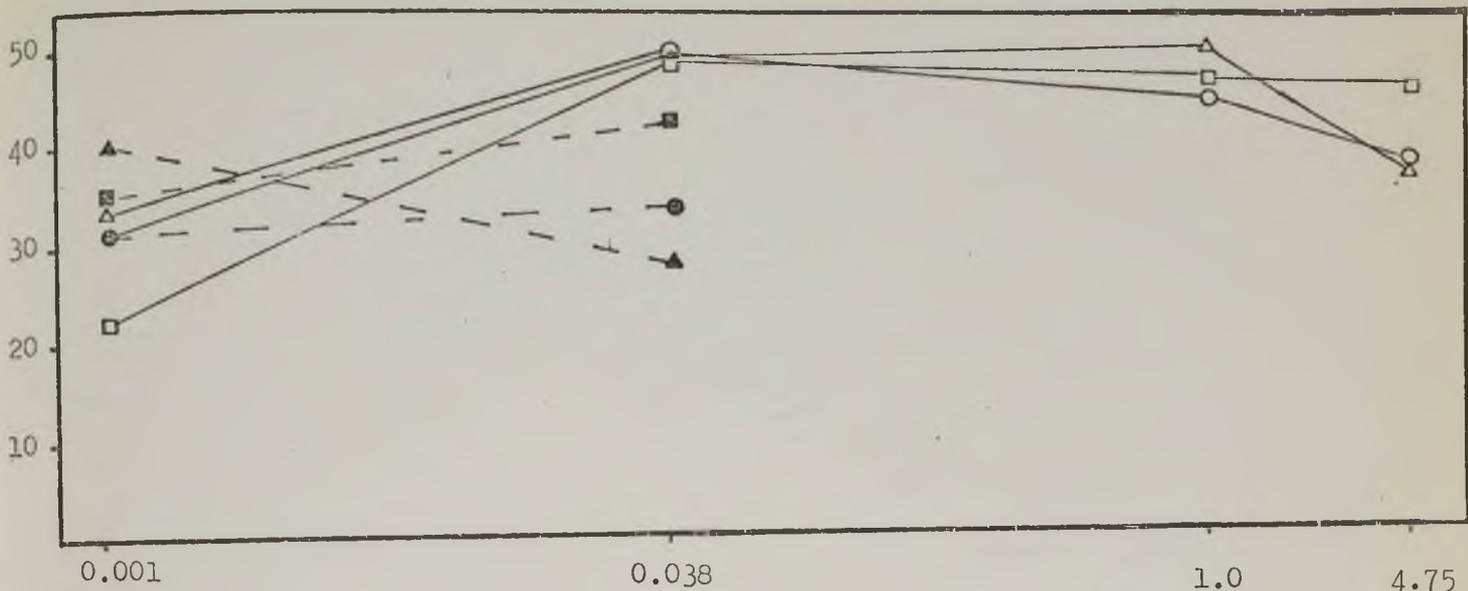


Fig. 6.11 Relation between chemical compositions and average sizes of the different fractions of Cuite soil.

▲ ▲ 1.0m depth - Left side
 □ ■ 2.0m " " "
 ○ ● 2.0m " Right side

— Fines
 - - - Concretions

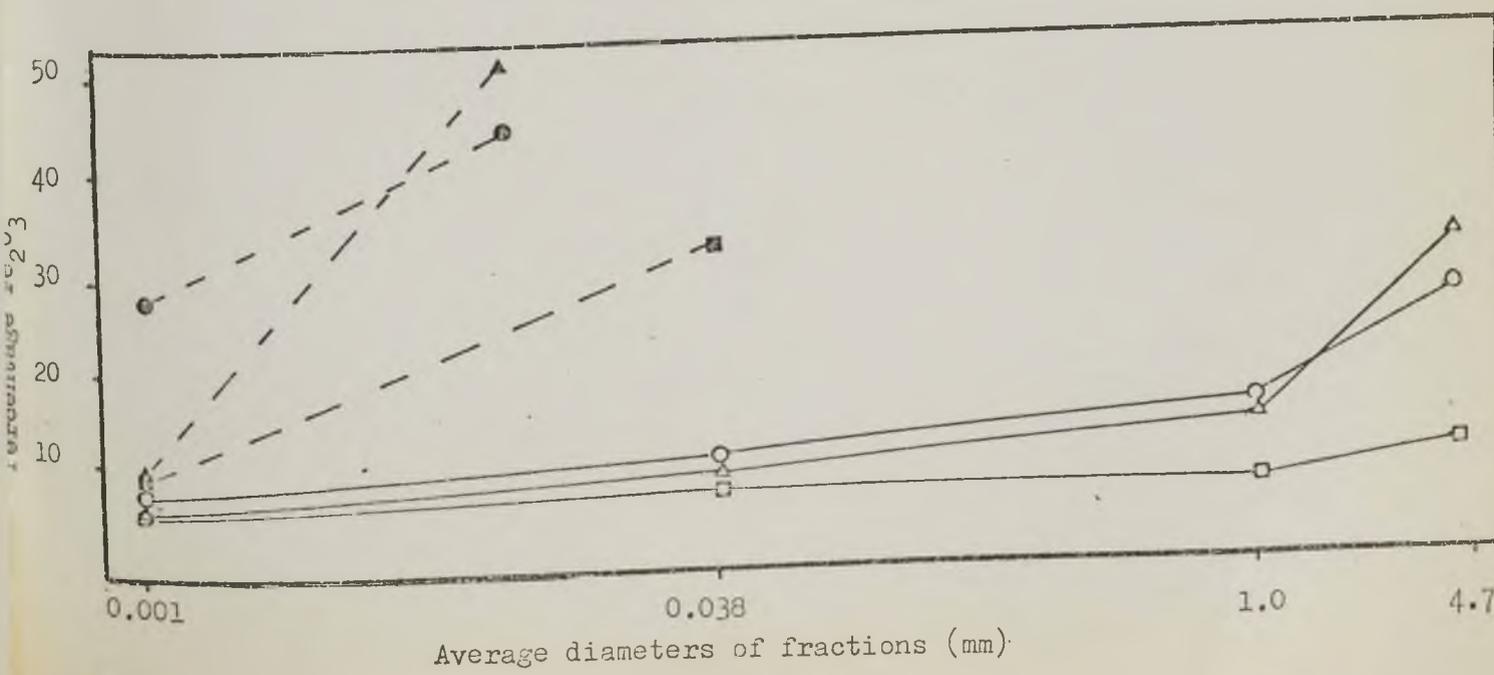
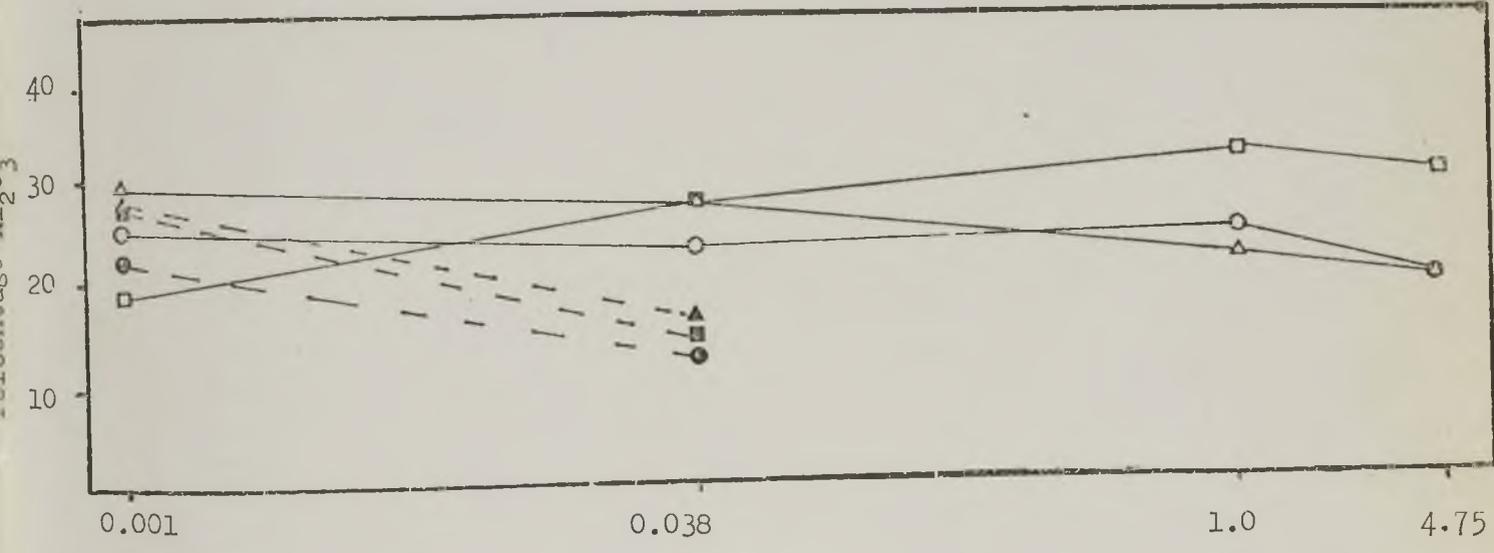
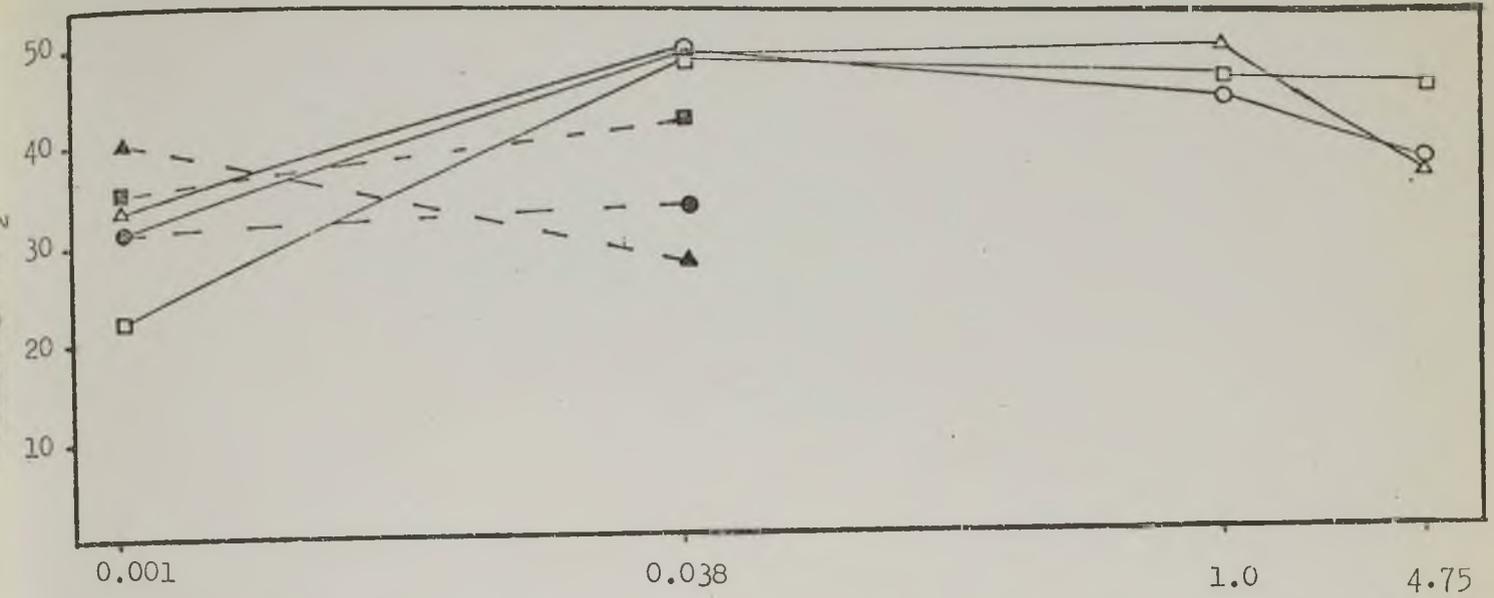


Fig. 6.11 Relation between chemical compositions and average sizes of the different fractions of Cuite soil.

▲ ▲ 1.0m depth Left side
 □ □ 2.0m " " "
 ○ ○ 2.0m " Right "
 — Fines
 - - Concretions

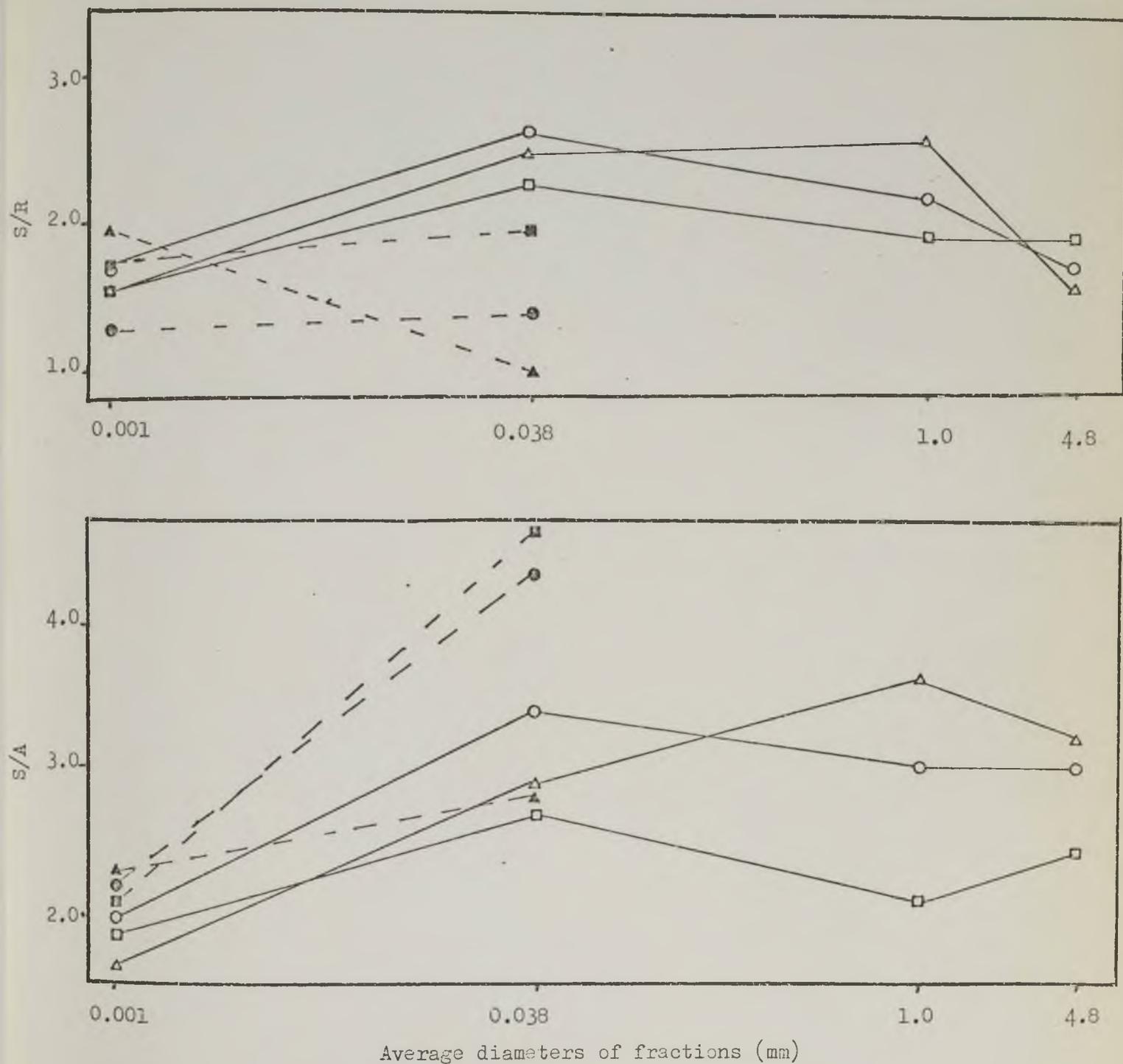


Fig. 6.12 The relation between silica-alumina/silica-sesquioxide ratios and average sizes of the different fractions of Cuite soil

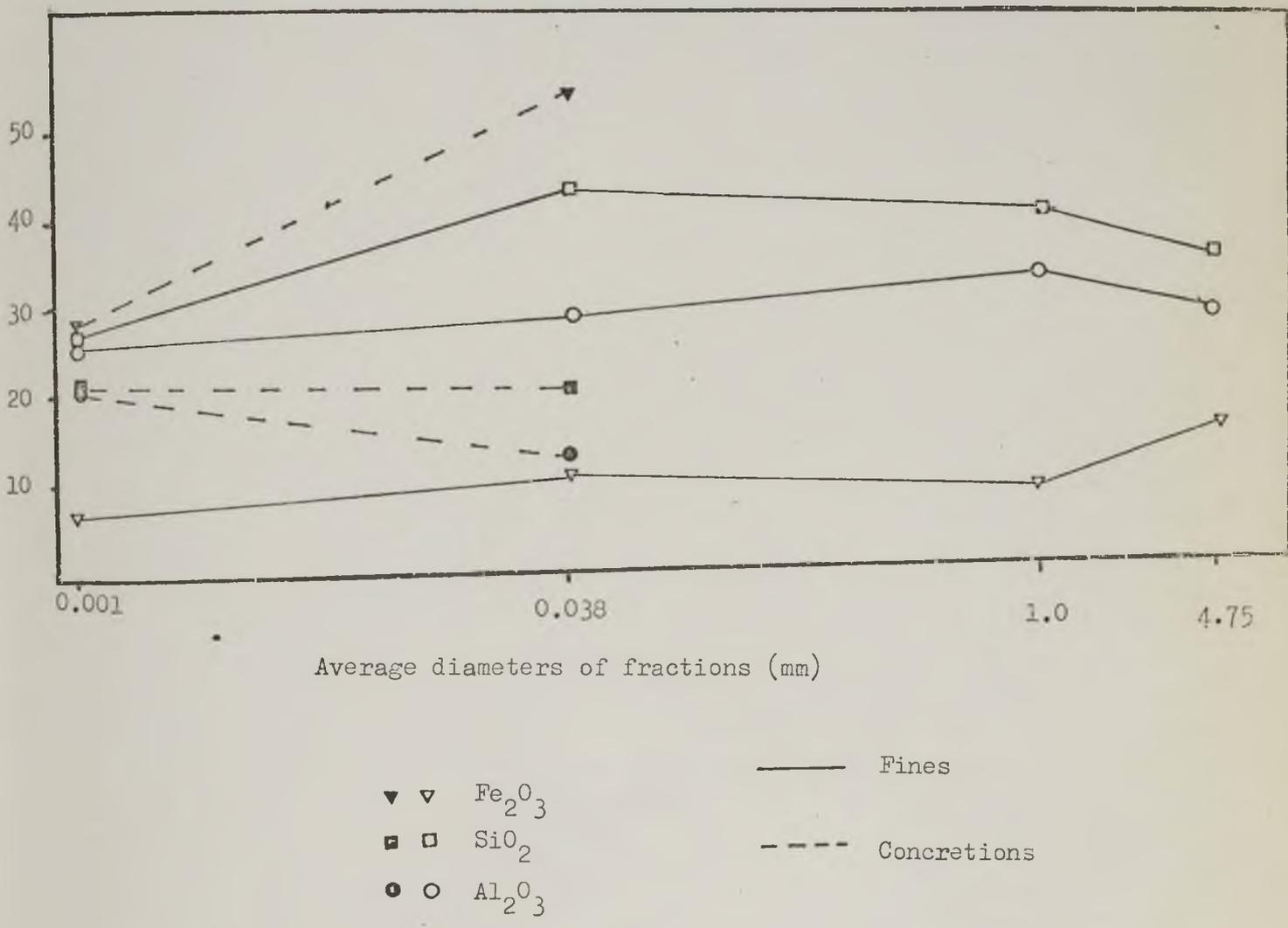


Fig. 6.13 Relation between chemical composition and average sizes of the different fractions of Nova Floresta soils.

— Fines
 - - - Concretions

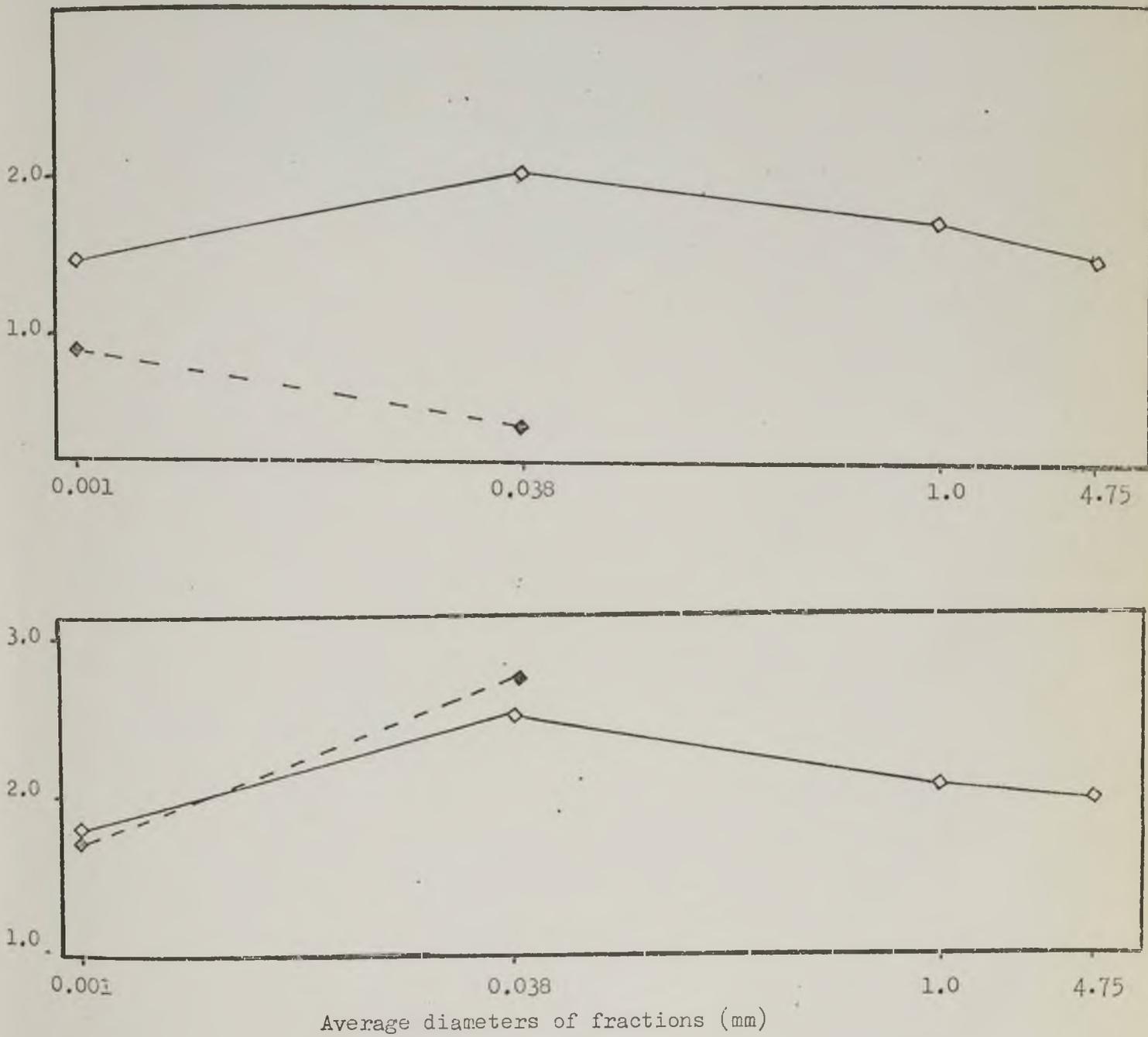


Fig. 6.14 The relation between silica-alumina/silica-sesquioxide ratios and average sizes of the different fractions of Nova Floresta soil.

6.2.4.2 Sape - The softer nature of the material from Sape is revealed by the fact that no material remains above 2 mm after washing of the soil with distilled water. However, the plot of chemical compositions versus average sizes shown in Figures 6.15 and 6.16 demonstrates that the variations are similar to those of materials from Cuite and Nova-Floresta. It is also worth mentioning that the highest values for the iron content are recorded from samples from the deepest levels.

6.3 Discussion:

6.3.1 Form and Distribution:

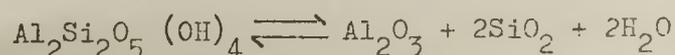
In this section, a discussion of the form of the different minerals identified will be carried out, as well as some comments on the different varieties present and the distribution of these in the fractions studied, which is intended to lead to an examination of the possible path to the genesis of these soils.

6.3.1.1 Kaolinite:

Since no form of uncombined alumina was identified by any of the mineralogical methods employed (X-ray, DTA and TG) the alumina composition of the chemical analysis can be safely attributed to the alumina in the kaolinite which was identified in the mineralogical analysis.

The kaolinite identified is a disordered one, which gives a slight shift in the (001) reflections in the X-ray diffraction, giving a d-spacing of 7.21\AA as compared to the 7.14\AA of well crystallised kaolinite (value from the ASTM powder diffraction data file 1965). This was identified in both the concretions and the fines.

The weight loss on dehydroxylation which occurs between $420\text{--}600^\circ\text{C}$ in normal thermogravimetric analysis is usually used in the quantitative determination of kaolinite. This loss of weight is used to determine the amount of kaolinite based on the stoichiometry of the transformation:



△ ▲ 2m
□ ■ 3m
○ ● 4m

— Fines
- - - Concretions

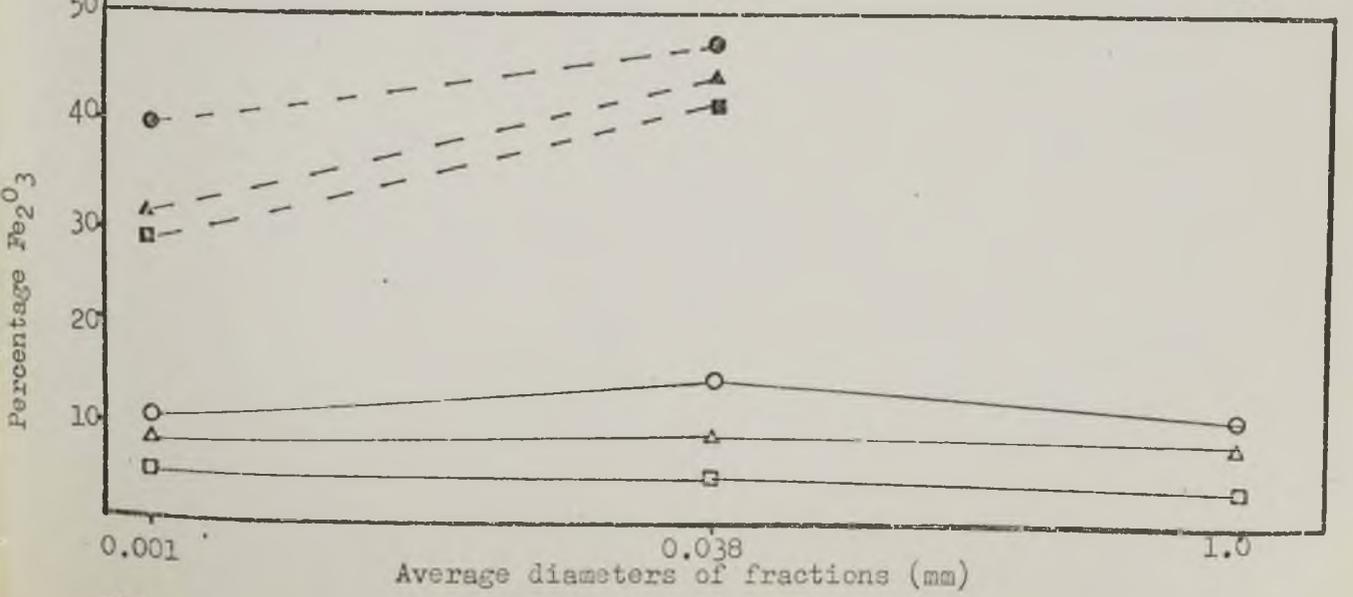
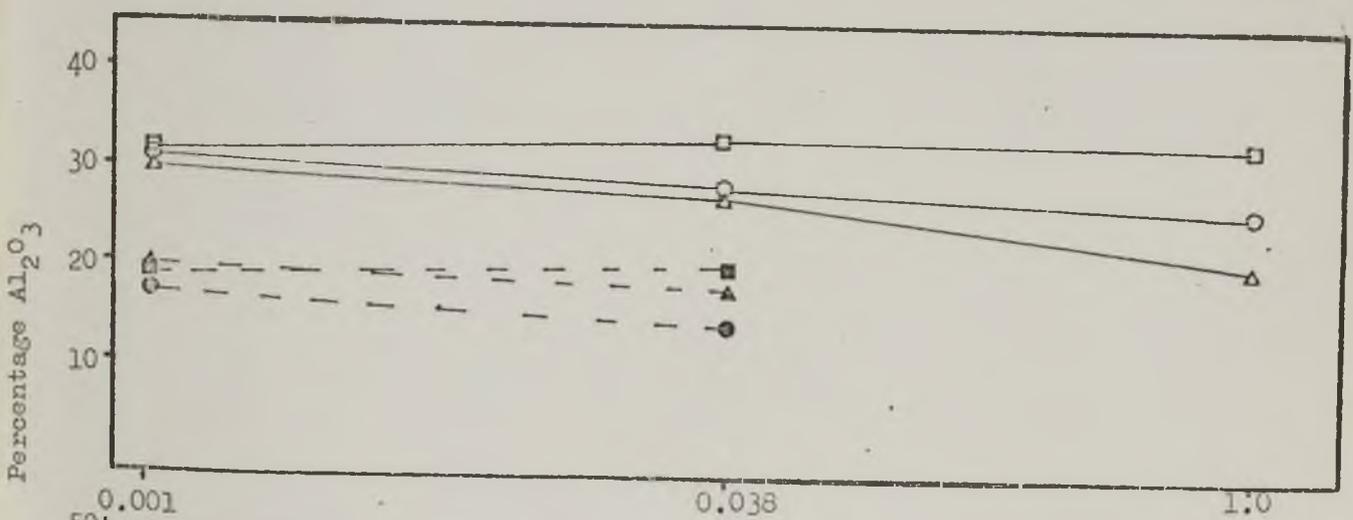
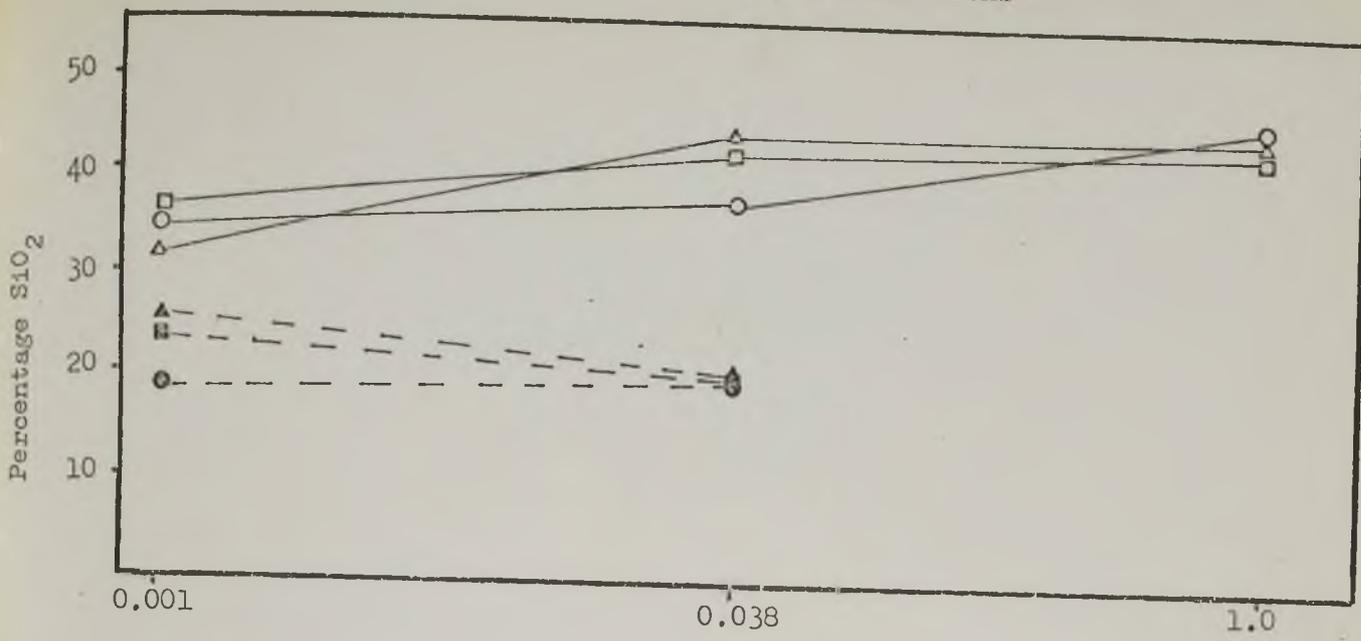


Fig. 6.15 The relation between chemical composition and average sizes of the different fractions of Sape soil.

— Fines △ ▲ 2m
 - - - Concretions □ ■ 3m
 ○ ● 4m

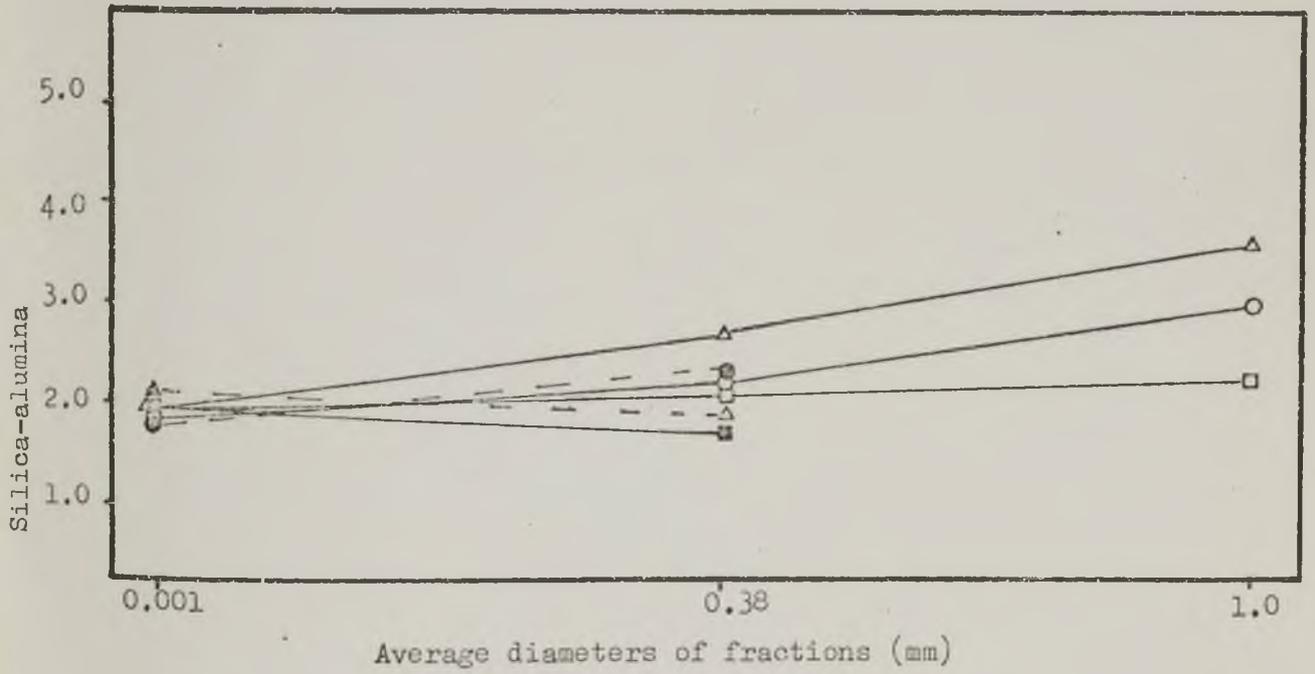
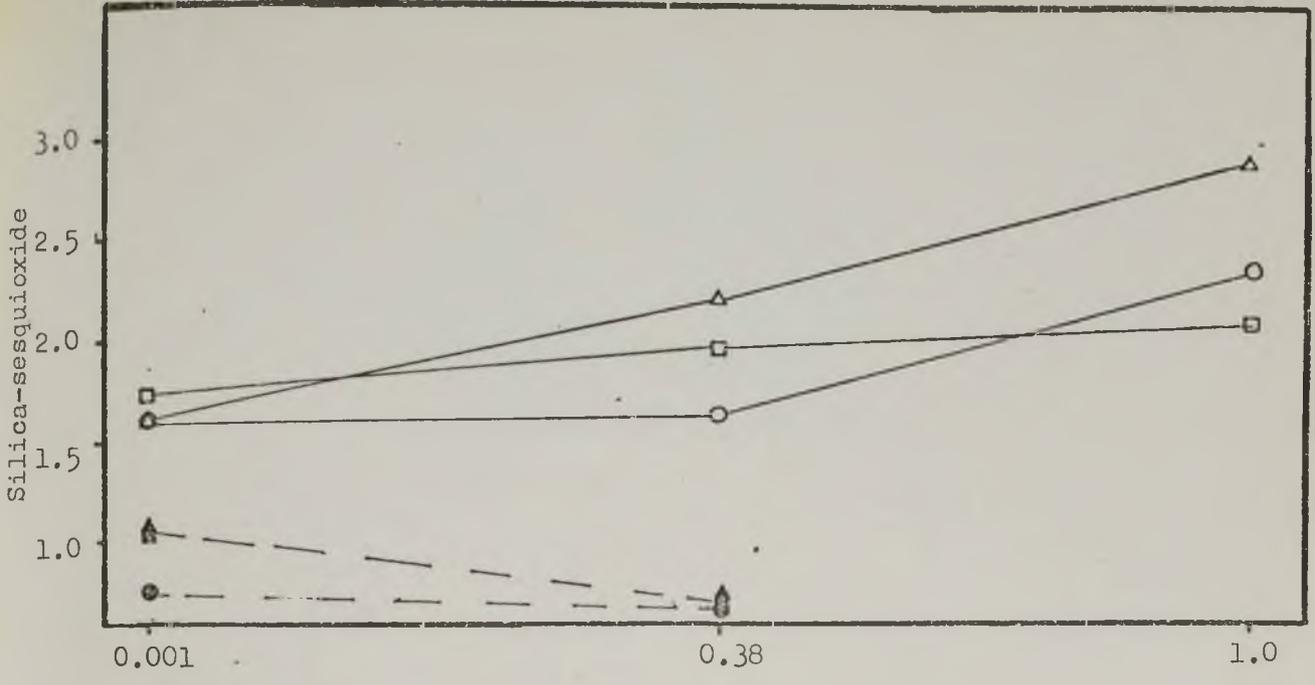


Fig. 6.16 The relation between silica sesquioxide/silica-alumina ratios and average sizes of the different fractions of Sape soil.

Another method of arriving at the same information is to use the alumina composition based on the chemical analysis, using the same conversion equation. When the amount of kaolinite obtained by the two different methods of analysis are compared, the values obtained from thermogravimetric analysis are found to be lower than those obtained from data of chemical composition.

Eliminating the effects of experimental errors, two possible explanations are available, both relating to the validity of the loss of weight values obtained from the thermogravimetric analysis:

- (a) Lower (OH) than expected - the possibility that the kaolinite encountered in these soils, in its disordered form, contain less (OH) groups within its crystal structure than the formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ of the unit crystal. Since kaolinite is basically composed of an alternation of gibbsite $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ and silica SiO_2 , deviations in the stoichiometry of the gibbsite layer (i.e. one molecule of H_2O to one of Al_2O_3) could affect the quantity of water held which will be reflected in the thermal behaviour of the mineral.
- (b) Not all the (OH) is given off - the possibility that the kaolinite encountered in these soils do not give off all the (OH) water during dehydroxylation between $420\text{-}600^\circ\text{C}$ and that a higher temperature will have to be reached to lose all.

The possibility of the retention of some water in the kaolinite heated beyond the dehydroxylation in normal thermal analysis has been the basis of Brindley and Hunter's (p.117) explanation of the small exothermic peak at 930°C obtained in the DTA analysis of kaolinite. However, Holdridge and Vaughan (1957) have shown that this peak can be explained by thermodynamical considerations without necessarily assuming retention of water in the dehydration product. Furthermore, Grim and Bradley (1948) and Grim (1968, p.300) have shown that the loss of (OH)

water in poorly crystallised kaolinite is accompanied by a fairly complete loss of structure, but that in well crystallised kaolinite, some structural remnants persist along with some water. Other observations based on the ease of acid attack of kaolinite and the replacement of OH by F^- when it is treated with HF,

have shown that poorly crystallised kaolinite loses its OH readily. The incompatibility of the chemical and mineralogical data is therefore more likely to be due to (a) the first possibility.

The possibility that the mineral contained less (OH) is in line with previous observations by Keeling (1962) based on the determinations of combined water and specific surface of a number of kaolinites and halloysites. He found that a continuous series can be suggested between well crystallised kaolinite through a poorly crystallised kaolinite to halloysite representing a decrease in the combined water to surface area ratio. He suggested that the ratio can be used to characterise a mineral and therefore aid in identification procedures. The decrease in the ratio which can be due to a decrease in amount of combined water or an increase in surface area can however be made to be a function of only the amount of combined water by using kaolinite of the same sizes. A decrease in the amount of combined water present in poorly crystallised kaolinite is therefore more probable.

A thermogravimetric analysis was run on a relatively pure kaolinite (API standard kaolinite No. 4) for comparison. The combined water was found to be 12.4% which is still short of the 13.96% expected from theoretical considerations of kaolinite, but higher than 11%, generally found for these soils. This result raises the question - "How pure is well-crystallised kaolinite; what is the influence of substitution in the octahedral site on the amount of combined water and what is the relationship between crystallinity and the amount of combined water?". The further development of the concept, i.e. the relationship between

the quantity of combined water and form of the kaolinite and answers to the above questions was hindered by the non-availability of apparatus and materials for this part of the research were carried out in North-East Brazil, where no API standards were readily available. Therefore, no strong conclusions can be arrived at, as to the link between the amount of water held and crystallinity. A good statistical analysis of the combined H_2O , for various types of pure kaolinites and disordered ones and a comparison with other suggested methods for determination of the degree of crystallinity of kaolinite is required. Hinckley (1963) suggested the use of the (002) reflection in X-ray powder diffraction of kaolinite for the determination of its degree of crystallinity.

6.3.1.2 Other forms of alumina:

It is interesting that no other form of alumina i.e. gibbsite, boehmite was identified, even in the slightest trace in any of the three soils. Many previous investigations have recorded the presence of some form of bauxite e.g. Alexander and Cady (1962), Sivarajasingham et al. (1962), and Maignien (1966, p.22). Hamilton (1964) contends that bauxites result from decomposition of kaolinites and can be considered as advanced stage of laterisation. This may probably be true for the Sape soil, but the Cuite and Nova Floresta are much older deposits (see Ch.5) and the absence of uncombined alumina in these is most likely to be due to some other factor and this has been ascribed to the presence of uncombined silica which would react with any uncombined alumina.

Kaolinite is found in all fractions of both nodules and fines from all levels. It is evident that some form of cementation is present to elevate the clays of $2\mu m$ sizes to silt and even sand sizes (comment on this can be found in Chapter 8).

Chemical composition and mineralogical considerations show that alumina is concentrated in the silt-sizes and its concentration parallels

continuous aggregation, therefore one can say that the accumulation of sesquioxides which leads to continuous aggregation promotes accumulation of alumina in non-clay sizes. The cementation of aggregated grains of clay are discussed in Chapter 8.

6.3.1.3 Silica:

Quartz was easily identified as the form of silica in all the fractions above clay size, from both concretions and fines by X-ray diffraction. The exothermic peak at 576°C that occurs in the DTA analysis which signifies the transformation of α - quartz to its high temperature equivalent β - quartz was not found to be very prominent. Since this transformation occurs within the major endothermic peak of the loss of structural water, i.e. dehydroxylation of kaolinite, it will only be detectable if high proportions of it exist in the specimen.

The ratio of silica to alumina in molecular terms has been used to classify these soils. Observation of the molecular ratio of silica to alumina in the clay fraction of the aggregated nodules show that this value is generally in excess of 2. The expected value of kaolinite based on the chemical formula $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ is 2 since kaolinite is a 1 : 1 mineral, i.e. the ratio of gibbsite layers to silica layers in the lattice structure is 1 : 1. The existence of an amount of excess silica, over the amount required by stoichiometry, to combine with alumina to form kaolinite points to:

- (a) The presence of ionic substitution for aluminium by other elements, usually iron, (Grim, 1968, p.68), in the gibbsite structure of the lattice leading to the depletion of aluminium and therefore a high value of silica to alumina.
- (b) The presence of anauxite - a silica rich kaolinite which has been identified (though controversial in various locations, Carroll

1970, p.13).

- (c) The presence of forms of silica non-detectable by X-rays, i.e. amorphous silica - conclusions easily arrived at by deduction.

The incorporation of iron into the gibbsite structure within the lattice of kaolinite is possible, due to the charge similarity between aluminium ion Al^{3+} and Ferric ion Fe^{3+} , and the fact that the clay was probably formed in an iron rich environment. However, the incorporation to such an extent as to give a Si/Al ratio of 2.33 in some of the clay fractions of the nodule is difficult to explain. Moreover, some workers have contended that the ionic substitution by iron into the lattice of kaolinite takes place within the silica rather than the gibbsite, (Follett 1965), a condition which will produce low values of Si/Al.

The possibility of the existence of anauxite seems less plausible than that of ionic substitution. It was first identified also on the basis of the molecular ratio of silica to alumina, it is now believed that much of such material is a discrete mixture of kaolinite and silica (Grim 1968, p.68).

The existence of some form of mobility of silica in the past is evidenced by growth of neoformations of silica found by microscopical investigations of indurated parts of the three soils. Obviously, if this is identified, the possibility of amorphous silica existing in the soils is not too far out.

The existence of amorphous silica partially explains the absence of all forms of aluminium oxide hydrates in all the levels of the three soils. Amorphous silica which is reactive will not normally co-exist with oxides of aluminium, unless in a metastable condition, otherwise reaction between the two will occur which will lead to the formation of a clay mineral, most likely kaolinite, under these conditions.

The possible existence of amorphous silica is reinforced by the

considerable reactivity of these soils to lime, in a study of soil stabilisation by the addition of lime. Ferro (1976) carried out a complimentary study on these soils and recorded a considerable reactivity to lime, far in excess of that normally expected from a kaolinitic soil. Kaolinitic soils are normally considered non-reactive with reference to lime stabilisation (Gray 1972, p.264).

Quartz is identified in all the fractions from both fines and concretions, it was found to be more concentrated in the concretions than the fines. The identification of quartz neoformations in discrete bodies show that many of the quartz might not be inherited (Chapter 3). Amorphous silica is 'identified' in the clay fractions of the nodules. Its existence in other fractions is possible but no assertions can be made since its non-detectability by X-rays and the coincidence of its loss of water peak with other forms of amorphous material, (e.g. iron oxides identified in some specimen) as well as its presence in conjunction with quartz at these sizes, makes it difficult for any strong conclusions.

The presence of amorphous silica in the discrete bodies indicated by the high silica to alumina ratio found for these soils when compared to those of pure kaolinite means that some form of mobility of silica existed sometime in the past. This mobility has been indirectly confirmed by the presence of authigenic quartz in the optical microscope. This was found to occur as growth from a deposition in a void mostly as an implantation without any reaction with the soil grain (Plates 12, 13).

6.3.1.4 Iron oxides:

The forms of iron oxides identified in the soils are haematite, goethite and some forms which are amorphous to X-rays, detectable only by thermal analysis. Haematite and goethite exist as finely crystallised material individual within voids or as coating and impregnations on clay.

The iron oxides are found in various proportions (see Tables 6.6-6.8) in all the fractions but most prominent in the concretion where they constitute up to 40%. The individual particle size in soils is less than $2\mu\text{m}$ (Oades, 1962), and presence in large fractions is due to cementation.

Amorphous iron oxides exist in finely divided state and impart considerable influence on the formation of these soils. They have enormous specific surface and can go into solution more readily than the well-crystallised minerals, they can also move as sols or gels to the places where they develop the crystalline framework (Alexander and Cady, 1962). Their presence is confirmed by thermal analysis and by physical observation of the soil using the microscope. They have also been synthesised in the laboratory, but few indications are present as to the relationship between natural soil materials and synthesised ones. Synthesis usually involves the precipitation of the hydroxides from a solution of an iron salt by the addition of a base. This can be achieved in a number of ways which lead to a number of states of hydration for ferric and ferrous ions,

Methods include gaseous ammonia, aqueous ammonia, ammonia and ethanol, ammonia and hydrazine, iodide and iodate, mercury chloramide. These methods are given a fairly detailed treatment by Duval (1963, pp.323-325).

It is recognised that various equilibrium conditions can be established between many forms of iron-oxide hydrates (Norton, 1973). Little attempt was therefore made to distinguish the various possible forms. The possible paths of transformation of an ageing gel has been mentioned and the identified amorphous material could be on any of the paths described (Figure 3.1). Valetton (1972, p.48) concluded that "...no stable stage is reached at room temperature, even after years." (pp.47-48).

Haematite and goethite are found in all the fractions of the three soils. They exist as coatings in channel walls and voids and as light coatings (staining) on cemented bodies, seen in the microscope. A greater percentage is probably formed from the crystallisation and transformation of less crystalline ones which are evident in Plates 16-19. The impregnation of clays and continuity of dehydration result in highly cemented materials. Their role as the originator of hardening in these soils has been suggested by Sivarejasingham et al. (1962). The continuity of the crystalline phase of iron oxide has been described in Chapter 8, where they can be seen to provide a link between different types of cemented particles of various shapes, sizes and juxtaposition.

Amorphous iron-oxides exist mostly as coating of pores and channels where they have been identified as rounded features of pre-haematite developing from amorphous mass of coating (Malomo, 1974 and Plate 16). They are probably present as stainings on the clay mineral particle, but their nature on the molecular level is not easily identifiable due to their small sizes and the considerable amount of cementation of particles found in these soils.

6.3.2 Formation of the deposits:

Analysis of the geology, physiography, profile and chemical characteristics allows some speculation as to the mode of these deposits.

The correlation between Cuite (1) and Cuite (3) is easily shown by the plots of the contents of alumina, iron and silica versus the particle sizes. The similarity in behaviour is significant and the different absolute values can easily be ascribed to the statistical differences one would expect from samples from the same horizon in two different localities which is an expression of the inherent heterogeneity of this horizon

A mention has been made of the accumulation of iron and silica in the profile (see result and observation) and a distinction has been attempted between accumulation in the horizon in general and that within the concretionary elements of the horizon on the basis of chemistry, however, a further separation of level of accumulation of ferruginous material from siliceous ones can be made.

The level from which the sample of Nova Flersta was taken can also be correlated with Cuite (1) and (3) on the basis of chemical behaviour particularly the trends in chemical composition when compared with sizes. From Figure 6.11 it appears that the main level of iron accumulation occurs here and main level of silica accumulation occurs at a different level, i.e. corresponding to Cuite (2). It is, however, possible that the difference between Cuite (2) and the others, i.e. Cuite (1), (3) and Nova Floresta is due to either silica or iron accumulation being particularly significant at certain levels and not to each one having its own preference level of accumulation.

The progressive transformation of smaller sized particles into larger ones, i.e. formation of concretions has been found, in this case, to be accompanied by concentration of iron in the larger sizes. The environment necessary for the deposition of iron has been elaborated in Chapter 2 (see Genesis). The condition necessary for the accumulation of iron has been described by Grant (1974, p.iv-31.4 - iv.31.6), who states that "...iron is mobilised in solution in the ferrous state from ferruginous parent material and moves as such until oxidising conditions are encountered. Under oxidising conditions ferrous iron oxidises into the ferric state which is insoluble in water. If the oxidation is rapid, all the iron is immediately transformed to the ferric state. Iron then precipitates as a hydrated ferric oxide gel", while that necessary for the accumulation of silica has also been described by him towards which

he mentioned that "...silica may be mobilised in solution by water (up to the limits of its solubility) and be transported in solution. It can be re-deposited only under dehydrating conditions. Silica deposits as a colloidal gel (silica gel) which, on further dehydration, forms the amorphous indefinite hydrate of silica, opal".

It is known that for iron to accumulate, in addition to the oxidation - reduction (Eh) conditions mentioned by Grant, there are other factors that exert considerable influence on the deposition of iron which has been mentioned in Chapter 2 (see Genesis). Important among these is the pH which should not be below 3.5 otherwise ferric iron will be unstable and can easily go into the ferrous state. It is also known that the deposition of silica is enhanced by non-alkaline conditions for, although silica is appreciably soluble at all pH values, it is least soluble under acid conditions.

As progressive accumulation and laterisation of Cuite and Nova Floresta deposits involves silica and iron concentration, one can suggest that the conditions of formation of these deposits entailed reasonably acidic, oxidising and dehydrating environments.

The theory of Campbell (1917) explains the formation of the Cuite and Nova Floresta soil. The accumulation of iron and silica being due to the movement of ground waters. The accumulation can be achieved in a tropical climate where there is an alternation of wetting and drying. There is a capillary fringe above the water table in the ground which represents a transition between saturated zones below and aerated zones above. With changes in the level of the water table due to seasonal variation in climate the capillary fringe moves up and down and defines a region of fluctuation. This region of fluctuation is referred to as the "zone of intermittent saturation".

During the wet season, the water table is raised above the zone of

intermittent saturation and reduction takes place below the table. Iron and silica are mobilised in solution and are carried to the surface. If there is a dry season immediately following the wet season the water table drops leaving behind a moisture rich zone containing the original components of the ground water which then suffers oxidation due to exposure to soil atmosphere and dehydration as a result of the heat of the prevailing conditions. These components are thus precipitated.

The transformation of the precipitated material into more stable forms is enhanced by the longevity of the dry period. The existence of such a long, continuously dry phase some time in the past is indicated by the presence of a hard cap, in some places, on the occurrence at Nova Floresta. Presumably the same cap has been removed, by denudation, for the occurrence at Cuite which is evidenced by the physiography of the region, the Cuite area being the most elevated within an area of about 1000 sq.km. The extent of the erosion on top of the occurrence is, however, not very considerable, since the upper horizon of the Cuite, is, in many ways, (as proved with chemical, mineralogy and particle size analysis) similar to the horizon immediately below the cap in the Nova Floresta deposits. The presence of these deposits at the top of an elevated region and the absence of it in the surrounding regions of lower elevation indicates, as already mentioned, the removal of material and also suggests that the adjoining removed areas may have been source regions for accumulation elsewhere.

The profile at Sape-Mari does not appear to have any direct relationship with the ones at Cuite and Nova Floresta. This is evident from profile characteristics (Figures 5.4-5.6) as well as consideration of the chemical nature (Figures 6.11-6.14).

The Sape deposit is found on top of the geologic formation Grupo do Barreira series, which is a younger tertiary series than the Serra dos

Martins of the Cuite/Nova Floresta.

The description of the profile which has already been given points to the colluvial nature for at least part of the components making up the profile. The rounded, abraded gravels which are found up to the second layer described in Figure 5.4, validates this assertion. It is also important to note that the deposit occurs in the lower part of the slope of a wide valley, obviously rejuvenated, which may form part of a formerly larger physiographic system.

From the foregoing one can suggest that the formation of the Sape deposit is due to a process akin to the one described by Hamilton 1964, for what he called low-level red soils.

This process involves the impregnation of slope deposits by mobile material which moves readily from upland areas to slopes and low-lying areas where they soak into the colluvial deposits. Ageing and crystallisation of the material within the deposits will be responsible for the production of red soil. The silica accumulation which was observable in the Cuite and Nova Floresta is less obvious as can be seen in Figures 6.11, 6.13.

The precipitation of silica which would have been removed from the top is less likely to occur unless dehydrating conditions are encountered. This is less probable at the lower part of the slope since this position is likely to be subjected to the influence of phreatic waters more than other positions.

CHAPTER 7

STRENGTH BEHAVIOUR OF THE SOILS

7.1 Methods of Testing:

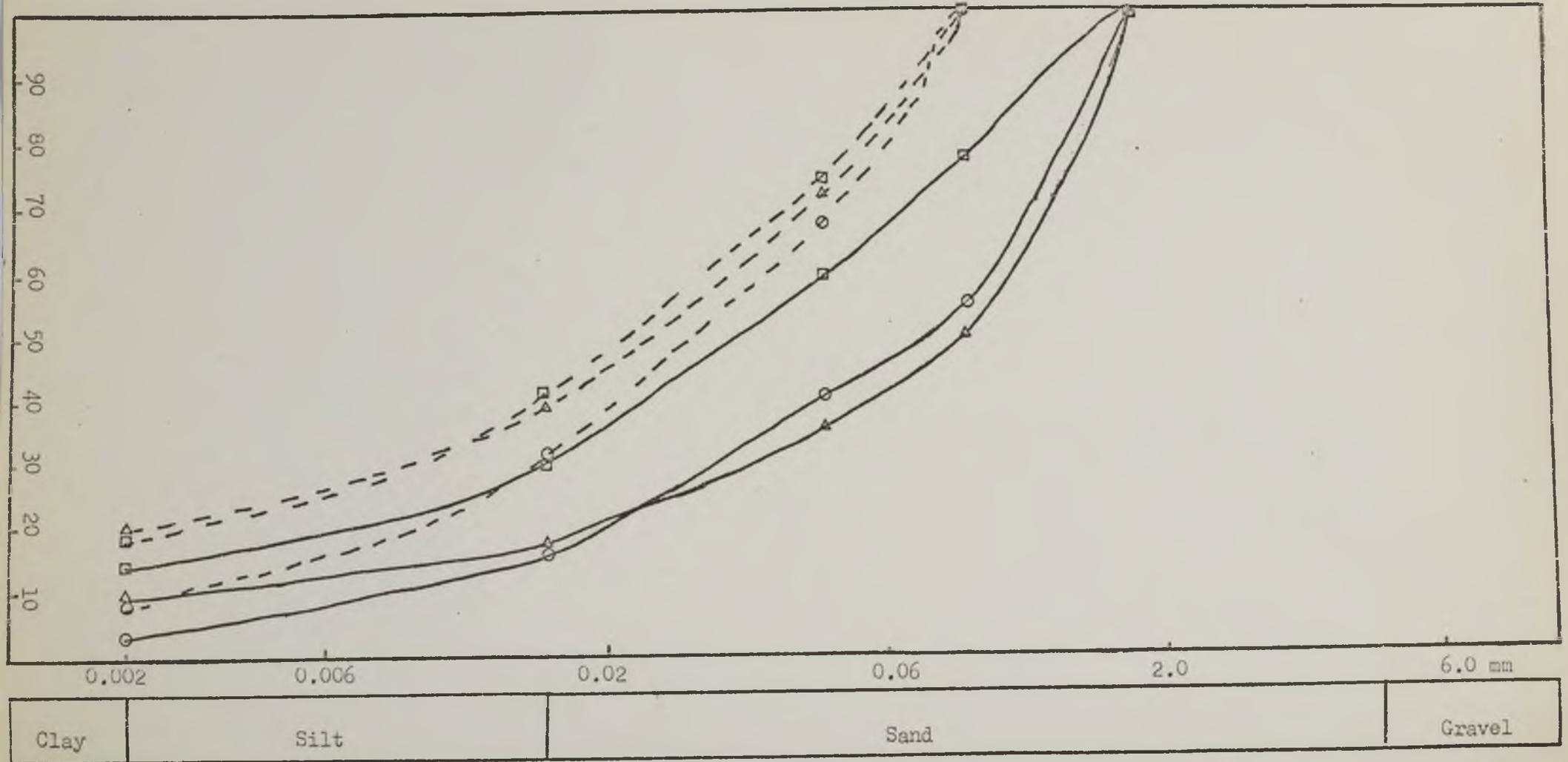
It was necessary to reduce the maximum size of the soil grains to 2 mm, due to the test procedures that were adopted in the investigation of the strength behaviour. The pulverised soils were sieved using B.S. standard sieve No.8 (2 mm) see Figure 7.1(a). The material passing was used to carry out the determination of the shear strength and compressibility.

Moisture-density curves were first produced following B.S. 1377 (1967) standard compaction test 12: The curves for the Cuite, Nova Floresta and Sape soils are shown in Figure 7.1(b). The results of Atterberg limits determined, using the same standard, is shown in Table 7.1

For the determination of the compressibility and shear strength properties, particular moisture contents were chosen from the density curves. Attempts were made to arrive at dry densities, corresponding to the chosen moisture contents, for the specimen produced by dynamic or static compaction.

7.1.1 Compressibility:

Determination of the compressibility characteristics was carried out for one soil (Sape). The procedure followed is as found in B.S.1377 (1967), with slight modification in the dimensions since S.I. units were used. Since loose materials were involved, specimens were produced by static compaction of moistened material in a cylindrical split mould, designed to facilitate the ease of loading of the specimen into the oedometer ring and to reduce the disturbance of the specimen after



○ Sape
 △ Cuite
 □ Nova Floresta

———— Whole
 - - - - After manipulation

Fig. 7.1(a) Particle size distribution of pulverised red soils as used for the investigation of strength characteristics.

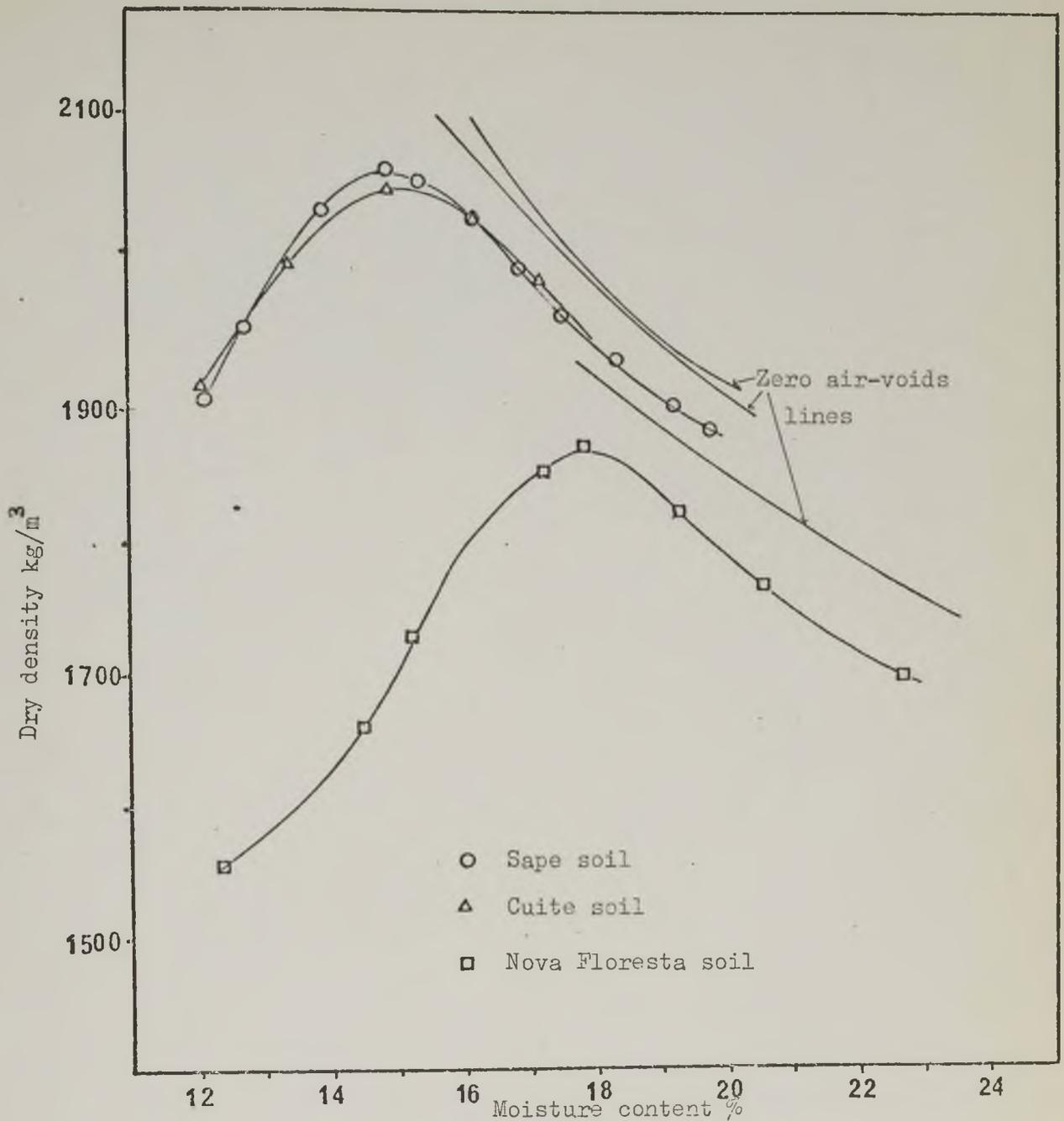


Fig. 7.1(b) Moisture density curve of the Sape, Cuite and Nova Floresta soils. The curves were obtained by dynamic compaction. Attempts were made to reach points on the curve by compacting specimens in the static mode. These specimens were used for the triaxial and compressibility tests.

TABLE 7.1

Atterberg limit values for Cuite Nova Floresta and Sape soils.

	Cuite	Nova Floresta	Sape
LL	22.00	27.10	34.00
PL	16.20	18.23	25.00
PI	5.80	8.87	9.00
Change in limits after drying at 60°C	LL - less than 3% PL - no change	LL - no change PL - no change	LL - less than 5% PL - no change

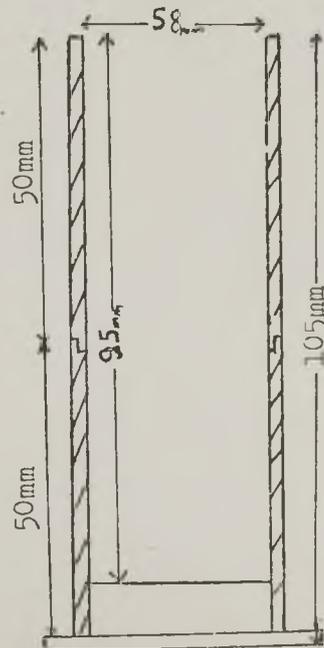
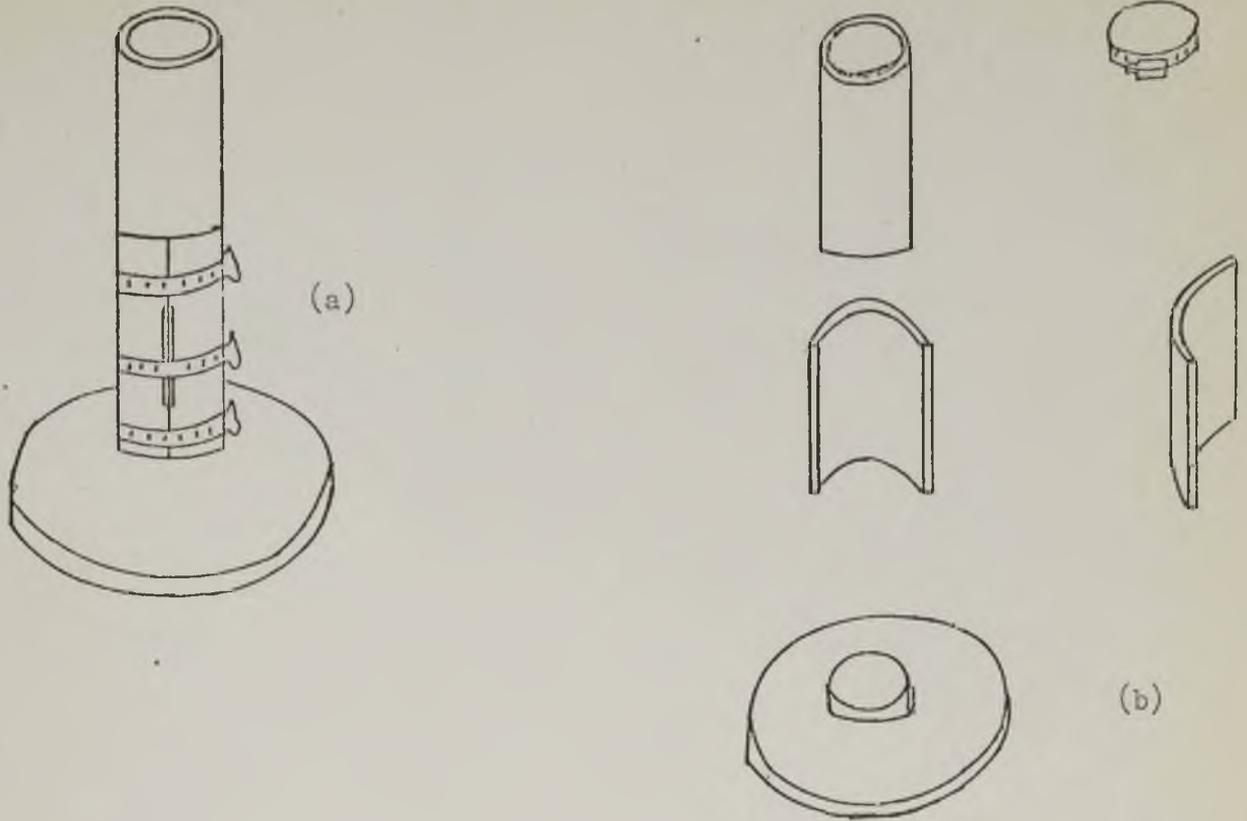


Fig. 7.2

Split-Mold designed for the preparation of compacted specimens.

Note: Dimension of mold element varied according to the size required for the triaxial and compressibility test;

(Mold dimensions as for specimen for the Oedometer tests)

compaction to a minimum.

Chosen moisture contents were the optimum, dry and the wet-side of the compaction curve. The set-up of the split mould is as shown in Figure 7.2(b). These were fitted as shown in Figure 7.2(a), well lubricated and easily separable.

Specimen preparations:

Soil samples were thoroughly homogenised with water at the required moisture content and a weighed quantity, (to give the dry density, corresponding to the moisture content on the compaction curves) was placed into the already mounted mould, which was loaded at a rate of 0.0406 mm/min, the depth of the penetration being measured by a graduated scale attached to the system. The loading was continued until a pre-determined point X was reached. The point X was so made that, the surface of the specimen being loaded was 5 mm clear of the top of the main mould, when this is reached. The stress developed at this point was then maintained for a period of two minutes. The main mould was then turned over, so that the formerly loaded top of the specimen, now lies on the base plate as shown in Figure 7.2. The specimen was then compacted further to produce the required height of the specimen, which was the exact height of the oedometer ring. The stress developed here was also maintained for two minutes. With the loading rod lifted and the base plate removed, the main mould was suspended by hand while the oedometer ring, on a flat plate, was placed under the mould and the circumference made to coincide. Since the internal diameter of the mould and the oedometer ring are equal, minimum further application of the loading rod will make the specimen slide into the ring.

Three specimens at the same moisture content were usually prepared, these were then ready for saturation, which was carried out using a vacuum desiccator designed by J.G. Cabrera.

A desiccator with a lid with a suitable opening was used, A Y - shaped glass tube fitted with a tough, flexible piece of rubber tube was connected to the inlet of the opening. One end of the Y tubing is connected by means of a rubber tube to a source of light vacuum and the other is connected to a source of water, both being fitted with tape.

A large filter paper is placed on top of a porcelain plate with perforations which rests on the angular section at the lower part of the desiccator. The specimens on porous stones are placed on the filter paper and another porous stone is placed on top of the specimen ring. The set-up was placed clear of the desiccator walls.

The water tap closed and air was first removed by opening of the tap to the vacuum source. A pump capable of reaching a maximum pressure of 72 cm was employed. The rate of suction was slow enough to avoid the disruption of structure. When a reasonable vacuum was achieved, the vacuum tap was closed and the water tap was slowly opened so that water was made to enter through the opening, slide through the wall of the lid to the wall of the desiccator and then onto the bottom. The desiccator was slowly filled with water until the level of the porcelain plate was reached. The filter paper takes up the water and passes it through the porous stones to the specimen. The production of low pressure in the pores of the specimen facilitated access by water. The water tap way is then closed and the lid was removed so that the space above the specimen was at atmospheric pressure and this also aids in the saturation of the specimen. Weighing of the specimen before and after saturation showed that all of the specimen can be easily saturated by this method.

It was decided to test the effect of suction on the specimen, i.e. if there was any disturbance of structure as a result of the method. This was done by using ordinary soaking, in the oedometer ring, for the saturation of the specimen. Up to three days were needed in certain

cases to achieve saturation, while comparison with the results of specimens saturated with the vacuum (which can be done in less than two hours) did not show any significant differences. All the tests were therefore carried out using suction as a means of saturation.

After saturation, the specimens in the ring were transferred to the loading system, water was added and the set-up was left for 24 hours, a small load being applied to prevent swelling. Loading was done in triplicate using 0.5, 1.0, 3.0, 6.0, 11, 21 and 42 kg weights in accordance with B.S.1377.

7.1.2 Direct Shear Strength:

The fraction below 2 mm was also used to carry out the shear box tests. Particular moisture contents were taken from the moisture - density curve obtained by dynamic compaction in a standard Proctor mould and samples were compacted at these moisture contents and under similar conditions so as to produce the exact points on the curve.

Compaction procedure - At each of the predetermined moulding water contents, a quantity of about 24 kg of soil was required which was adequate for 12 specimens. Mixing was accomplished in 6 kg batches to produce 2 compacted specimens. This was carried out in an electrical mixer with enough water to produce the desired value on the moisture density curve. Total mixing period was 3 minutes, with breaks being observed after each minute while the side of the mixer was scraped with a knife to allow thorough and total homogenisation. After compaction, specimens were kept in polythene bags and stored in a humid environment prior to cutting.

Specimens for the tests were cut from cylindrical compacted specimen using a thin knife in combination with a very thin saw. This effort proved to be very delicate since dense, fairly granular in cases, compacted soils were involved. The observed safeguard of preparing two

compacted specimens proved to be a wise decision, for the second specimen had to be fallen back to, for many of the cases, particularly to the dry side of optimum moisture content.

Testing procedure:

The testing procedure adopted is in accordance with the recommendation of T.W. Lambe (1952) - Soil Testing for Engineers.

7.1.3 Triaxial strength tests:

7.1.3.1 Sample preparation and treatment:

As already mentioned, the soils used for the triaxial tests were those from the Sape deposit. In this case, samples represent a fairly homogenised mixture of all the levels and sizes.

The materials were fairly dry in-situ, but were still subjected to further air drying before pulverising. Pulverising was carried out in a pulveriser equipped with mandibles to reduce the maximum size to 9.5 mm, the same as for the other tests. This arbitrary process was chosen for the convenience of the experimental procedure adopted as well as an attempt to simulate the conditions of use of the soil in construction.

Hoping to investigate the propagation of pore pressure with time and knowing that the size of the specimen has an influence on this, the dimensions adopted for the specimen were 76.2 mm (3") in height and 38.1 mm (1.5") in diameter. To prepare a specimen of this dimension, it was necessary to reduce the maximum size of the soil particles and the crushed material was therefore sieved to a maximum of 2 mm and was later dried in the oven at 60°C.

A mould, similar to the one employed in the determination of compressibility characteristics, was used to prepare the specimen. This consisted of a main cylinder of copper 106 mm in height and 38.1 mm in diameter which could be separated into two vertical halves fixed together

by a belt. The upper end of the cylinder has an internal groove into which another cylinder of the same internal diameter and 50.8 mm in length fits. The lower end rests on a base made of iron, that has at its centre a cylindrical relief of 12.7 mm in height and a diameter of 38.1 mm that fits into the main cylinder, all being easily dismantled.

7.1.3.2 Triaxial tests:

The specimens used for all the triaxial tests were compacted at different moisture contents. Specimens were compacted in static mode in the mould shown in Figure 6.2. Attempts were made to arrive at density on the moisture density curve produced by dynamic compactations shown in Figure 6.1.

The velocity used was 0.0228 mm per minute. An E.L.E. (Engineering Laboratory Equipment) equipment was used consisting of loading apparatus, triaxial cell and apparatus for pore pressure measurement.

The method adopted was that suggested by Bishop and Henkel (1962).

Undrained test:

Undrained tests were carried out with pore pressure measurement using compacted, partially saturated specimens. This type of test was carried out to investigate the propagation of pore pressure with time in compacted red soils. The pore pressure measurement was carried out at regular intervals after the application of the confining pressure σ_3 . The steadiness of the confining pressure was used in determining Skempton's pore pressure parameter B.

The deviator stress was only applied after a steady pore pressure was achieved.

Consolidated - Undrained Tests

These tests were carried out with saturated compacted specimens. Saturation was done by using back-pressure by the application of water-pressure 98.1 kN/m² at the base of the specimen and submitting at the

same time a vacuum pressure of 78.5 kN/m^2 at the top. The extent of saturation was measured by determination of Skempton's parameter B. The specimen was considered saturated when the parameter reached a value equal or greater than 0.95.

The tests were carried out with the measurement of pore pressure and the strain to determine change in Skempton's parameter A with pore pressure and the shear strength parameter in terms of effective stresses.

Drained Test

The drained tests carried out with compacted specimens. Three confining pressures (98.1 (1), 196.2(2) and 294.3 (3) kN/m^2 (kgf/cm^2)) were used.

7.2 Results:

7.2.1 Direct Shear:

The behaviour of the three soils, whose nature and mineralogical properties have been investigated, were studied under direct-shear conditions. Stress-volume change-strain behaviour were examined for materials compacted at three moisture contents on the moisture/density curve (this was obtained by compaction in a standard Proctor mould) viz. the dry-side of optimum, the optimum and the wet-side of optimum.

The limitations of this method in the investigation of strength have already been described by Terzaghi (1944) and these were fully realised before the initiation of the tests. However, because of the simplicity of the tests, both in operation and interpretation, (Taylor, 1948, p.330) and the fact that it is repeatable on sets of the same material produced under similar conditions (Nivargikar 1971), it was considered useful enough as a means of portraying the behaviour of these soils, particularly when under different normal loads.

7.2.1.1 Stress-Strain:

The stress strain curves for the Cuite soil compacted on the dry side for three different normal loads are shown in Figure 7.3. At low normal stresses the behaviour approximates to that generally ascribed to natural overconsolidated clays or dense sand, in that the shear stress increases with strain, reaching a peak (at values less than 3%) thereafter decreasing and later attaining a steady value with increasing strains corresponding to the residual shear strength.

At higher normal stresses, the shearing stress also increases rapidly at low values of strain. On reaching values of about 5% (usually equal or higher than the value of strain corresponding to the peak stress for specimens loaded at lower normal pressure) the rate increase changes, and therefore the slope of the stress-strain plot, with the shear-stress now increasing rapidly without reaching a maximum during the duration of the tests. In certain cases up to 20% strain - a behaviour pattern usually associated with normal consolidated clays.

The same general behaviour is shown by the Sape and Nova Floresta soils. Plots of stress-strain curves of materials compacted at other moisture contents are shown in Figures 7.4, 7.5.

A similar trend is observable, i.e. one of change from over-consolidated clay behaviour, to that of a normally consolidated clay with increase in normal stresses.

Figure 7.6 shows the stress-strain curves of the Sape soil compacted at three different moisture contents, 15%, 19% and 21% and sheared under the same normal load. The same trend predominates. A behaviour analogous to that of an over-consolidated clay at low moisture of compaction and to that of a normally consolidated clay at high moisture of compaction.

The foregoing thus appear to show that these materials can change in behaviour depending on the level of stress and moisture content.

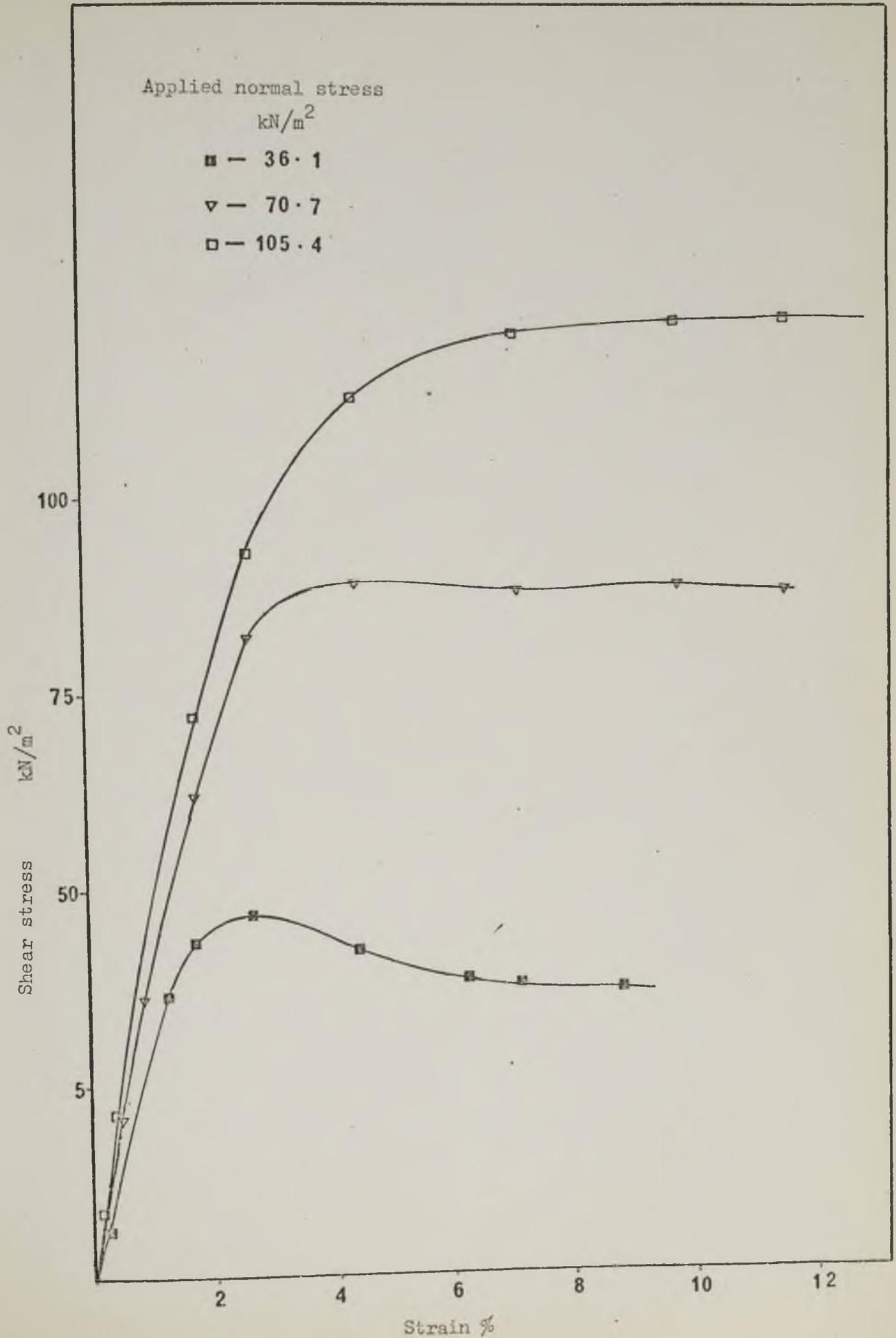


Fig. 7.3 Stress-strain curves for Cuite soil in the direct shear test at three normal stresses (moisture content 12%)

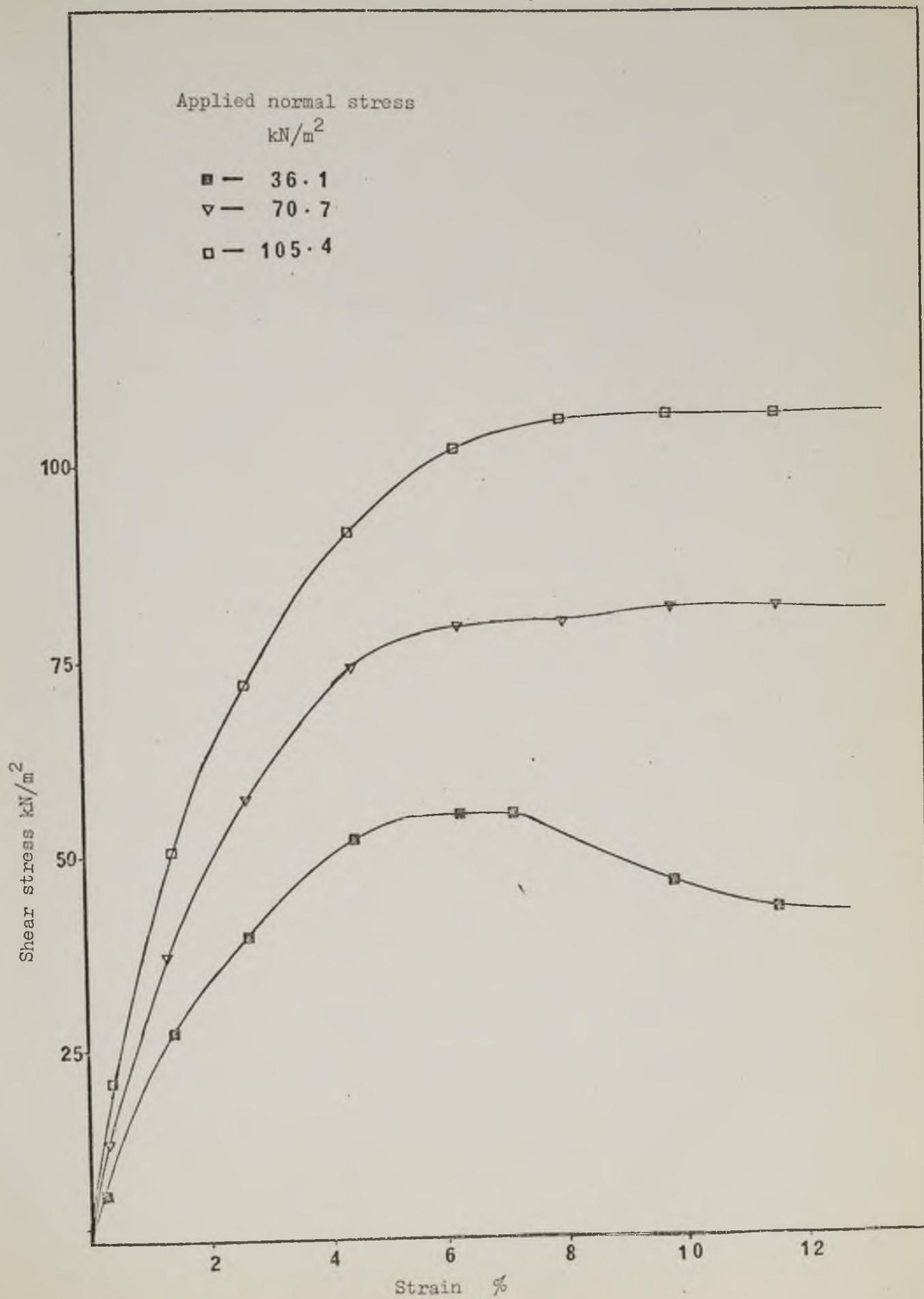


Fig. 7.4 Stress-strain curves for Sape soil in the direct shear test at three normal stresses (moisture content 21%)

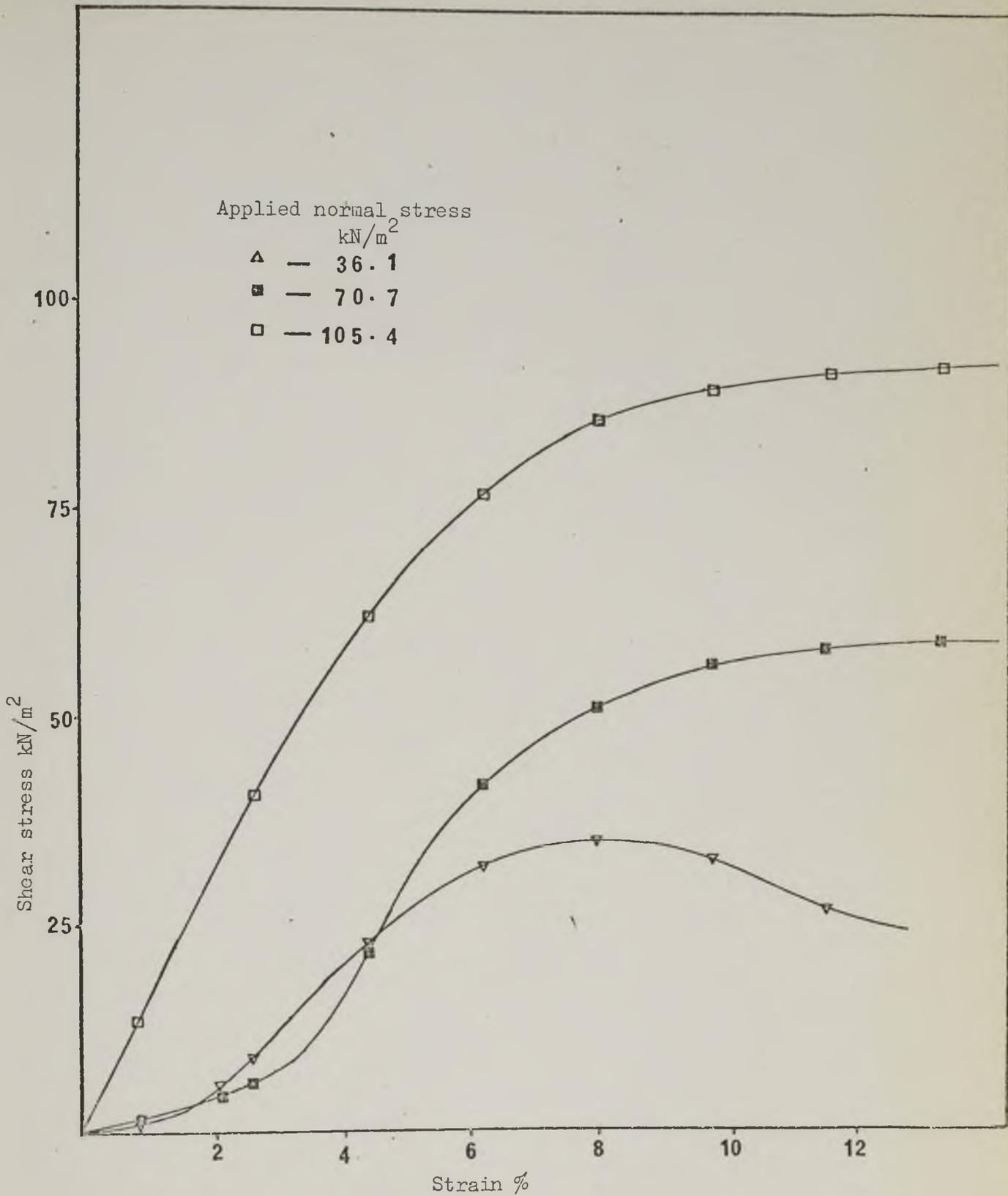


Fig. 7.5 Stress-strain curves for Nova Floresta soil in the direct shear tests at three normal stress (moisture content 14.6%)

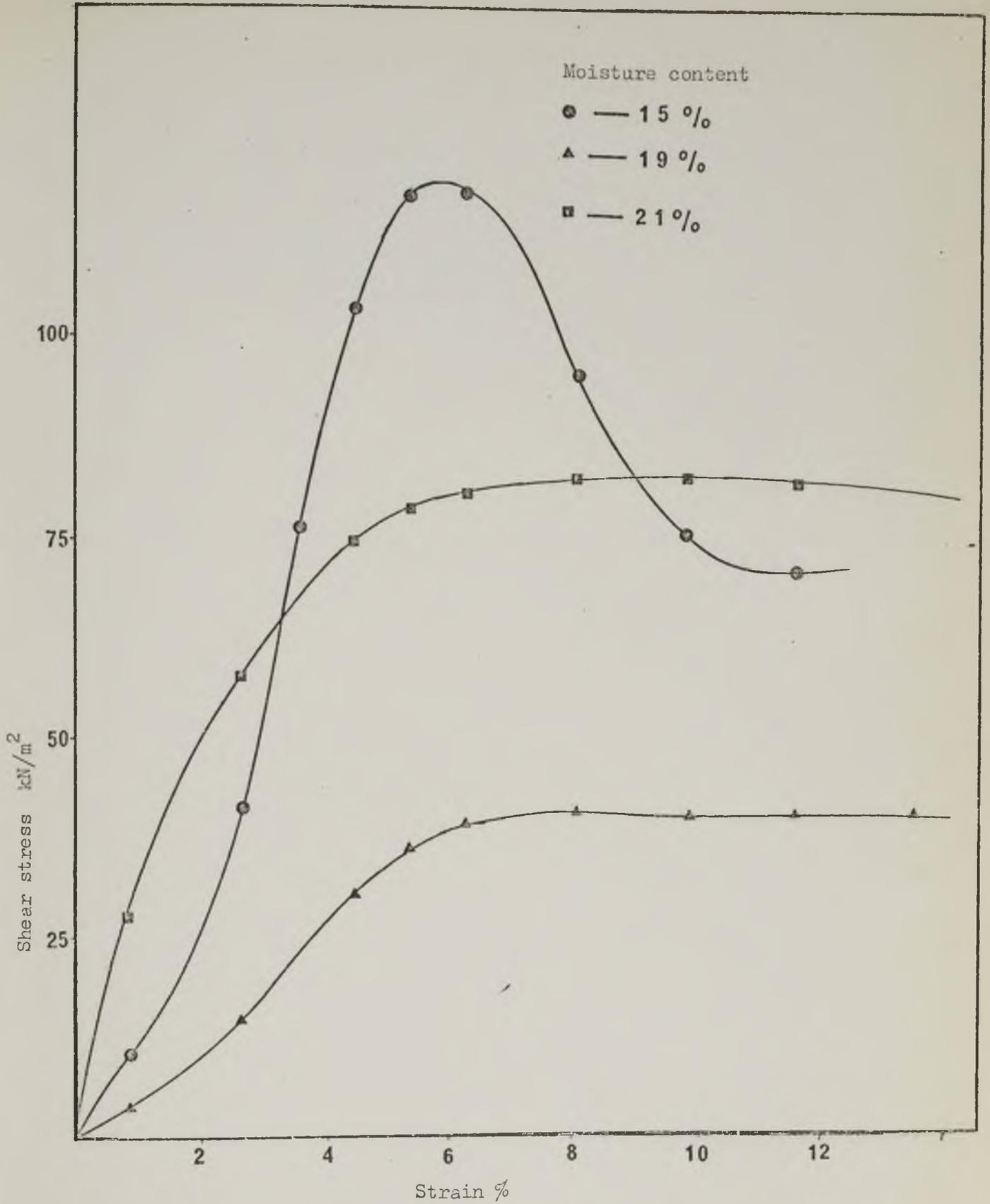


Fig. 7.6 Stress-strain curves for Sape soil at three different moisture contents applying the same initial normal stress (Applied normal stress = 70.7 kN/m^2)

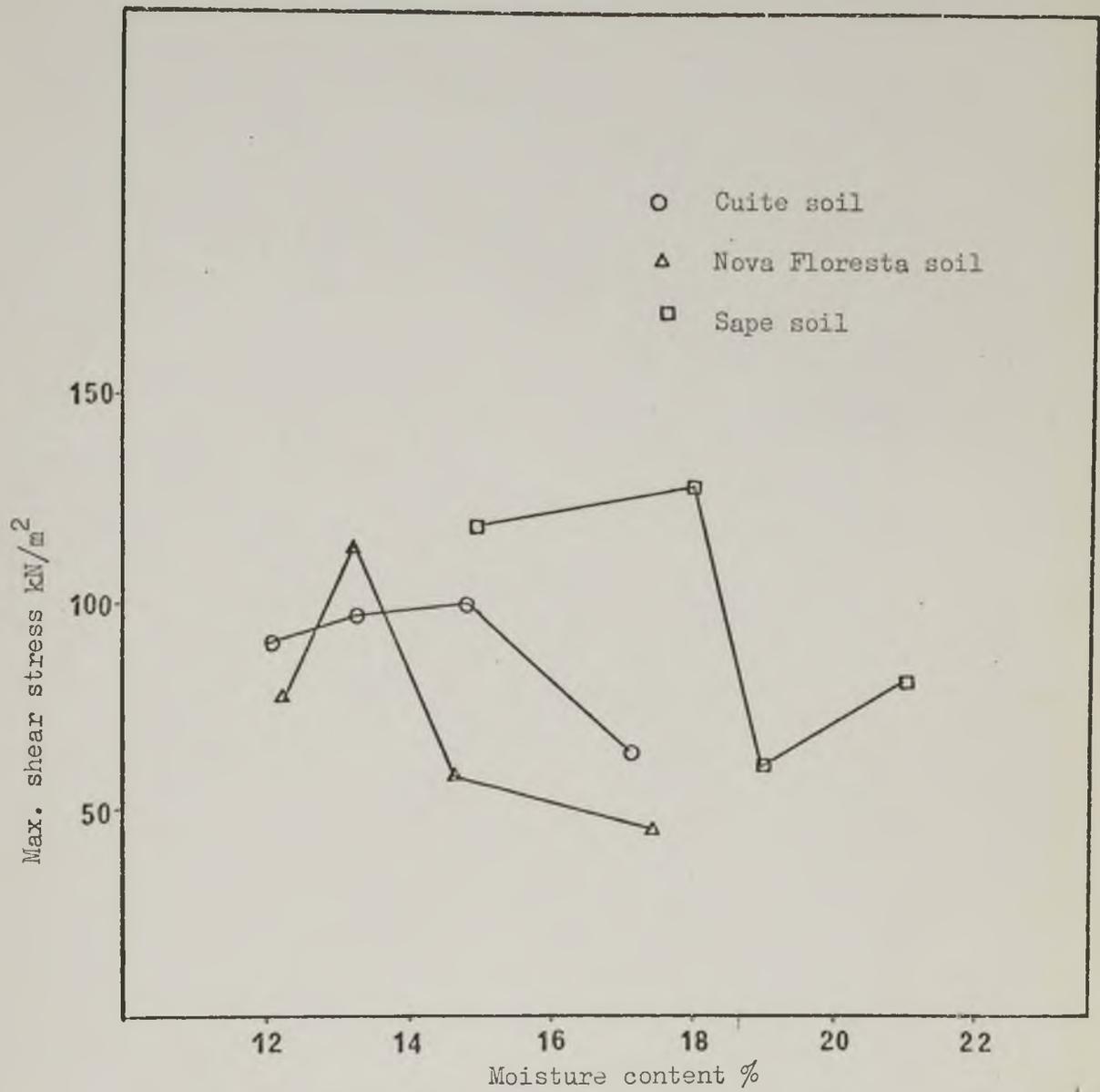


Fig. 7.7 Maximum shear strength stress versus moisture content for Cuite, Nova Floresta and Sape soils (Applied normal stress - 70.7 kN/m²)

The stress-strain plots for other moisture contents and different normal loads besides the ones mentioned above can be found in Appendix B and show that the behaviour patterns are similar. The similarity in behaviour suggests that a single common factor is present in all the three soils.

7.2.1.2 Volume Change:

The change in volume of specimens under shear was indirectly measured by change in their vertical dimensions with increasing strain. The plots of vertical deformation against strain are presented in Figures 7.8, 7.9.

A conclusion similar to that of the stress-strain behaviour can be reached i.e. over-consolidated clay behaviour under low normal stresses to normally consolidated clay behaviour at high normal stresses.

At low normal stresses the specimens dilate during shear, but at high normal stresses the specimens are compressed. The change from dilatant behaviour to one of volume decrease is also observable with increase in moisture content, for samples with different initial moisture conditions.

7.2.2. Triaxial Tests:

The Mohr envelopes for determination of the shear strength parameters were obtained by testing a minimum of three identical specimens at each moisture content for all the forms of triaxial tests carried out.

It is necessary to use a rate of strain slow enough to allow for drainage in the drained tests and precise measurement of pore pressure in the undrained tests. The adequate rate of strain can be determined from calculations depending on the coefficient of consolidation, specimen size and conditions of drainage as suggested by Bishop and Henkel (1962).

Alternatively, a trial and error method can be used until the adequate

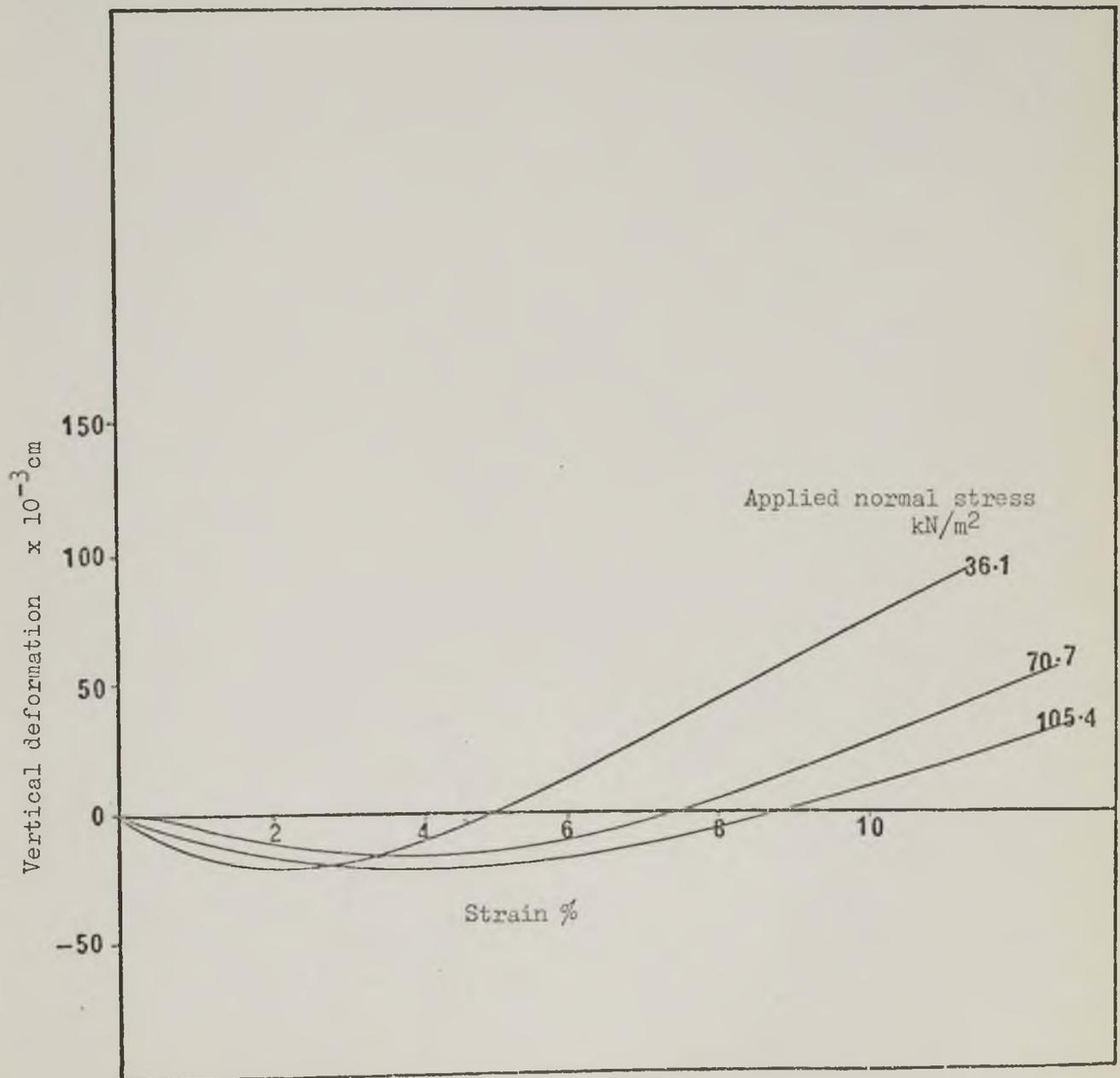


Fig. 7.8 Vertical deformation-strain curves for Nova Floresta soil in the direct shear tests at three normal stresses (moisture content = 14.6%)

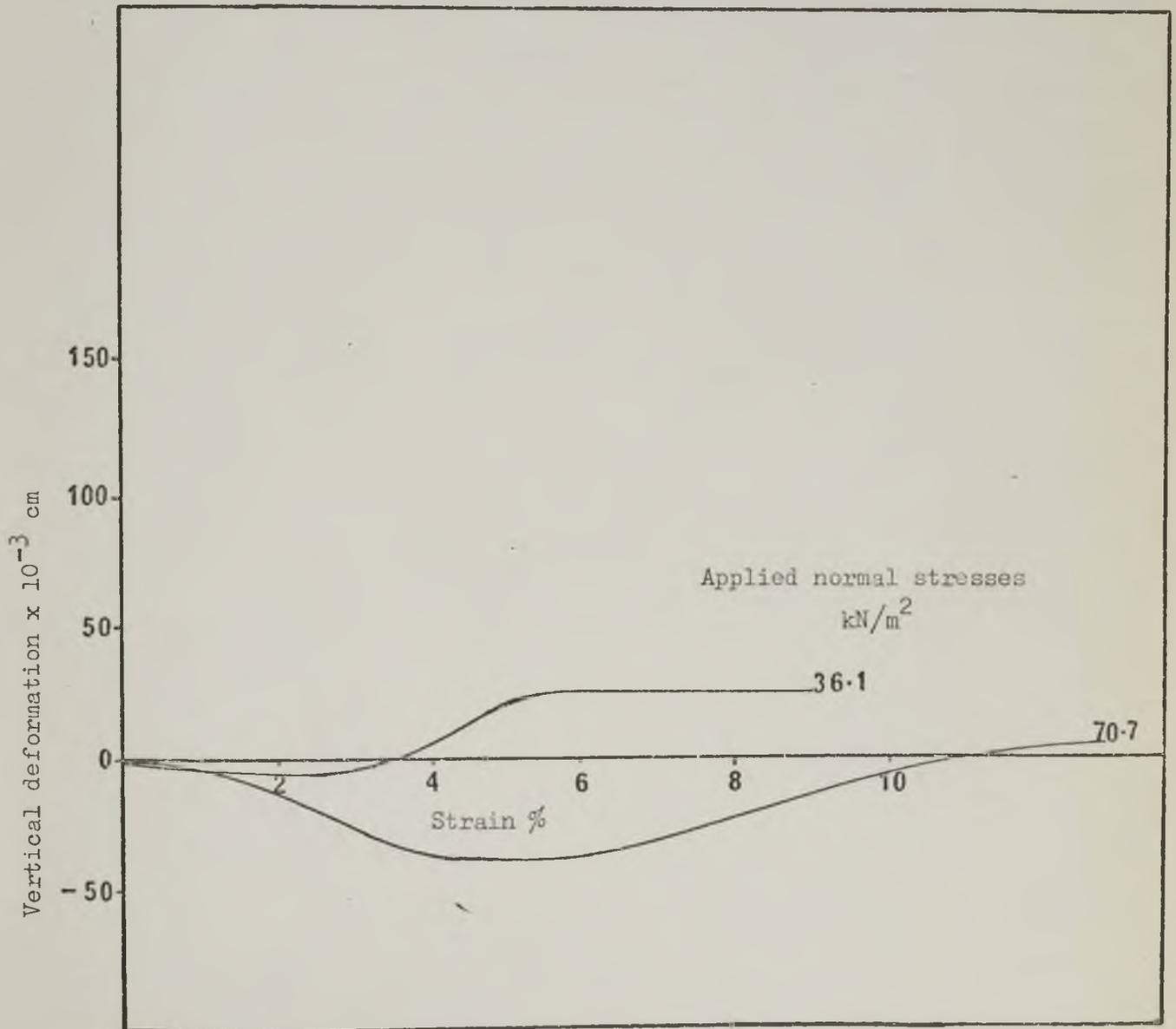


Fig. 7.9 Vertical deformation-strain curves for Sape soil in the direct shear tests at two normal stresses (moisture content = 14.6%)

rate of strain is determined. The latter method was used to determine the adequate rate of strain and one of 0.0228 mm/min was considered fast enough to prevent pore-pressure build-up in the drained tests and its measurement in the undrained tests.

7.2.2.1 Drained Tests:

The tests were carried out with confining pressures σ_3 of 98.1 (1), 196.2 (2), 294.3 (3) kN/m^2 (kgf/cm^2). Note that here, since the dissipation of pore pressure is allowed the applied deviator stress and the confining pressure are both effective. The plot of effective stress parameters C' and ϕ' versus moisture content are shown in Figure 7.10. In Figure 7.10(b) the angle of friction ϕ' increases considerably with increase in moisture content, attaining a maximum value of 27° , at a moisture content of 17.5%. Figure 7.10(a) shows that C' varies inversely with moisture content attaining a maximum value of 129.4 kN/m^2 at 15.6% moisture content on the dry-side of the moisture density curve. Minimum value of C' is 19.62 kN/m^2 and corresponds to a moisture content of 20.8%.

7.2.2.2 Undrained Tests:

Specimens were initially submitted to confining pressures of 34.33 kN/m^2 in the triaxial cell, an attempt to achieve homogeneous pore pressure within the specimen. When this pressure attained a constant value, confining pressures for carrying out the tests were applied and the pore pressure developed within the samples was measured with time until consistent values were obtained. The final value was used in determining Skempton's pore pressure parameter B . After the attainment of consistent values of pore pressure, the deviator stress was applied and tests carried out with pore pressure measurement. The tests were carried out with confining pressures of 68.67, 137.34, 206 kN/m^2 .

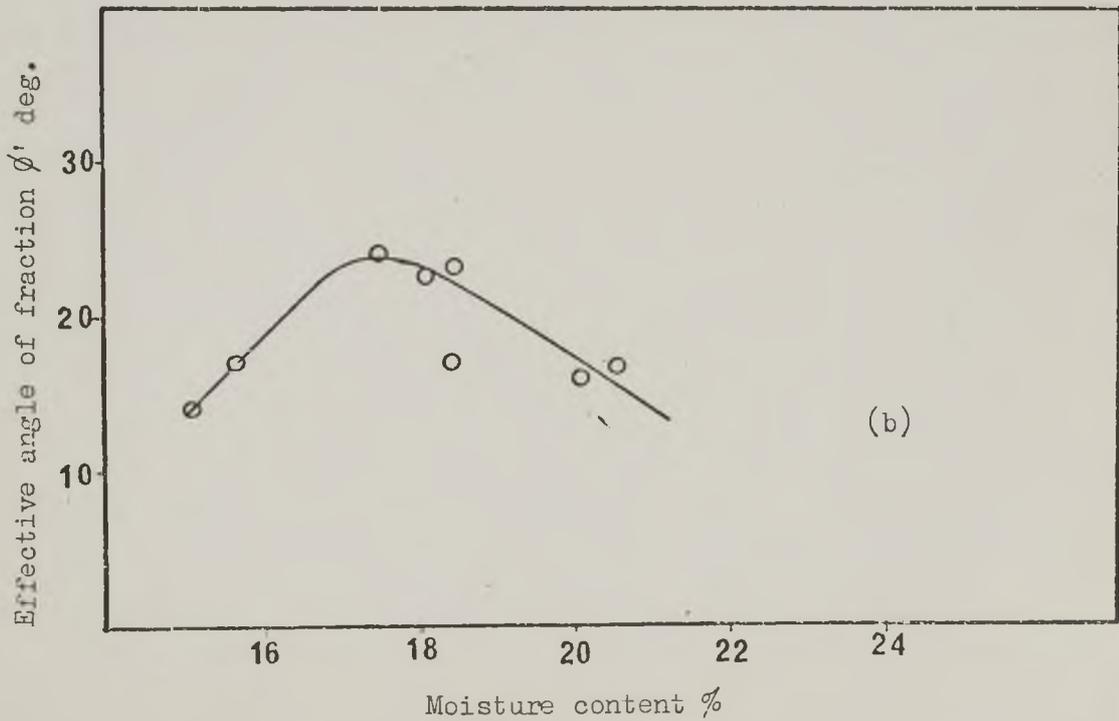
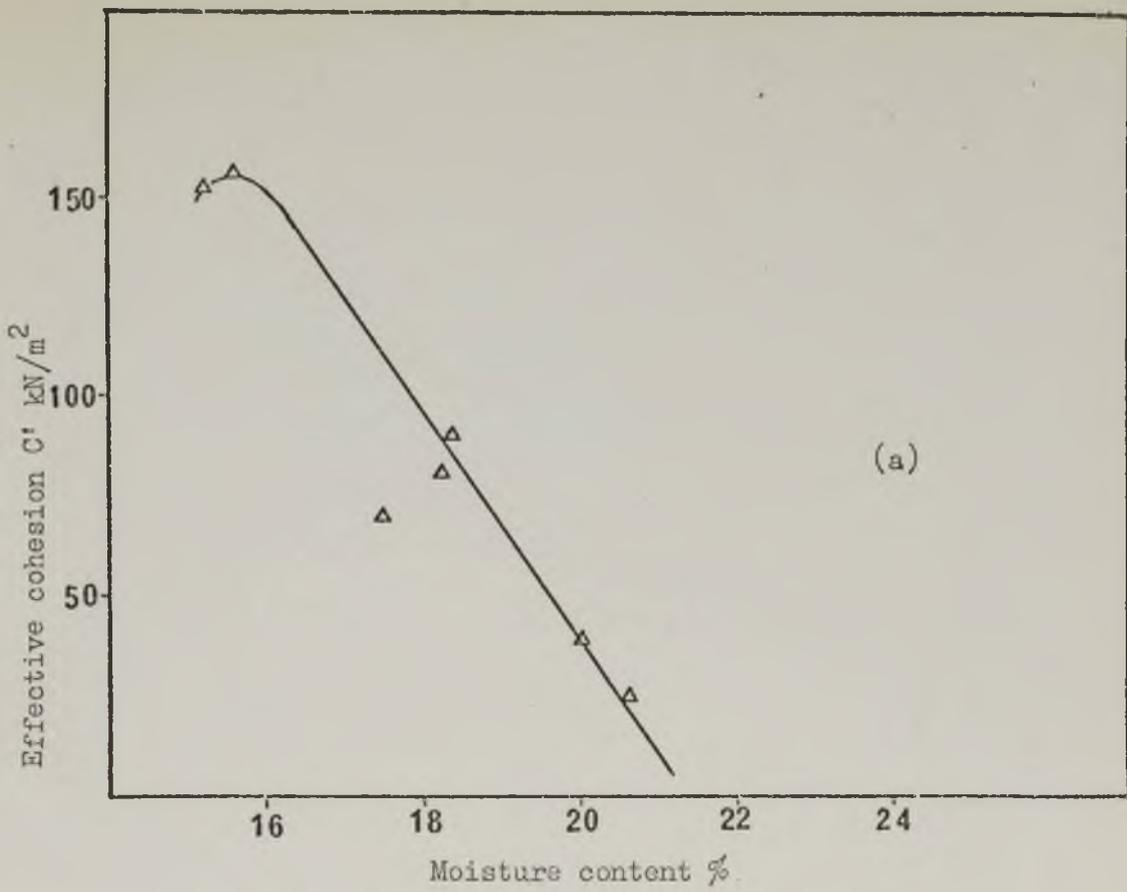


Fig. 7.10 Effective cohesion and effective angle of friction in the drained triaxial test. Specimens were compacted at different moisture contents along the moisture-density curve. Sape soil.

The values of the shear strength parameters obtained are shown in terms of total and effective stresses. Figure 7.11(b) shows the plot of ϕ_u versus compaction moisture content. The trend of ϕ_u versus moisture content here is not similar to that obtained for the drained tests. Change in ϕ_u with moisture content does not appear to have a maximum value for the range of moisture content examined. It decreases with increase in the moisture content. The highest value of $\phi_u = 29^\circ$ was obtained at a moisture content of 15% and the lowest value $\phi_u = 9^\circ$ was obtained at 21%.

The plots of ϕ' versus moisture content Figure 7.11(d) exhibits the same tendency, differences only lie in the actual ϕ' values for the same moisture contents. The highest value of $\phi' = 33^\circ$ is for 15.50% moisture content and the lowest $\phi' = 12^\circ$ is for 21%.

The plots for C_u and C' versus moisture content are shown in Figures 7.11(a), (c) respectively. Both show a tendency of having a maximum and minimum value. The maximum value of $C_u = 80.44 \text{ kN/m}^2$ at a moisture content of 17.3% while that of $C' = 77.49 \text{ kN/m}^2$ at about the same moisture content. The minimum values were 15.70 kN/m^2 and 27.47 kN/m^2 for C_u and C' respectively at a moisture content of 21%. It is evident that the numerical differences are very small and if the repeatability of the results are taken into consideration, it will be difficult to conclude that any real differences exist between the maximum and the minimum values of cohesion in terms of effective stress or total stress.

It is probably important to mention that for the partially saturated soils in these tests, the effective stresses were obtained by subtracting the values of pore pressure multiplied by coefficient χ (see Bishop and Henkel (1962) from the values of the total stresses. This assumes that a linear relationship exists between u and S_r the degree of saturation. Figure 7.12 shows the plot of Skempton's pore pressure parameter B

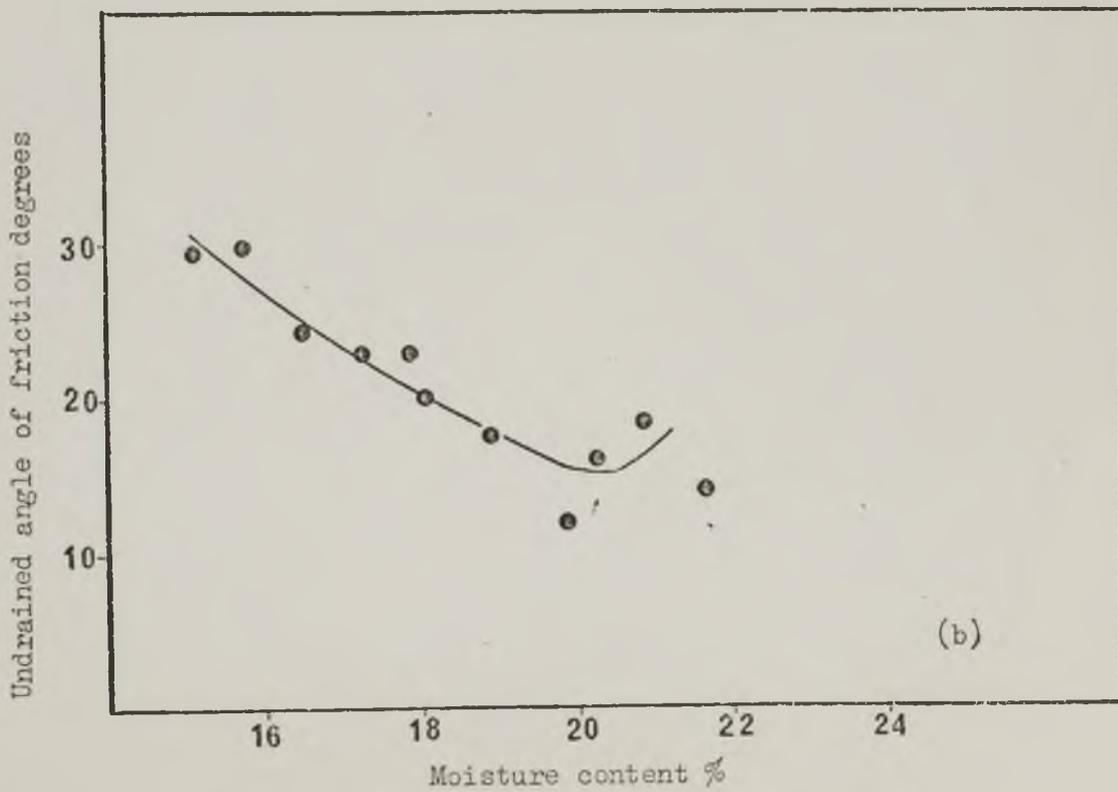
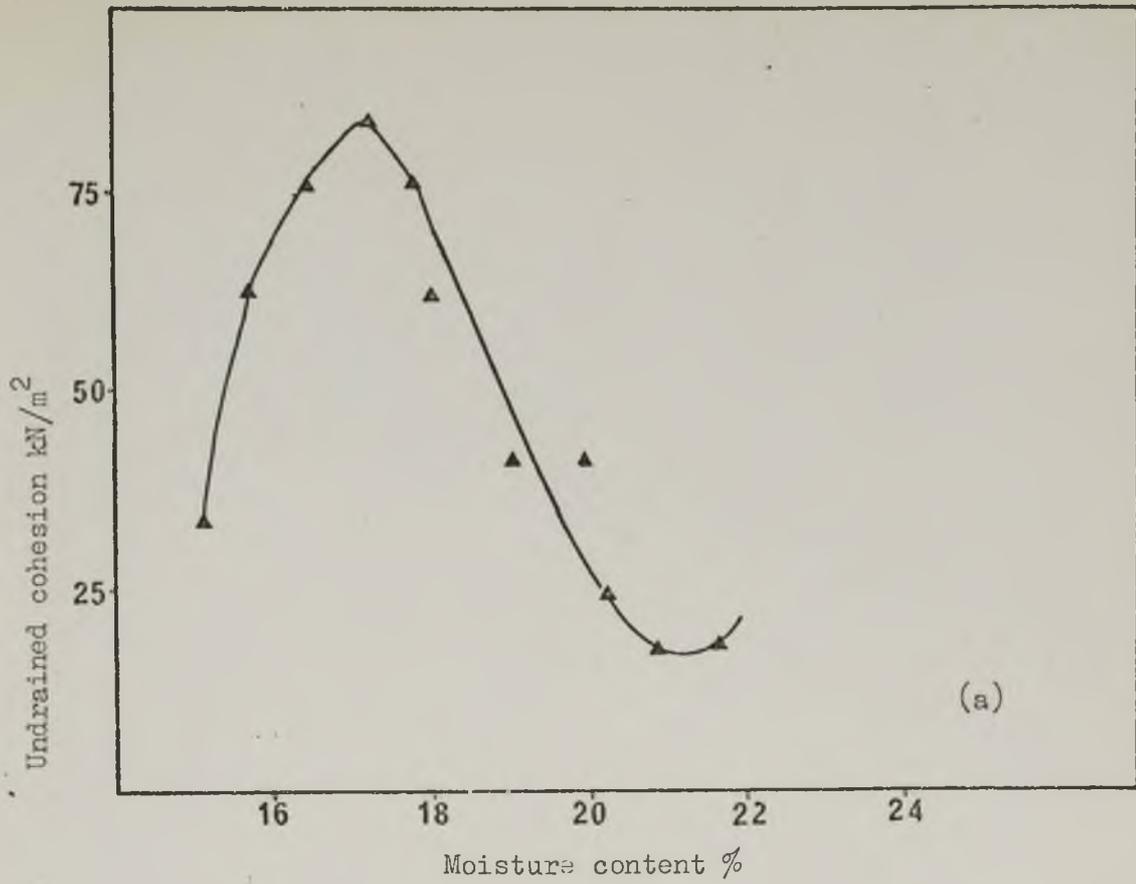


Fig. 7.11 Undrained cohesion and undrained angle of friction in the undrained triaxial tests. Specimens were compacted at different moisture contents along the moisture-density curve. Sape soil.

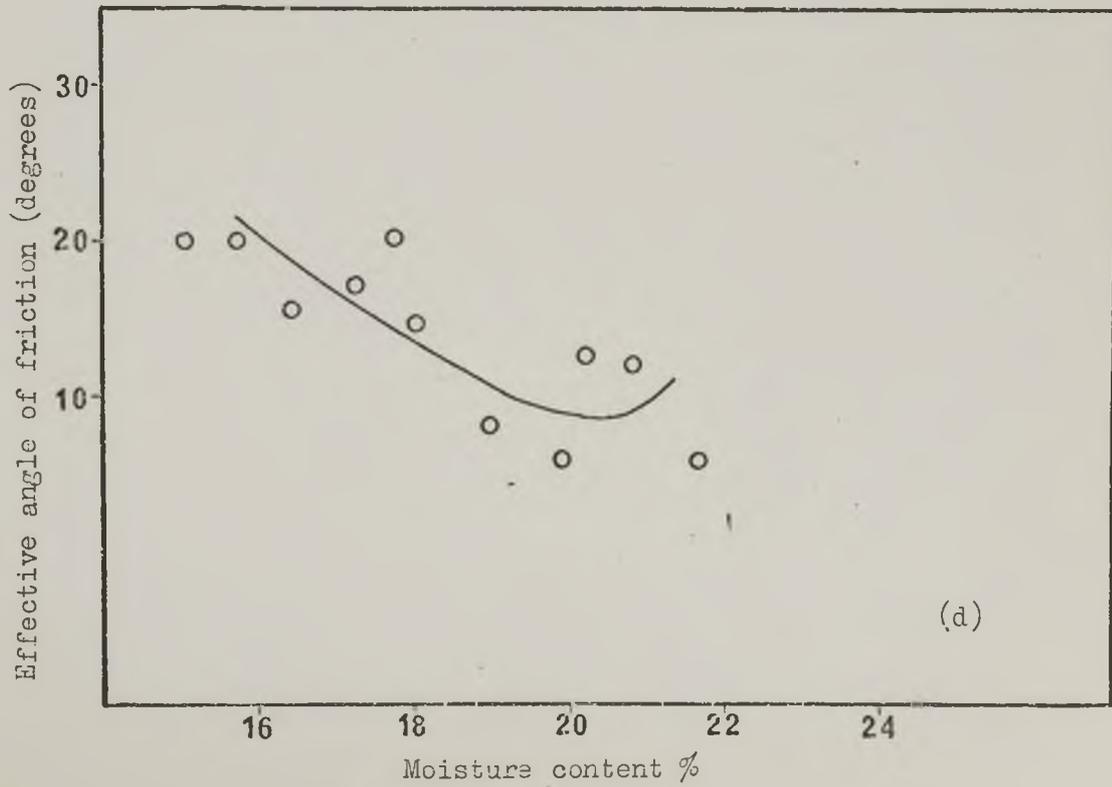
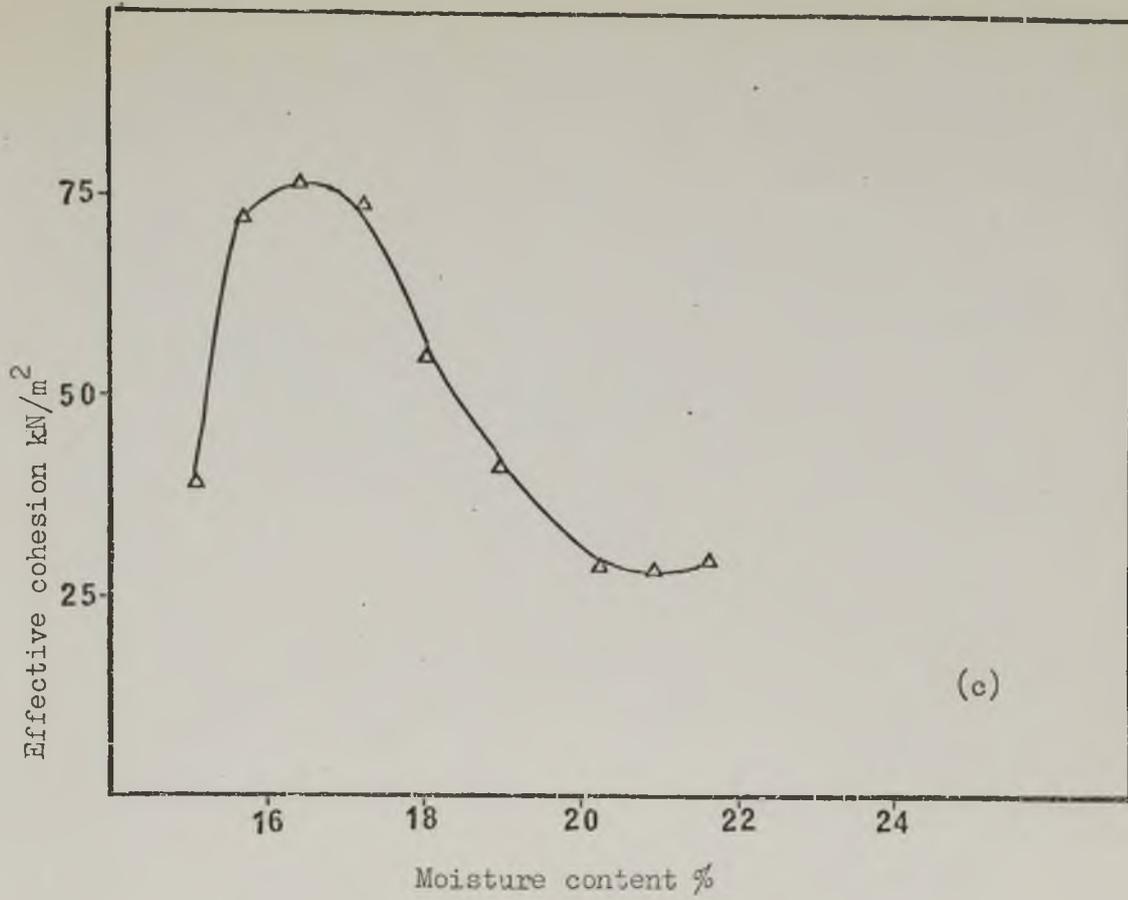


Fig. 7.11 Cohesion and angle of friction in the undrained triaxial tests. Specimens were compacted at different moisture contents along the moisture-density curve. Sape soil.

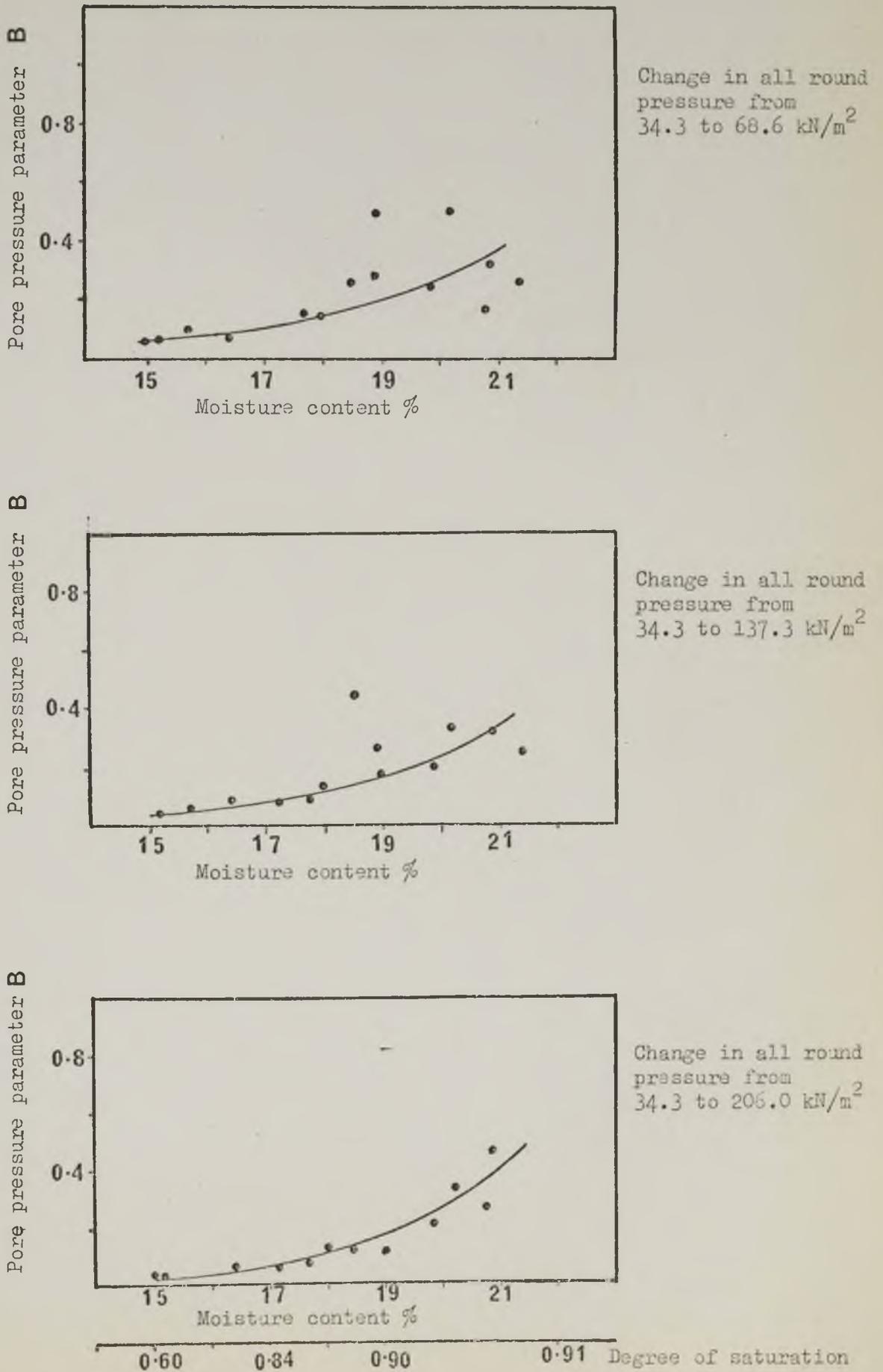


Fig. 7.12 Pore pressure parameter B versus moisture content for three changes in all round pressure. Sape soil.

versus molding moisture content.

7.2.2.3 Consolidated-Undrained Tests:

The specimens compacted at different moisture contents were saturated in the triaxial cell using back pressure as already described. The pressure employed for consolidation are the same as the ones for carrying out the tests. These are 98.1, 156.96, 255.06 kN/m².

Figures 7.13(b) and 7.13(d) show that the behaviour of the angle of friction shearing resistance in terms of total stress ϕ_{cu} and effective stress ϕ'_{cu} are similar. The maximum value of $\phi_{cu} = 25.39^\circ$ and ϕ'_{cu} occur at a moisture content of 17.5%, which is the optimum moisture content for the maximum dry density on a moisture density curve obtained by compaction in the standard Proctor mould.

The plots of cohesion in terms of total and effective stresses versus moisture content are shown in Figure 7.13(a), (c). Here a behaviour similar to the one recorded for non-saturated specimens was observed.

7.2.2.4 Stress-strain Relationships:

The plots of deviator stress versus strain are shown in Figures 7.14, 7.15(a). The behaviour under stress of this soil can be considered from the viewpoint of the closeness or the deviation from the general pattern established for natural soils.

The plots of stress versus strain show a general behaviour characteristic of a plastic system that is ascribable to normally consolidated clays in that the deviator stress increases with increase in the axial strain and do not reach a maximum even at high strains (up to 20%). Figures 7.14, 7.15(a).

The plots of σ_1/σ_3 for unconsolidated-undrained tests also did not give a maximum. Figure 7.15(b).

Plots of pore pressure versus axial strain, Figure 7.16 also show

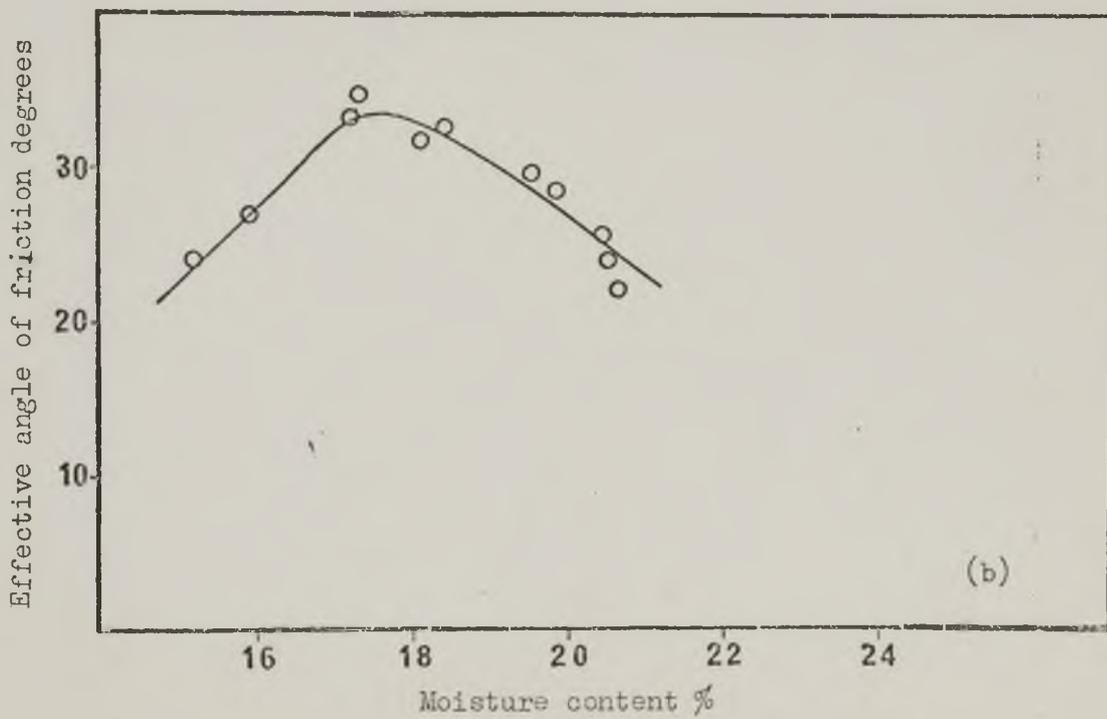
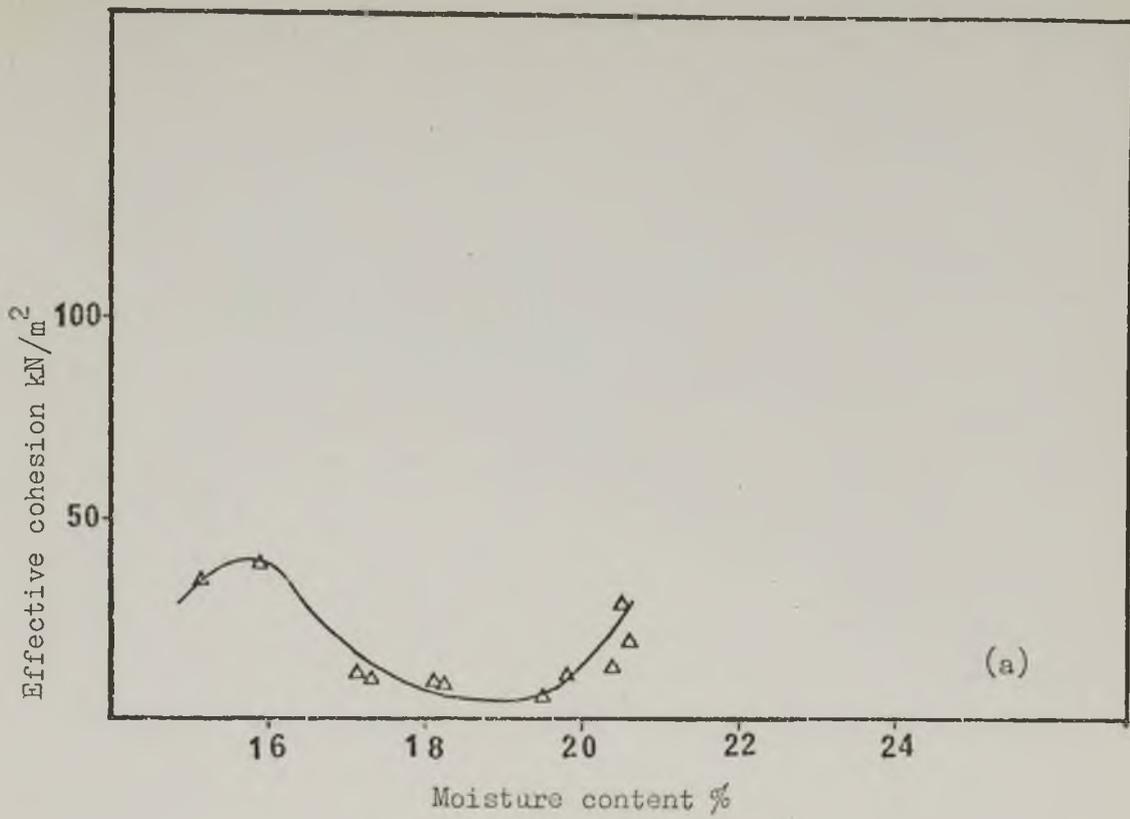


Fig. 7.13 Effective cohesion and effective angle of friction from consolidated undrained triaxial tests. Specimens were compacted at different moisture contents along the moisture-density curve. Sape soil.

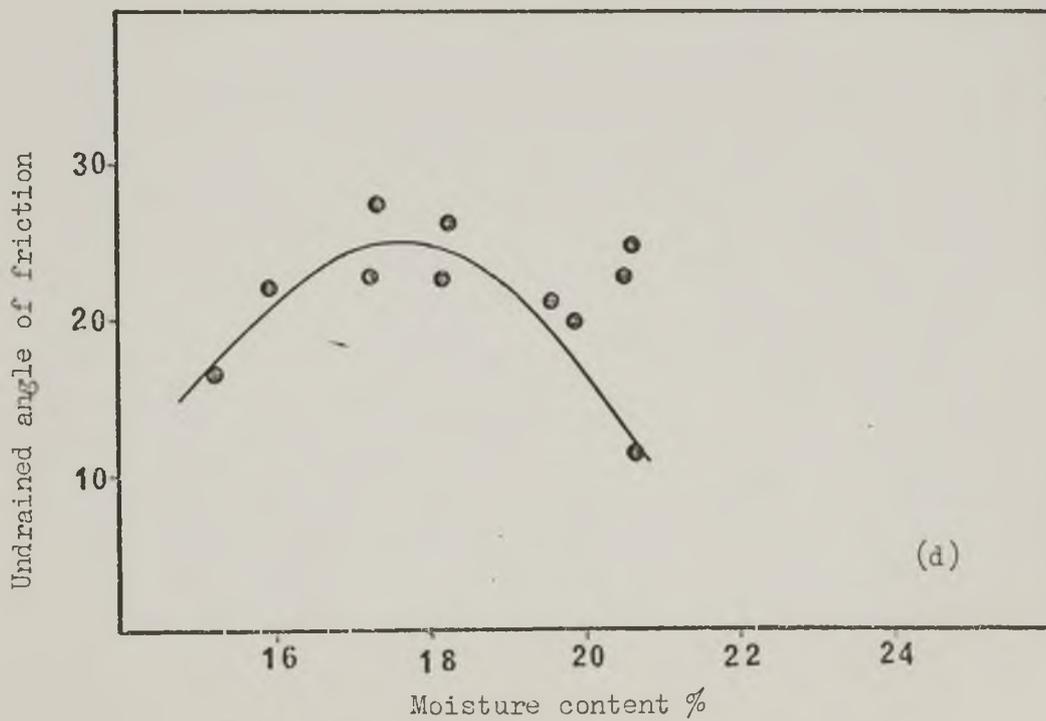
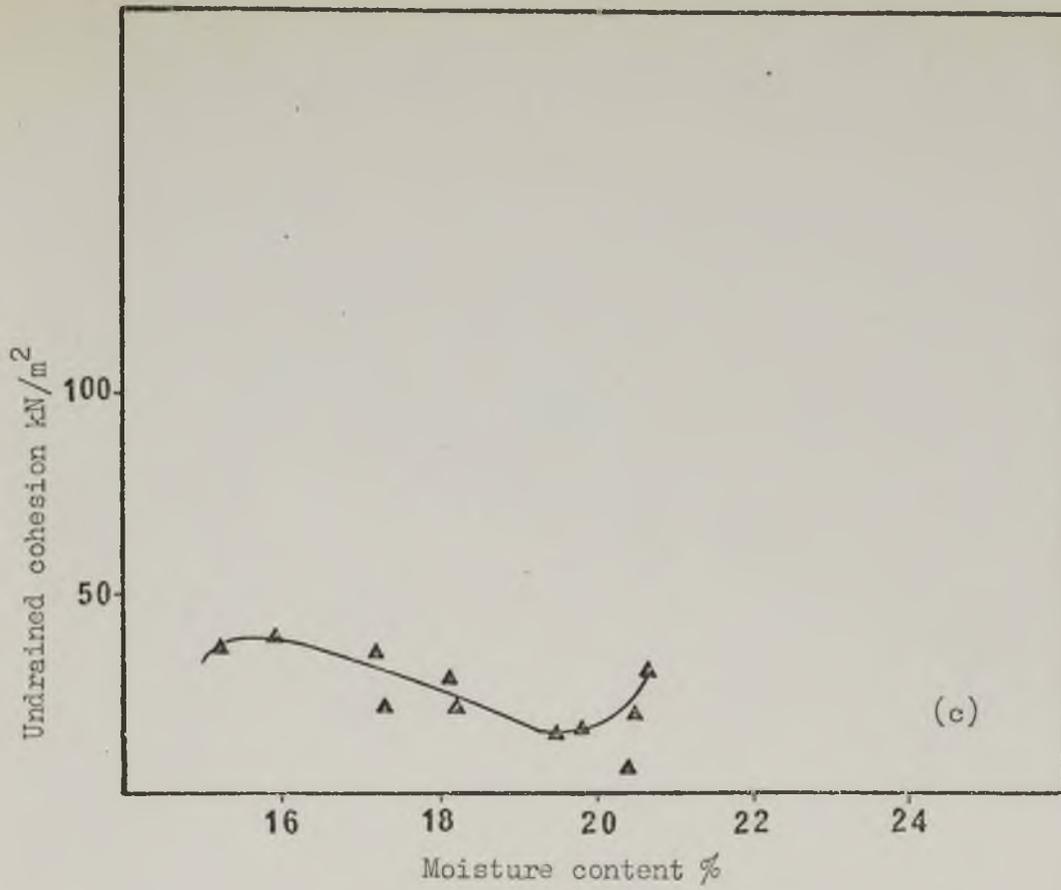


Fig. 7.13 Cohesion and angle of friction from consolidated-undrained triaxial tests Sape soil.

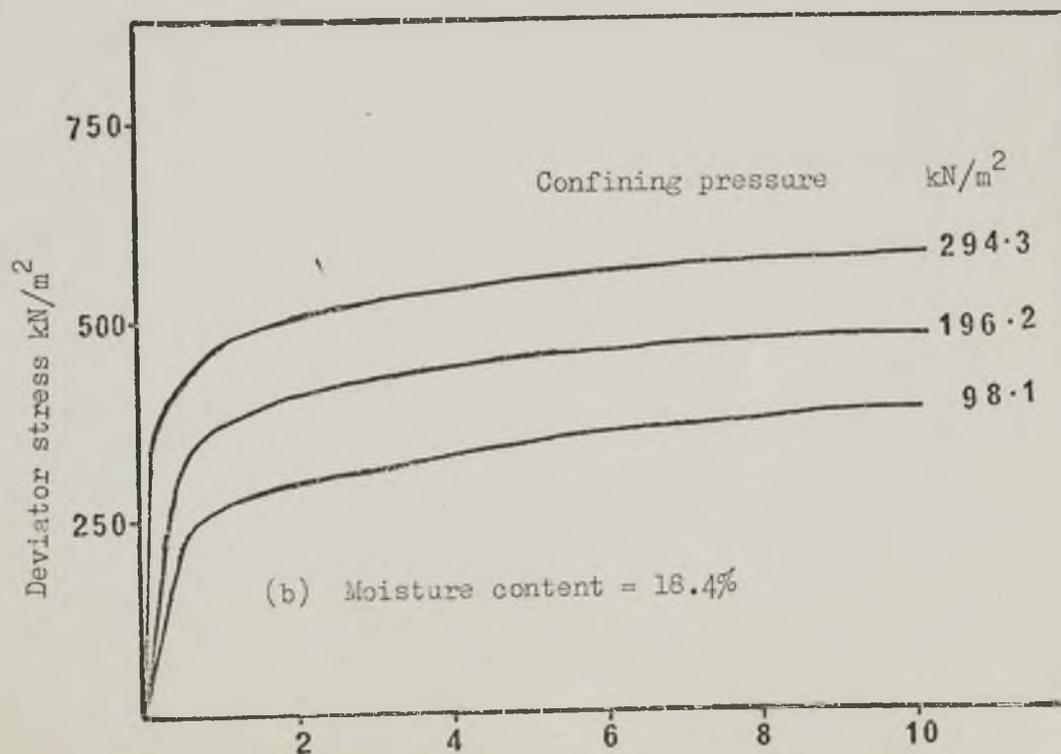
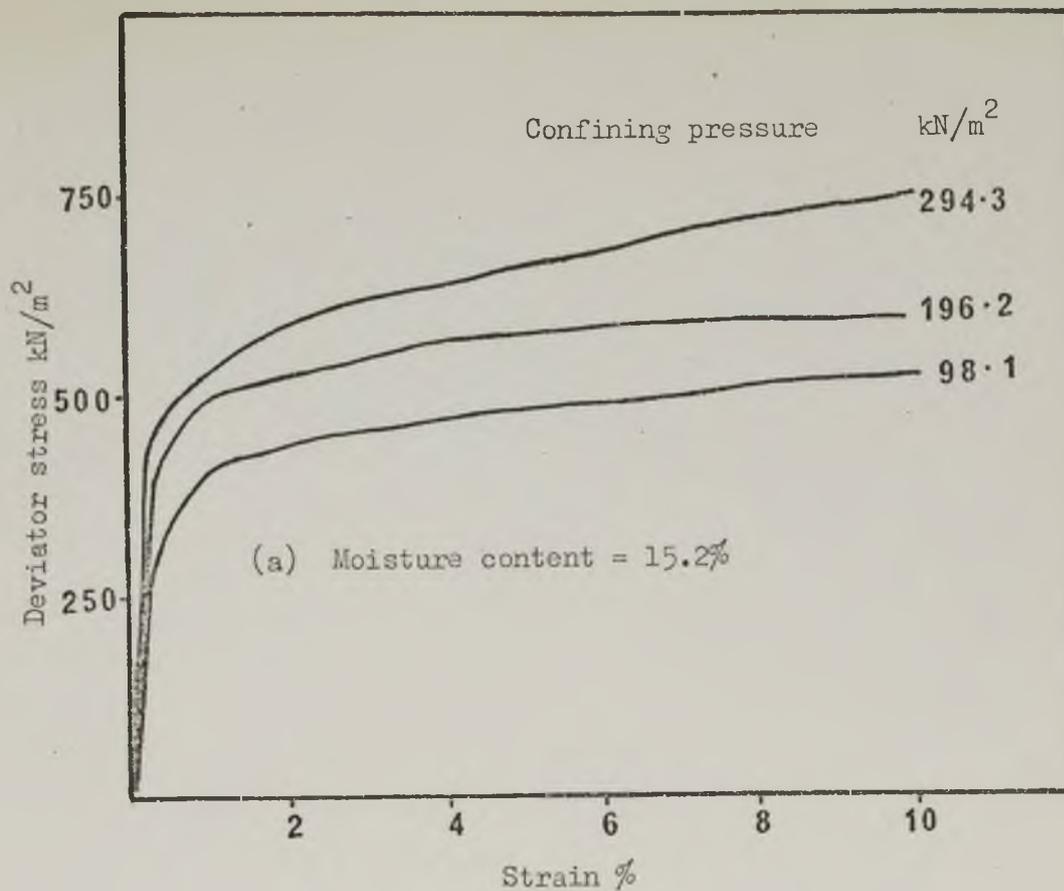
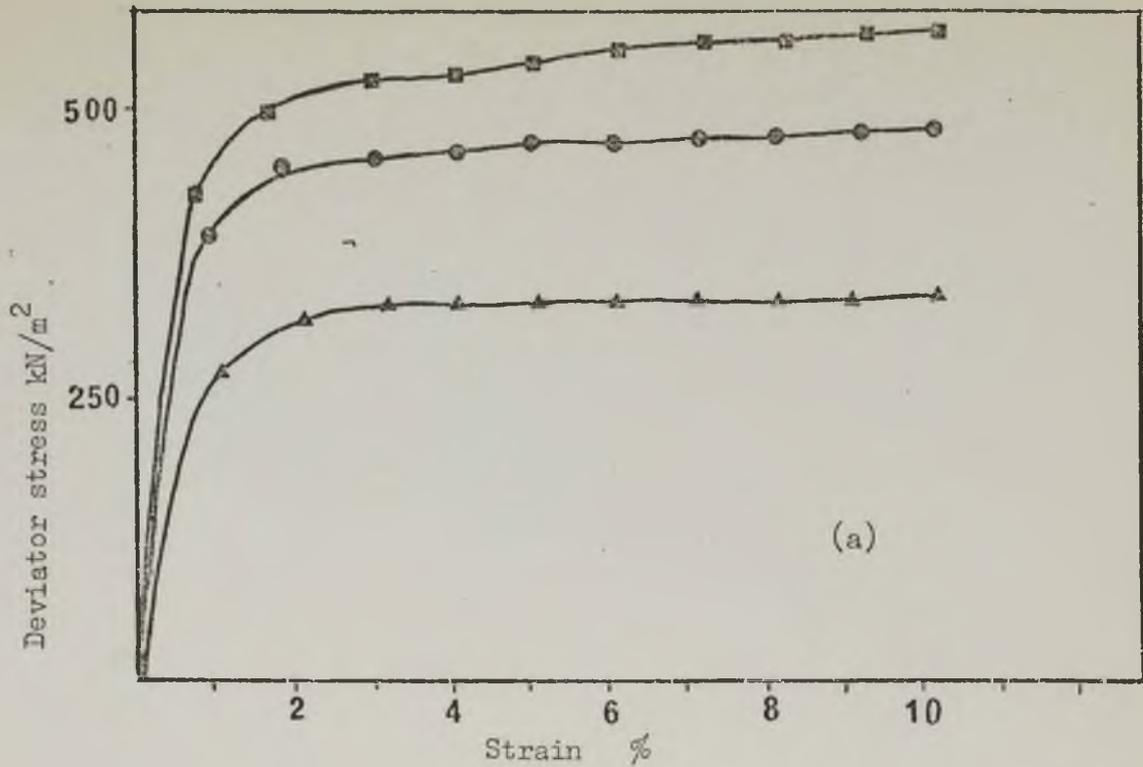
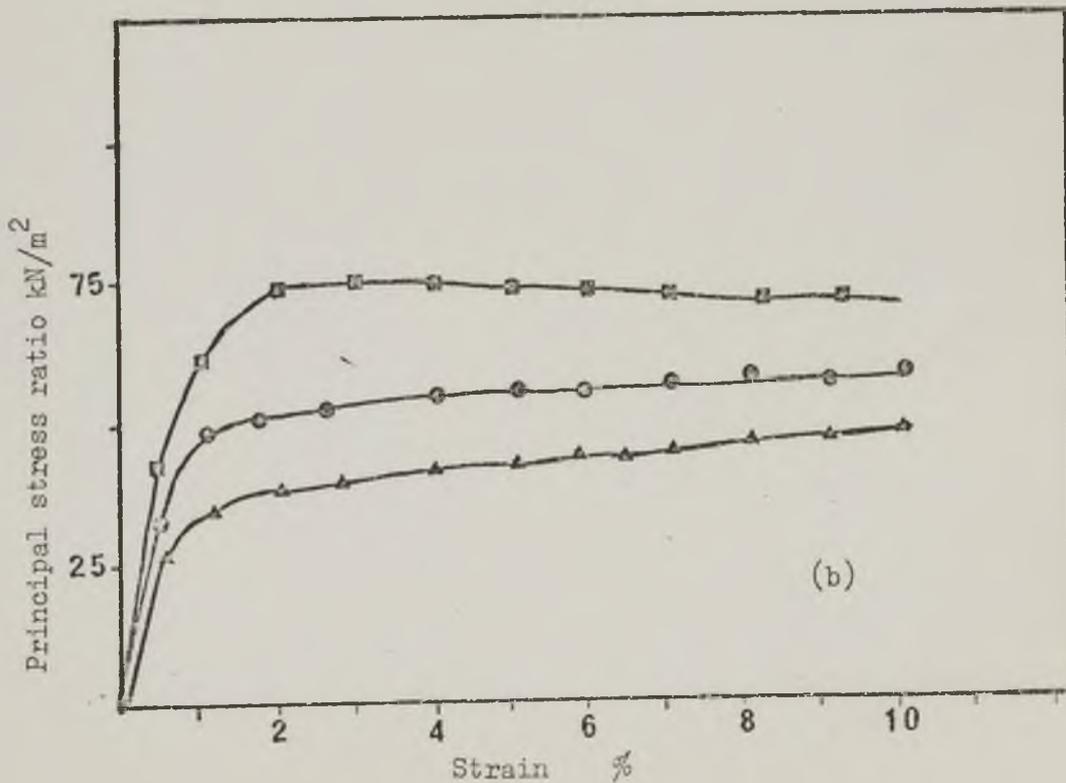


Fig. 7.14 Deviator stress versus strain curves for drained triaxial tests at three confining pressures Sape soil.



Deviator stress versus strain

- 206.85 Confining pressures
- 137.90 kN/m^2
- ▲ 68.95



Stress ratio versus strain

Fig. 7.15 Stress-strain curves for undrained triaxial tests at three confining pressures (moisture content 16.43%) Sape soil.

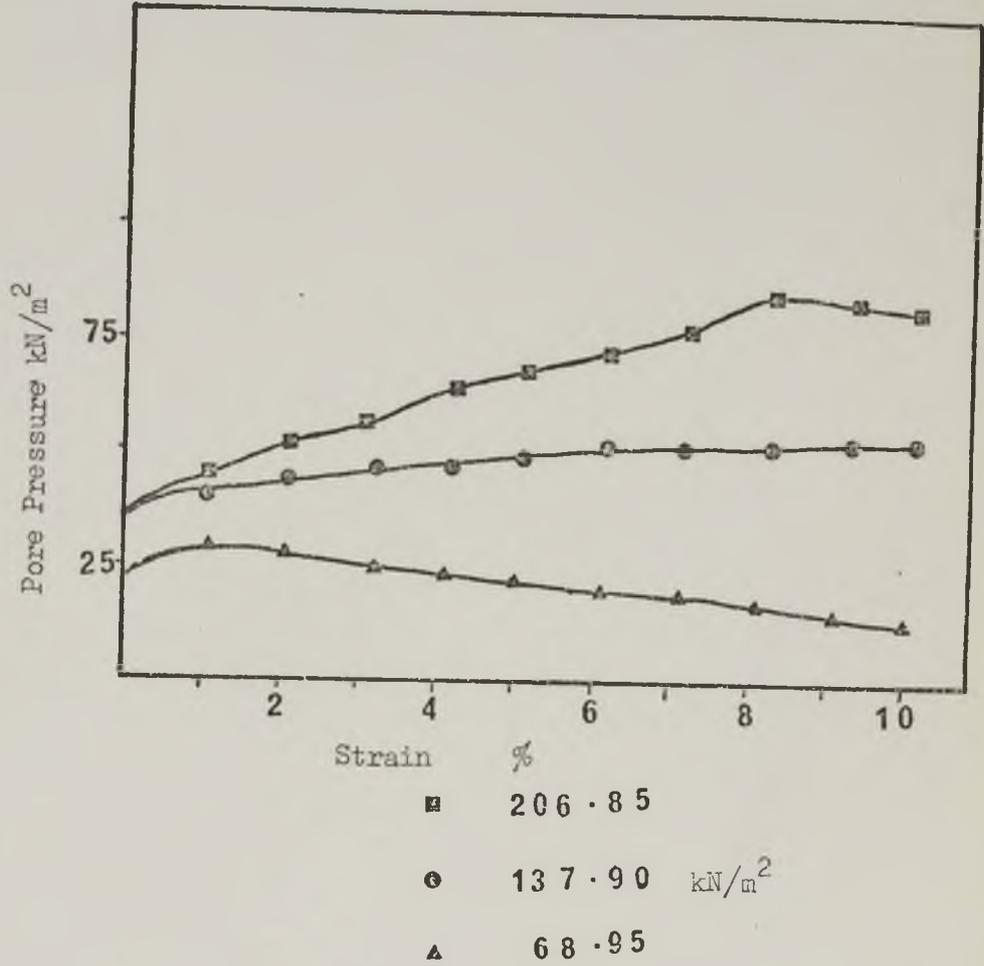


Fig. 7.16 Pore pressure developed in undrained test (moisture content 16.437). This corresponds to plots shown in Fig. 6.15 Sape soil.

a continuous increase, though small, for all values of axial strain in most of the tests. This continuous and steady increase is incompatible with the behaviour of a normally consolidated clay in that when a 'peak' is reached (usually defined by the intersection of tangents to the two straight line portions of the curve) the pore pressure will decrease as a result of volume increase on the failure surface. The existence of positive excess pore pressure throughout the test implies that there is volume decrease during failure, which again is incompatible with the observed features, i.e. no apparent failure surface was observable, all the specimens produced failure by buckling.

Although no peak stress was reached, in most of the tests 80% of the value of the deviator stress at large strains (say 20%) was reached at very low strains.

The implications of this in the design criteria of an embankment or an earth dam is that strength is not a critical factor, for at large values of strain considerable strength is still presented and thus the amount of allowable strain in the design becomes the important factor.

7.2.2.5 Pore pressures developed during loading:

The pore pressure change at the initial stage of the test is very small (see Figure 7.16). This usually increases gradually up to the end of loading.

The slight increase in pore pressure during shear implies that there is a decrease in volume during failure which is in line, with the expected over-consolidated clay-like behaviour of the compacted soil and is compatible with the shape of the failed specimens (i.e. failure by buckling). It is probably worth mentioning again, that volume measuring apparatus for the triaxial tests were not available in Brazil where these tests were carried out. The change in volume during loading was therefore not measured. It is, however, considered that fairly adequate analysis

can be made using the properties evaluated.

The development of pore pressures can be more clearly understood, if the Skempton's pore pressure parameters are considered. These parameters would show the response of the specimen to the externally applied load.

7.2.2.6 Pore Pressure Parameters A & B:

The pore pressure parameters A and B are parameters in an equation derived by Skempton (1954) to account for the response to loading in an undrained system. They are referred to as Skempton's pore pressure parameters A and B.

Parameter B relates the pore pressure developed to the applied stress under isotropic loading. A condition encountered in the common triaxial test where an all-round pressure is initially applied before the application of the axial pressure

$$B = \frac{\Delta u}{\Delta \sigma_3} = \frac{1}{1 + \frac{\eta C_r}{C_c}}$$

η = Porosity

C_r = Voids compressibility

C_c = Skeleton compressibility

Parameter B:

The pore pressure parameter B measured for non-saturated undrained soils all give very low values. The plot of B versus moisture content Figure 7.12 shows that for values below optimum moisture content on the moisture-density curve (about 90% degrees of saturation) the values of B are generally very low, hardly greater than 0.15. Beyond this, a slight increase is recorded for increase in saturation.

The parameter B also shows an unusual behaviour of decreasing with increase in $\Delta\sigma_3$ (despite a number of spurious observations, this trend is predominant). This will mean that the developed pore pressure was lower at higher confining pressure than for lower confining pressure.

The decrease in the values of the measured pore water pressure suggest the existence of negative pore pressure (suction) whose effects increase with increasing confining pressure.

Parameter A:

Skempton's pore pressure parameter A relates the pore pressure developed to the deviator stress responsible during loading in the normal triaxial tests, i.e. Δu and $\Delta\sigma_1 - \Delta\sigma_3$. A is given in $u = B(\Delta\sigma_3 + A(\Delta\sigma_1 - \Delta\sigma_3))$.

It was determined for saturated consolidated-undrained soils. A 3% strain criteria of failure was employed.

$$A = \frac{\Delta u - \Delta\sigma_3}{\Delta\sigma_1 - \Delta\sigma_3}$$

The values of A are also very low (see Table 6.1.) and reflect the response of the material to applied deviator stress. Values of A decrease with increasing density and in general increase with increasing $\Delta\sigma_3$.

7.2.2.7 Stress Paths:

Stress paths depict the successive states of stress that exist in a specimen as the specimen is loaded. It is a curve joining a series of stress points, i.e. it gives a continuous representation of successive states of stress.

The stress paths for consolidated-undrained soils compacted at three confining pressures are shown in Figure 7.17. The amount of mobilised

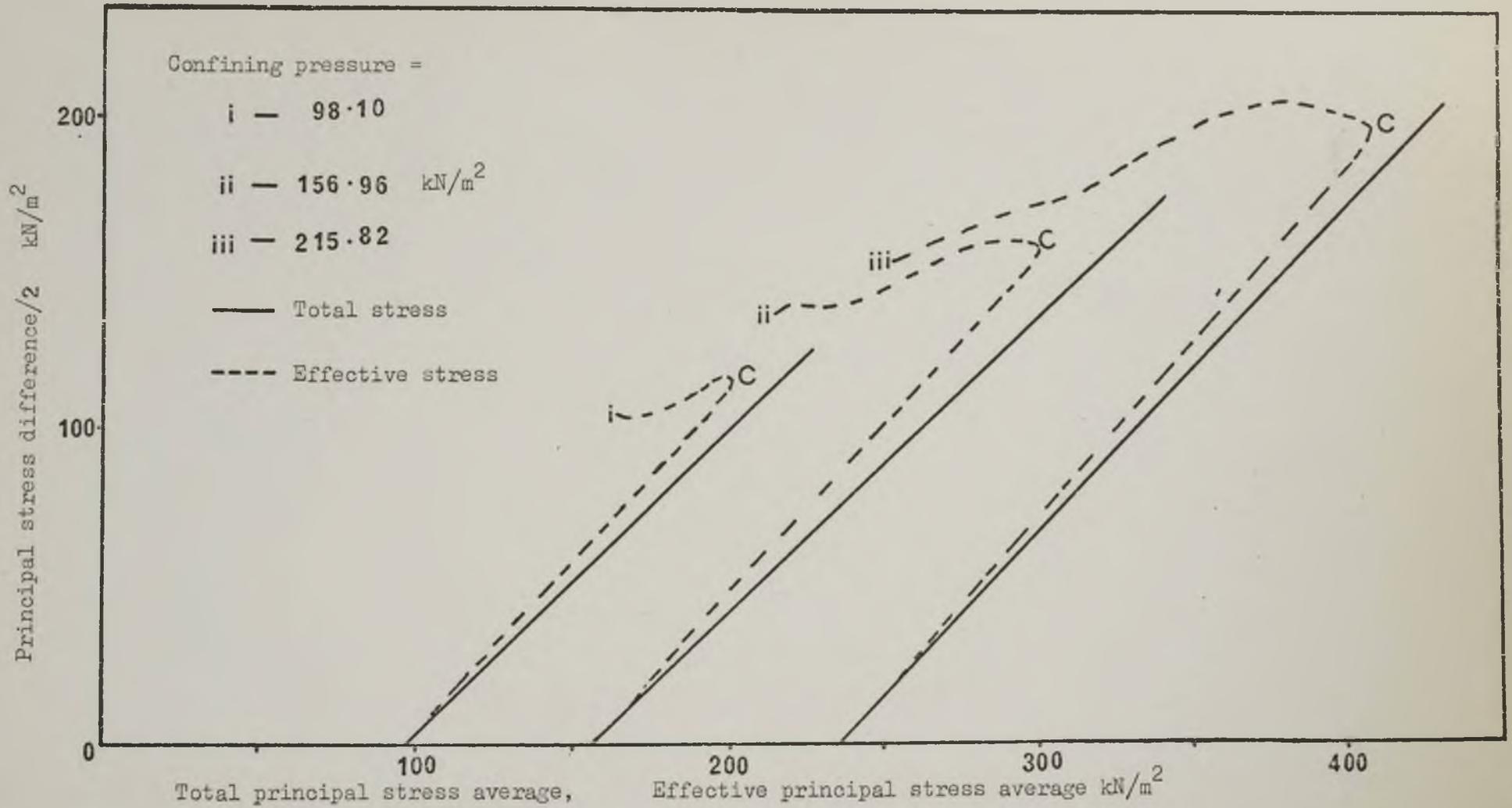


Fig. 7.17 Typical stress paths for consolidated undrained triaxial tests (moisture content 17.18%.)
Sape soil

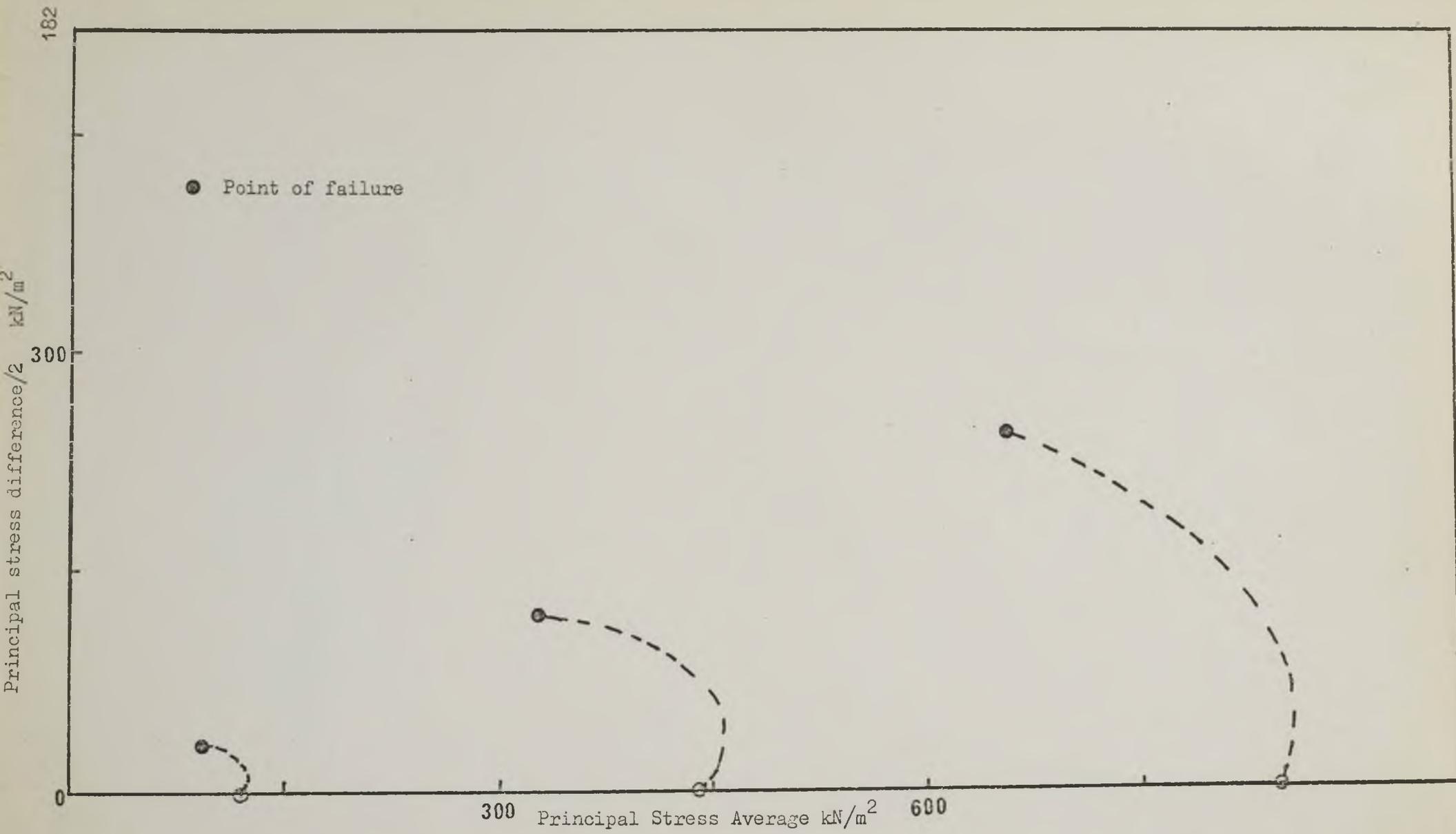


Fig. 7.18 Effective Stress paths from consolidated-undrained test on Weald clay (after Lambe and Whitman, 1969).

TABLE 7.2

Pore pressure parameter A determined from consolidated-undrained triaxial tests on Sape soil. Values correspond to a 3% strain criterion of failure.

Moisture of Compaction %	Dry Density kg/m ²	Confining Pressures kN/m ²		
		98.1	196.2	294.3
Pore Pressure Parameter A				
15.2	1744	0.171	0.261	0.260
15.9	1812	0.102	-	0.158
17.2	1857	0.096	0.174	0.212
17.3	1849	0.058	0.084	0.113
18.1	1858	0.078	0.254	0.287
18.2	1857	0.050	-	0.132
19.5	1825	0.168	0.218	0.284
19.8	1799	0.268	0.338	0.350
20.4	1799	0.207	-	0.203
20.5	1809	0.168	0.156	0.163
20.6	1769	0.186	0.360	0.463

effective stress at low values of strain is unusually high for a clay. It is characteristic of a granular material. For low values of $q = \frac{\sigma_1 - \sigma_3}{2}$, the value of effective stresses is very near to those of total stresses and the difference between them, i.e. the pore pressure, increases only gradually. A sudden change with marked increase occurs at point C. The q value of point C increases with increase in confining pressure.

This point corresponds to a large increase in pore pressure. Lambe and Whitman (1969, p.442) have drawn attention to this sort of behaviour as observed for cemented sensitive clays as a case where the strength characteristics of drained and undrained soils is not linear, i.e. the effective stress principle (as defined by Terzaghi (1925a)) does not directly connect the two states. Though the behaviours are similar, the mechanism operating here cannot be the same since the conditions of the soils are different.

Lambe and Whitman (1969) describe the stress-strain curves for consolidated tests on Weald clay (p.427), a diagram from which is shown in Figure 7.18. Positive excess pore pressures developed during tests on normally consolidated clays and $p' = \sigma_1' + \sigma_3'$ decreases after a slight initial increase.

The decrease in p' which usually means an increase in volume was just enough to compensate for the decrease in volume which usually accompanies shearing, since there was no net volume change, the strain necessary to fail the specimen was less in a consolidated-undrained test than in a consolidated drained test.

In overconsolidated clays positive excess pore pressure develops and p' increases markedly during the test. Thus the tendency towards volume increase during shear is counteracted by an increase in effective stress. For heavily over-consolidated specimens, the tendency towards

volume expansion exists out to large strains and consequently, the excess pore pressure induced by undrained shear continues to decrease to large strains. The decreasing pore pressure imply increasing effective stress and the stress-strain curves continue to rise out to very large strains.

In the case of the Sape soil, positive excess pore pressure also developed, but this pore pressure is not large enough, initially, to produce a decrease in effective stress and therefore p' increases during the test in a manner similar to over-consolidated clays.

Since the specimens are compacted prior to testing, the comparison with over-consolidated clays is not out of place and seems to account for the increase in the mean effective stresses with increase in mean deviator stress until failure takes place.

7.2.2.8 Strength Parameters ϕ and C :

The strength parameters, cohesion and the angle of frictional resistance determined in terms of total and effective stresses show some unusual trends.

Cohesion:

The plot of effective cohesion versus moisture of compaction for drained soils is shown in Figure 7.10. It can be seen that on going from the dry to the wet-side, the cohesion increases slightly, reaching a peak at values slightly lower than optimum and then decreases more rapidly towards the wet-side.

If this is compared with the plot of cohesion versus moisture of compaction for saturated consolidated undrained specimens (Figure 7.13), a similar trend is recorded. At the dry-side of optimum C'_{cu} also increases up to a maximum near optimum moisture content and then decreases. Here a minimum value of cohesion is reached on the wet side beyond which C'_{cu} increases.

The difference in magnitude and in behaviour of the cohesion parameter in terms of effective stresses under drained and undrained conditions possibly suggests that the boundary conditions accepted for the tests are not what are actually operating, i.e. the effective stress parameter does not linearly connect the two.

Angle of frictional resistance ϕ :

The angle of frictional resistance ϕ' for the drained soils varies between 14.3° and 22.9° , while those of consolidated undrained test varies between 22.1° and 34.7° .

The plots of these values as well as those of ϕ_u are shown in Figures 7.10, 7.11, 7.13. We can see that the values determined from drained tests with pore pressure measurement is similar but not identical. The magnitudes are different though the curves follow similar trends.

7.2.3 Compressibility:

The compressibility characteristics of the Sape soil compacted at three moisture contents as in the direct shear test, were determined in an oedometer cell (see 7.1.1 for methods of preparation and mode of saturation).

Figure 7.19 shows the plot of actual specimen thickness versus log of applied pressure. The compressibility is fairly low in the stress range considered. This was found to increase from very low values at low pressures to higher values at higher pressure. At no point is a linear relationship found between the voids ratio (determined from specimen thickness) and the amount of applied pressure, see Figure 7.21 (a) and (b).

The compressibility characteristics of undisturbed residual soils have been investigated by Vargas (1953) for residual soils of Southern Brazil, Sowers (1963) for residual soils of Southern United States and Wallace (1973) for the soils of the continually wet highlands of

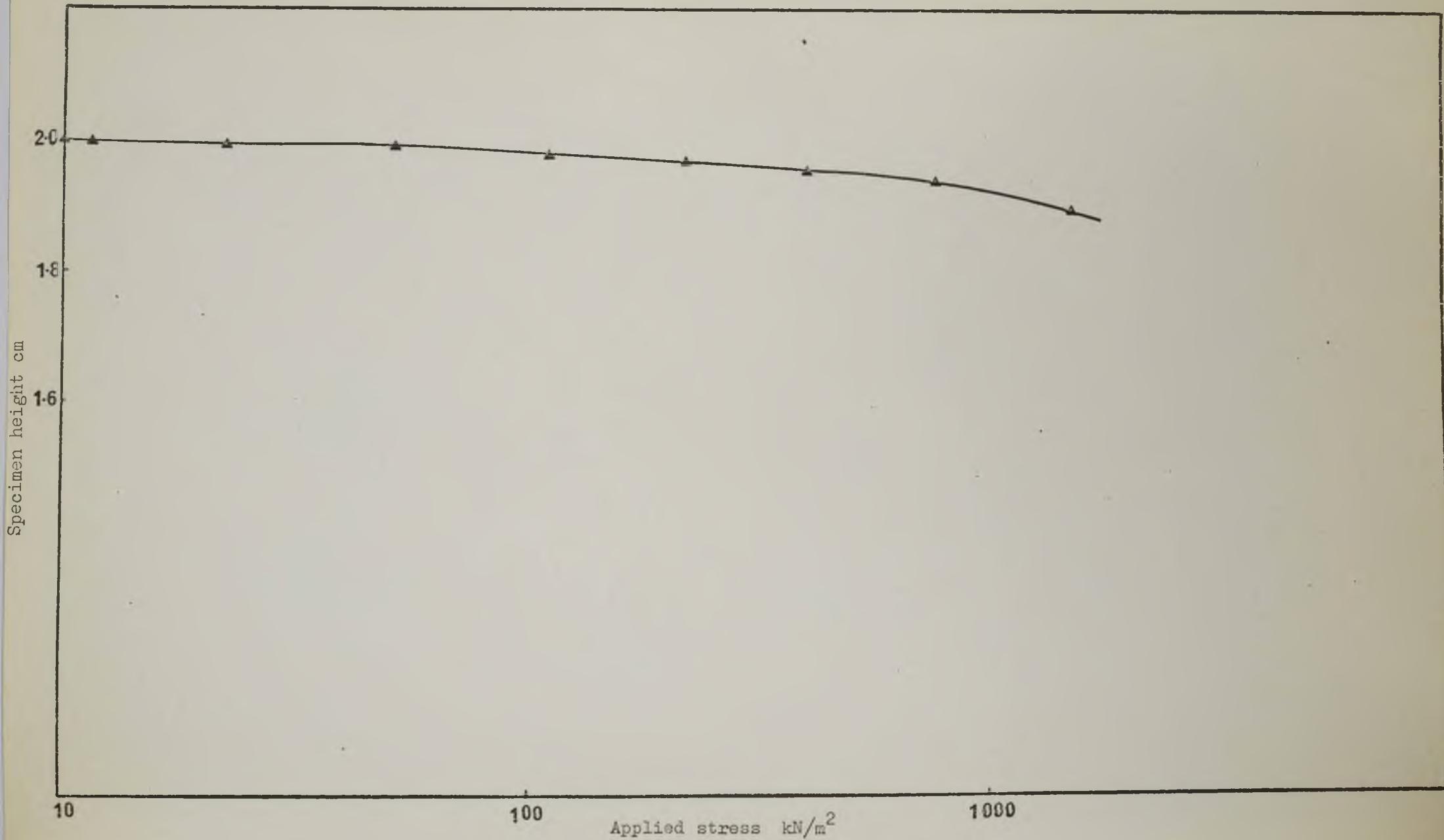


Fig. 7.19 Compressibility of Sape soil at 15% (Dry-side) in the oedometer tests.

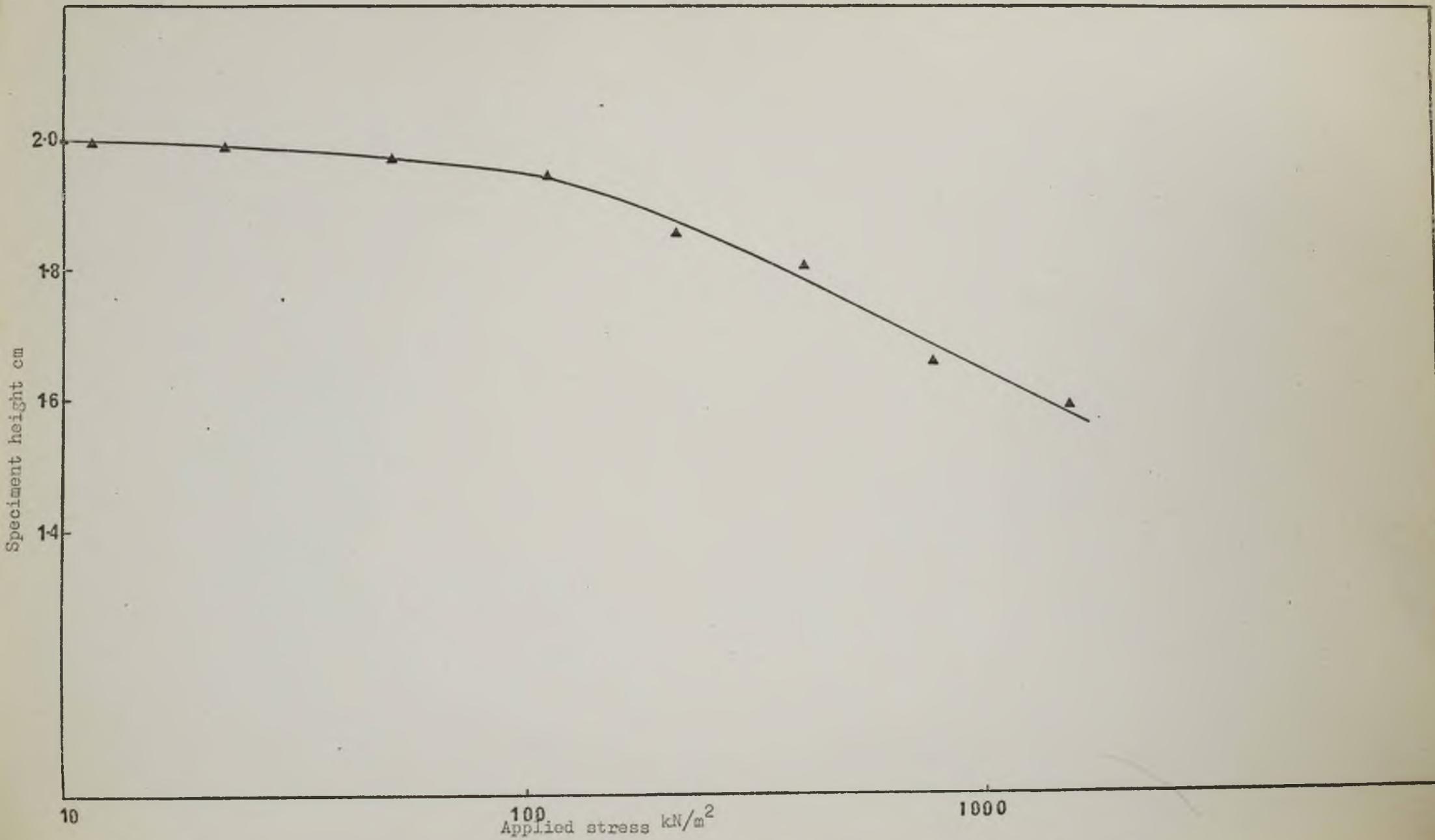


Fig. 7.20 Compressibility of Sape soil at 18% (optimum moisture content) in the oedometer tests.

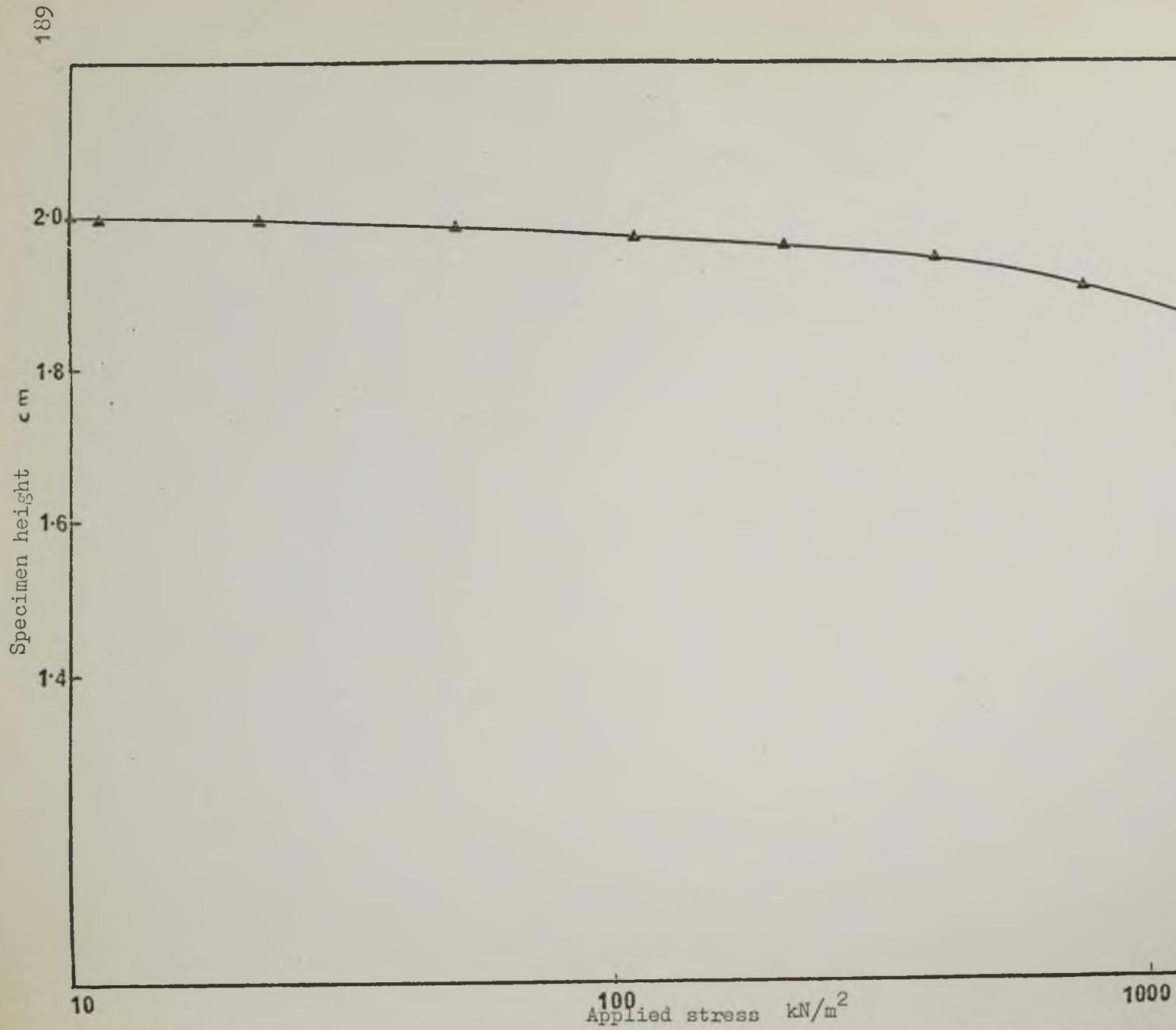


Fig. 7.21(a) Compressibility of Sape soil at 21.5% moisture content (wet-side) in the oedometer tests.

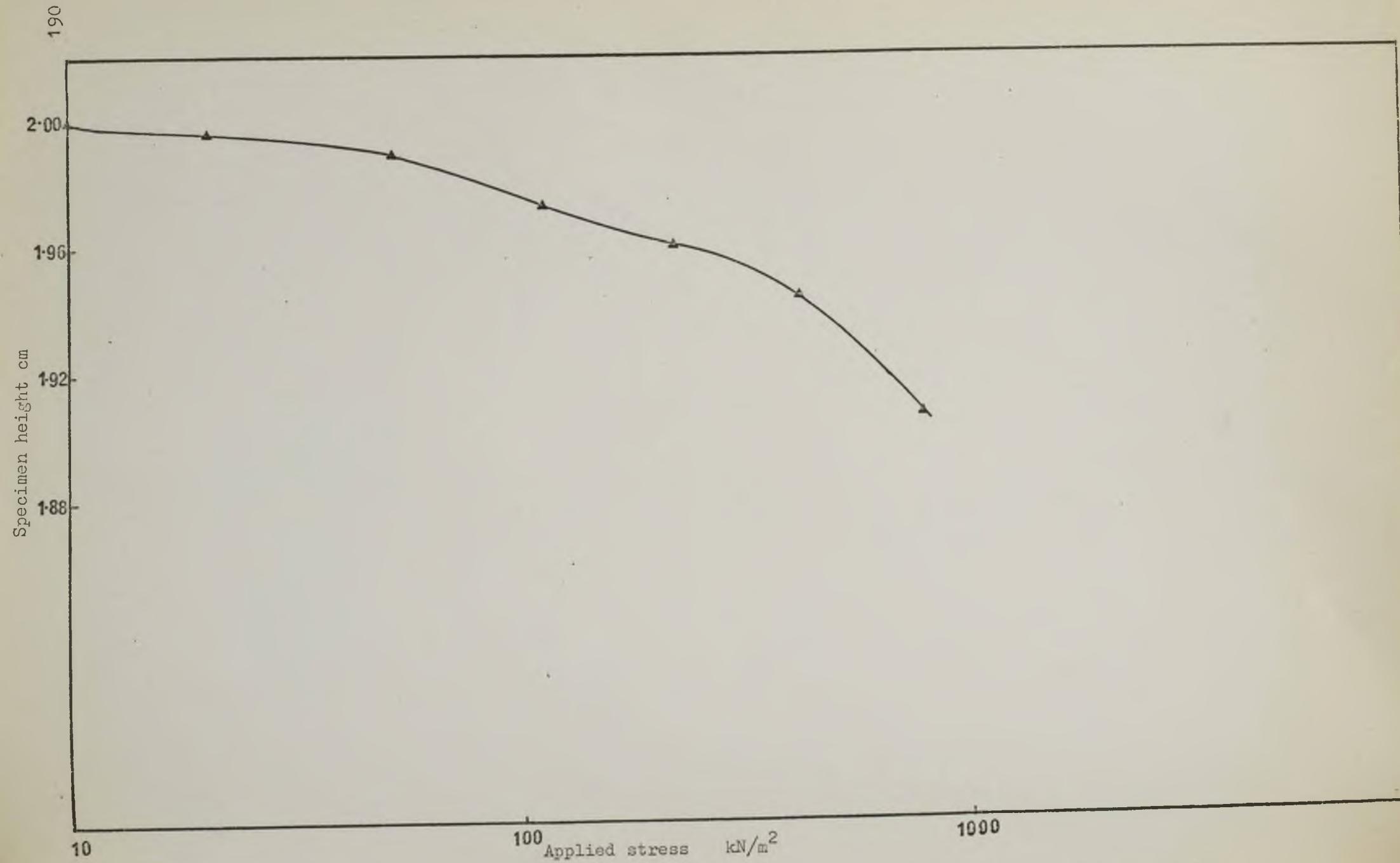


Fig. 7.21(b) Compressibility of Sape soil at 21.5% (wet-side) plotted on a different vertical exaggeration showing non-linearity of Fig. 6.20(a).

Papua New Guinea. They all reported the existence of low compressibility at low applied pressure up to a "critical pressure" beyond which the soils become more compressible. This behaviour was compared to over-consolidation in natural clays, which Wallace interpreted in terms of cementing bonds between soil particles in the soil structure which has to be broken before the linear relation between voids ratio and log of applied pressure can exist.

Figures 7.19, 7.20, 7.21 show that the soil is least compressive when compacted on the dry side and most compressible when compacted at optimum moisture content. This is very unusual behaviour since the density was highest at the optimum moisture content. Furthermore, the compressibility on the wet-side was higher for that on the dry-side which should be of higher voids ratio.

Matyas (1963) has reported that the compressibility of compacted Sasumua clay was higher on the wet-side than on the dry-side. He noted the apparent disagreement of this with Lambe's (1958) concept of the clay structure on the wet-side being dispersed and on the dry-side being flocculated.

The void pattern and the soil grains, as observed in the microscopic examination of structure (Chapter 8) is not simple. Complex juxtapositions of soil grains, pores of various sizes, channel fillings and crystallising material exist and a simple mode will not be expected to apply. However, it seems that the compressibility is also controlled by factors other than the void ratio and applied pressure, examination of which will be considered later.

7.3 Discussion

7.3.1 Direct Shear:

The tendency for volume increase during shear is shown by the specimens compacted at the dry side and for higher moisture contents up to about optimum moisture content. Beyond this a change is recorded and the specimen decreases in volume as the test progresses (see Appendix B).

For some samples the effect of the applied load on the volume-strain behaviour is easily shown by the switch from a dilatant behaviour at low pressures to one with decrease in volume at high pressures.

It is clear from the foregoing that the stress-strain behaviour and the volume change-strain behaviour are functions of both the level of stress and the moisture content

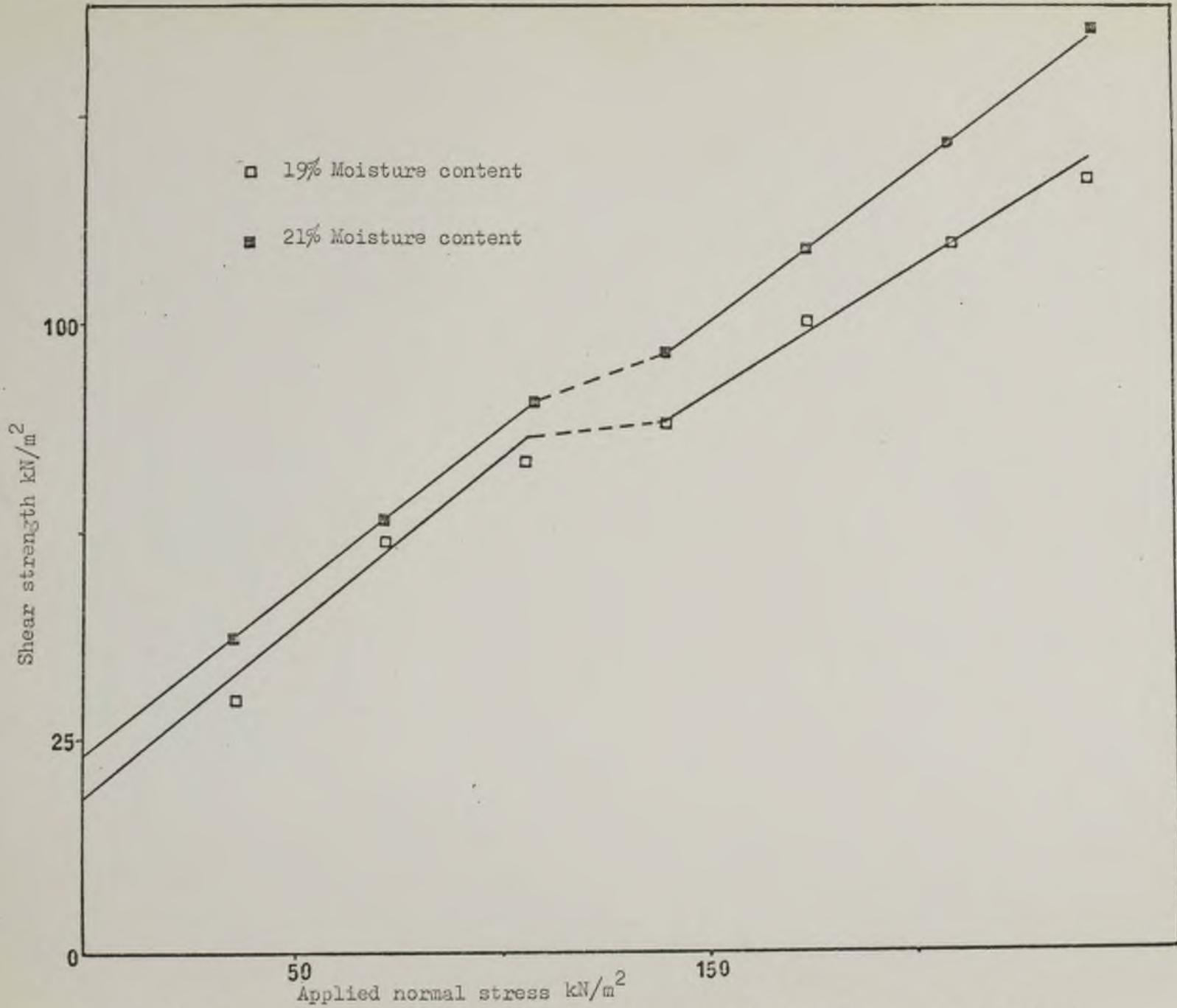
$$S = f(\sigma, w)$$

The measured value, or the observed behaviour is dependent on an interplay between moisture content and stress level. The relationship of each with moisture content and with one another is not linear.

Figure 7.7 shows the plot of peak strength (τ_{\max}) versus moisture content for tests carried out with the same values of normal load. (Note: τ_{\max} is taken as ξ at 10% strain for cases of no distinct maximum). τ_{\max} increases with increase in moisture content up to about optimum moisture and later decreases on the wet-side of optimum.

The plot of τ_{\max} versus normal load for the same moisture contents Figure 7.22, show points that are distributed about two non-colinear lines. One existing at lower values of normal load and another with a slightly lower slope at higher values.

This relationship of τ_{\max} with normal load and with moisture content earlier mentioned points to the existence of a level where a



Shear stress at failure is the max. shear stress or shear stress at 10% strain for cases of no distinct maximum

Fig. 7.22 Shear strength versus applied normal stress for higher values of applied normal stress in the direct shear tests. Same soil.

rather spurious strength response is given with respect to moisture content or normal stress.

The dependence of the soil behaviour on the level of stress has also been observed by Lohnes and Demirel (1973) for direct shear tests, carried out on undisturbed samples. They explained this in terms of a collapsible structure which is resistant to loads at low normal stresses and "fails" at high normal stress. They identified a 'collapse pressure' as the average between the highest value of normal stress for the material to behave in a manner similar to over-consolidated clays and the lowest value of normal stresses for the material to portray a behaviour associated with normally consolidated clays.

Since these soils are remoulded, the idea of dislocation of continuous network of skeleton, usually suggested for soils in-situ does not appear to be directly applicable, however, the idea of change in the internal property of the material due to stress application cannot be dispensed with, if it is remembered that a real particulate nature does not exist in these soils and that the cemented aggregates can break down under stress, more so with the presence of moisture, as was the case when nodules were fractionated in tests carried out to determine the nature and distribution of the chemistry and mineralogy of the aggregations (see Chapter 5).

The dependence of the strength on the level of stress suggests that though the "collapse" observed by Lohnes and Demirel (1973) could be due to breakdown of skeleton, the breakdown of aggregated soil particles also occurs during loading and this can greatly affect the strength.

The change from over-consolidated clay behaviour at low moisture content to normally consolidated clay behaviour at higher moisture content points to the occurrence of particle degradation. The phenomena of breakdown of aggregations under stress occurs more readily with

increase in moisture content and specimens with higher moisture content would be expected to disaggregate more than those compacted at lower moisture content.

However, it may well be that the very compaction, at different moisture content, also imposes particular structures to particular moisture-compaction condition, and the difference one is observing is not only due to the breakdown of aggregations as suggested above, but to basic differences in the compaction-determined structure.

To test the idea of "collapse" or breakdown, the shear strength of the material from Sape was determined at two moisture contents while the range of normal stresses was widened to cover the "collapse region". An attempt was made to draw the Mohr-Coulomb failure line between the strength values. The plots are shown in Figure 7.22, and show that for a particular moisture content, a single failure line cannot be drawn through these points. There are two failure lines, one exists at lower normal stresses and the other at higher normal stresses. A range (rather than a point) of normal pressures exists which can be equated to the collapse pressure of Lohnes and Demirel.

Figure 7.23 shows the plot of initial vertical deformation versus normal stress causing it. For specimens loaded at low normal pressures, amount of compaction and therefore disaggregation is low and the effect of breakdown will not be very apparent on the shear strength. For specimens loaded above it, the effect of breakdown on the structure becomes manifest on the shear strength of the soils.

The triaxial tests, to be described later, were carried out, partly to observe the effect of this non-conformity to particulate nature, (due to comminution) on the shear strength.

Bishop (1966) has shown that for sands at high stresses, a similar behaviour occurs, i.e. strength being dependent on the stress level.

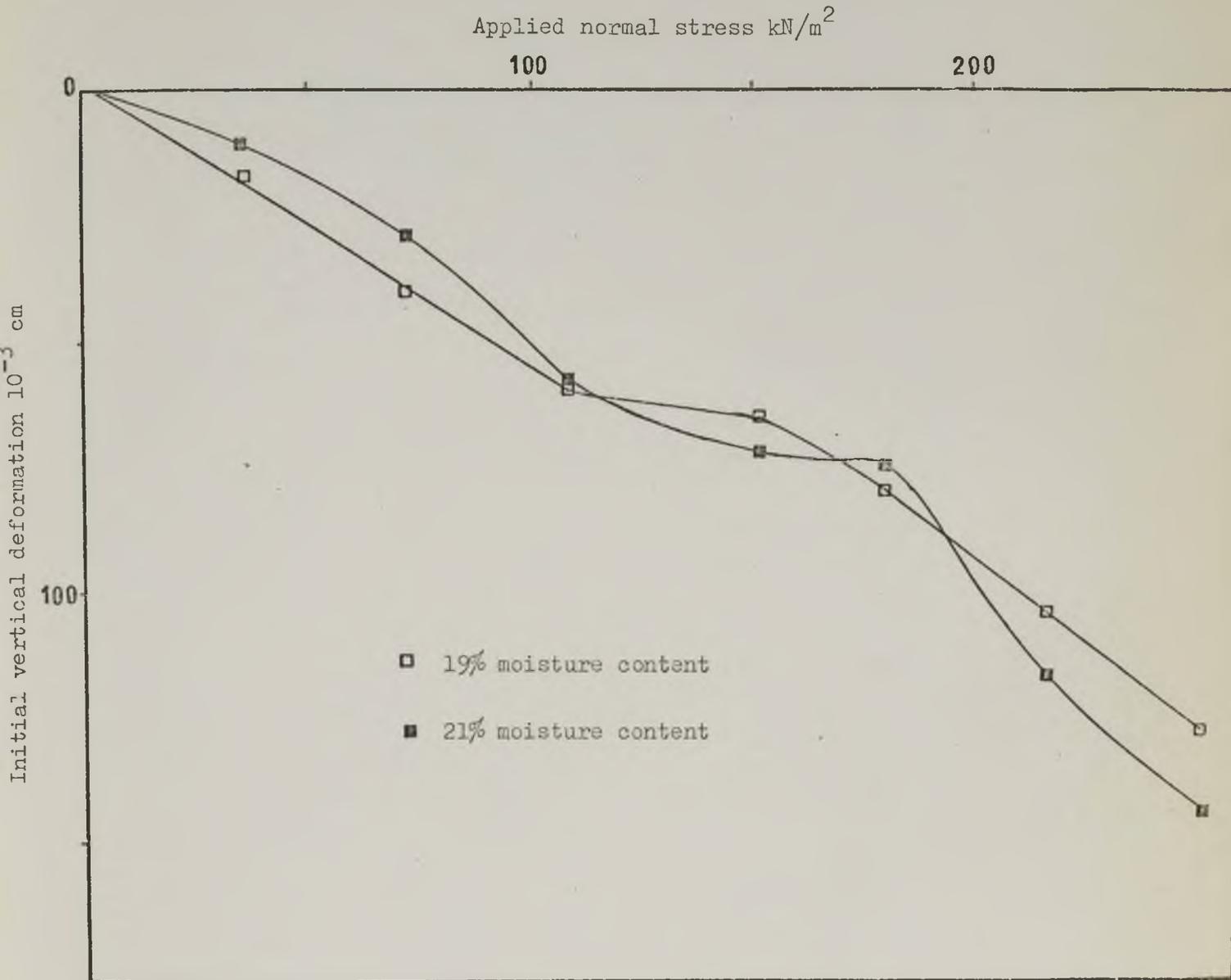


Fig. 7.23 Plot of Initial vertical deformation versus applied normal stress for Sape soil in the direct shear test.

This he also attributed to the crushing of particles that takes place at these high stresses. The existence of crushing in loaded soil has been confirmed by the works of Roberts and de Souza (1958) and Lee and Farhoomand (1967). The extent of crushing-produced compressibility will be expected to influence the strength properties.

The transition from granular behaviour to that of a more plastic material occurs in a sequence of loading for a moisture content. Since the change can be accomplished by the increase in moisture at constant load, the breakdown or transition is a function of moisture content and stress level. A particular combination of stress level and moisture content is required to produce the change. A value of stress for increasing stresses and constant moisture content and a particular value of moisture content for increasing moisture content and constant stress.

This value for the different test loaded at the same test conditions varies mainly because of the dependency on the type of bonds and the structural development in-situ, in terms of the pore spaces, aggregated soil particles and links between them.

The linkages between the aggregated bodies is of paramount importance in this consideration for it is these that impart the "unusual behaviour". At the start of the loading the particulate assemblages and true particles are presumably mostly intact. The breakdown can be associated with the compressibility, interlocking and stripping that takes place in the failure zone of a granular material. The disaggregation of the assemblages in contact produces materials of lesser sizes, although still granular (evidence for continuity of aggregation is presented in Chapter 8). The smaller aggregations produce larger surfaces and more contact areas which results in a decrease in the effective value of normal pressure and the shear strength will therefore decrease.

Loads higher than the "critical" value of normal pressure for disaggregation would produce a lower value of shear strength than is expected.

Breakdown of an assemblage of aggregated particles will lead to an increase in the surface area, reduction of size and consequently, increase in the co-ordination number in a packed system.

The increase in co-ordination number means an increase in void ratio since for ideal simple packings

$$N = 26.486 - \frac{10.726}{\eta}$$

N = Co-ordination number

η = porosity

Porosity is related to void ratio by $\eta = \frac{e}{1 + e}$

Mitchell (1976, p.141) has suggested that random packings of equal spheres can be considered to be composed of clusters of simple packings, thus by looking at simple packing systems, i.e. cubic, tetrahedral, etc. one can gain an insight into the amount of pore space for less ideal systems.

The increase in surface area and therefore co-ordination number means that more grains are effectively in contact and the intergranular stress will decrease, if one maintains a constant confining pressure in a triaxial test.

The breakdown of aggregations though likely to occur throughout the specimen as deformation takes place under load, will be most prominent in the failure zone, which is the direction of the maximum shear stress. The effect of loading therefore is to comminute the aggregations along the shear failure zone and a granulating or crushing effect is produced.

Thus it is possible to formulate the following: In a compacted residual soil, two structure-dependent systems exist:

1. The fabric of the soil skeleton consisting of particles, pore spaces and juxtaposition of these which is dependent on the conditions of the compaction, i.e. moisture content and energy (mode and amount).
2. The internal fabric of the "particle" which in most cases is an assemblage of the smaller particles whose relationship to each other and to the pore spaces is dependent on the structure in situ and is defined by the soil forming parameters attendant during formation of the soil.

In the case of an ordinary soil, the strength of the compacted soil would depend on the structural system 1.

For a residual soil, the stability of the assembly of particles is pertinent to any considerations of strength. Its existence in variable degree of stability have profound effects which can be manifest in the mechanical behaviour.

If two test specimens produced by compaction at the same moisture content present different behaviours just because of failing under different normal loads, such change is most likely to be due to changes in the second structural system than in the first.

Nova Floresta soil compacted at optimum moisture content show this change in behaviour for different normal stresses (see Figure 7.5). For samples loaded at low normal stresses, the shear stress in the stress-strain diagram, increases with deformation reaching a peak value and subsequently decreases with increasing shear strain. At higher normal stresses the shear stress increases with deformation and continue to increase till failure.

The effect of the second structural system, which can be referred to as "second level structure" probably explains the comminution of the particles. Apparently a particular level of stress is required for this breakdown to occur.

7.3.2 Triaxial:

The mechanical behaviour of these materials differs in behaviour from sand that has low cohesion, high permeability and high angle of friction despite the similarity on the basis of initial grain size distribution.

It does not agree with clay either, that has considerable cohesion, high compressibility and low permeability, despite the considerable amount of clay minerals present on the basis of mineralogy. At best, it can be referred to as a cross between these classical materials existing at two ends of a scale.

Since the study of the nature and the intrinsic properties of the soils show that they are largely composed of cemented aggregations (see Chapter 8) resulting in the elevation of fine-particle clay minerals into the sand-size ranges. The degree of stability of these aggregations, is therefore expected to determine greatly the mechanical behaviour of these soils.

The concept of breakdown suggested earlier, seems to explain the observed characteristics in that continuous collapse of particle grains will occur as failure progresses. The expected interlocking that would lead to a volume increase in a dense "granular" material, does not actually occur due to particle compressibility. The soil will therefore be compressed during failure.

7.3.2.1 Stress Paths:

The stress paths shown in Figure 7.17 show some interesting trends in that the low values of pore pressure result in high values of effective stresses even at low strains.

During the initial phase of the loading, the effective stresses are almost equal to the total stresses, i.e. almost all the load is being carried by the soil skeleton and little by the pore water even for a

saturated soil. This is a behaviour typical of a granular soil, where the pores are of considerable sizes and the pressure induced in the water due to low surface area.

With increase in loading, however, the effective stress paths reverse to one of decrease and a continual decrease in the value of q is recorded with increase in p .

This decrease, as can be seen from the stress-strain curves (Figure 7.14), is due to the change in the rate of increase of the deviator stress. The change from rapid rate of increase to a gradual one at strains about 3% is not commensurate with the increase in pore pressure and the effective stress therefore decreases.

The point of change in the value of effective stress in the stress paths is considered to be a good value for failure criterion of this soil. The presence of this point at values just below or about 3% strain, thus justifies the validity of the criterion of strain adopted.

7.3.2.2 Pore pressure developed during loading:

The dramatics shown by the deviator stress versus strain is generally not repeated here. The pore pressure increased steadily at a slow rate and continued to increase throughout the period of the tests (Figure 7.16). The change in the rate of deviator stress increase at about 2% strain is not reflected in the measured pore pressure. This behaviour and the fact that when the deviator stress changes to one of steady slow increase, this rate of change was usually lower than that for the pore pressure, has a considerable effect on the shapes of the effective stress paths.

As mentioned, specimen failure can actually be considered to have taken place just after the change in slope of the deviator ^{stress} versus strain curve.

After the change in the rate of deviator stress increase, most of

increase in total stress results in an increase in pore pressure and the effective stress decreases.

The measured pore pressure is an overall summation of the pore pressure in the whole specimen. The difficulty of knowing the true picture of what is happening in the failure zone, therefore exists. It is suggested that the crushing of porous materials and the subsequent exposure of fine voids at failure is responsible for the non-dramatic increase in pore pressure at failure.

The possible existence of two void types within two fabric systems has been suggested (see 7.3.1). The high suction capabilities of the small pores within the second fabric system (p.199) will result in only a gradual increase in the measured pore pressure.

With respect to the moisture density curve, the pore pressure developed at any particular value of strain (see Appendix B (stress paths)) increases with molding moisture content. This increase is most likely to be due to the value of pore pressure parameter A which increases with increase in density.

7.3.2.3 Cohesion and Angle of Friction:

The plots of cohesion C' versus moisture contents in terms of effective stress for drained and undrained conditions are basically different (see Figures 7.10, 7.11). Under drained conditions the plot follows a similar trend to that of classical soils. For the undrained conditions C' increases slightly with moisture of compaction, decreases with further increase reaching a minimum value and later increases. Accepting that difference in magnitudes could exist, between drained and undrained conditions (though not to the extent shown) the further increase in cohesion, at moisture contents of about 21%, after the decrease in the undrained plot, is unusual.

A plausible explanation appears to be the already suggested concept

of disintegration, where smaller particles are produced due to comminution of the aggregation. The breakdown of the particles which results in the creation of finer particles and therefore more surface area, would lead to a more cohesive behaviour. This implies that more disaggregations take place for specimen compacted at or above 21% than below. This moisture content is therefore the threshold moisture content for the stress-range employed during the tests and confirms the direct shear observations, where the shear strength is dependent on moisture content.

The behaviour of ϕ^* determined from drained and undrained test is similar but not identical. The magnitudes are different though their relationship with moisture content follows the same trend. The earlier reasoning of comminution of particles can also be suggested here. The breakdown would create finer particles along the failure zone, which has the effect of reducing the previous amounts of interlocking and friction as well as creating systems with lower amounts of these properties. This reduction in interlocking will result in reduction in ϕ' .

7.3.3 Compressibility:

The general mechanical behaviour of these soils have been interpreted in terms of a collapsible "particle" influencing the stress-strain behaviour. This concept is readily applicable to compressibility. Previous investigations of particle breakdown have been recorded for granular materials (sand) at high stresses.

Terzaghi (1925^b) performed compression tests on sands and clays and observed that the compressibility of both types of soils was similar. However, the volume changes in sand were not as great as those in clay soil when tested under the same magnitude of load. Nevertheless, the sand was found to be quite compressible, even at pressures of the order

of 295 kN/m^2 (3 kg/cm^2).

Taylor (1948 p.217) has mentioned that in sands the major part of the compression takes place almost instantaneously and all but the final few per cent of compression takes place within the first few minutes (see Figure 7.24).

Rowe (1955), Roberts and de Souza (1958) have reported one dimensional compression tests on sand.

Lee and Farhoomand (1967) on the basis of work carried out on a granitic gravel reached a number of conclusions:

- (a) That granular soil is quite compressible under an applied stress.
- (b) Compression is usually accompanied by a certain amount of particle crushing and the two phenomenon can be seen to be somewhat related to each other.

Lambe and Whitman (1969) have mentioned that although for most engineering problems, the effect during compression of sands are of little practical importance, at large stresses, however, there is a significant time lag between application of stress and response from the material. They further made the very useful observation that though this occurs for most soils, at large stresses, it can occur at ordinary stresses for weakly cemented particles.

Roberts (1964) has produced curves of $\%$ 24 hr compression versus time to point out the time dependency compression that occurs in a granular material susceptible to breakage.

Lee and Farhoomand (1967) also mentioned that the particle breakage and crushing is time dependent and occasional cracking sounds of particle breakage was heard in a two-hour compression of granitic gravel.

The compression time characteristic determined from the compressibility are here presented (Figure 7.25) and reinforces the hypotheses that considerable particle breakdown occurs that affect the mechanical behaviour.

Percentage of Total Compression

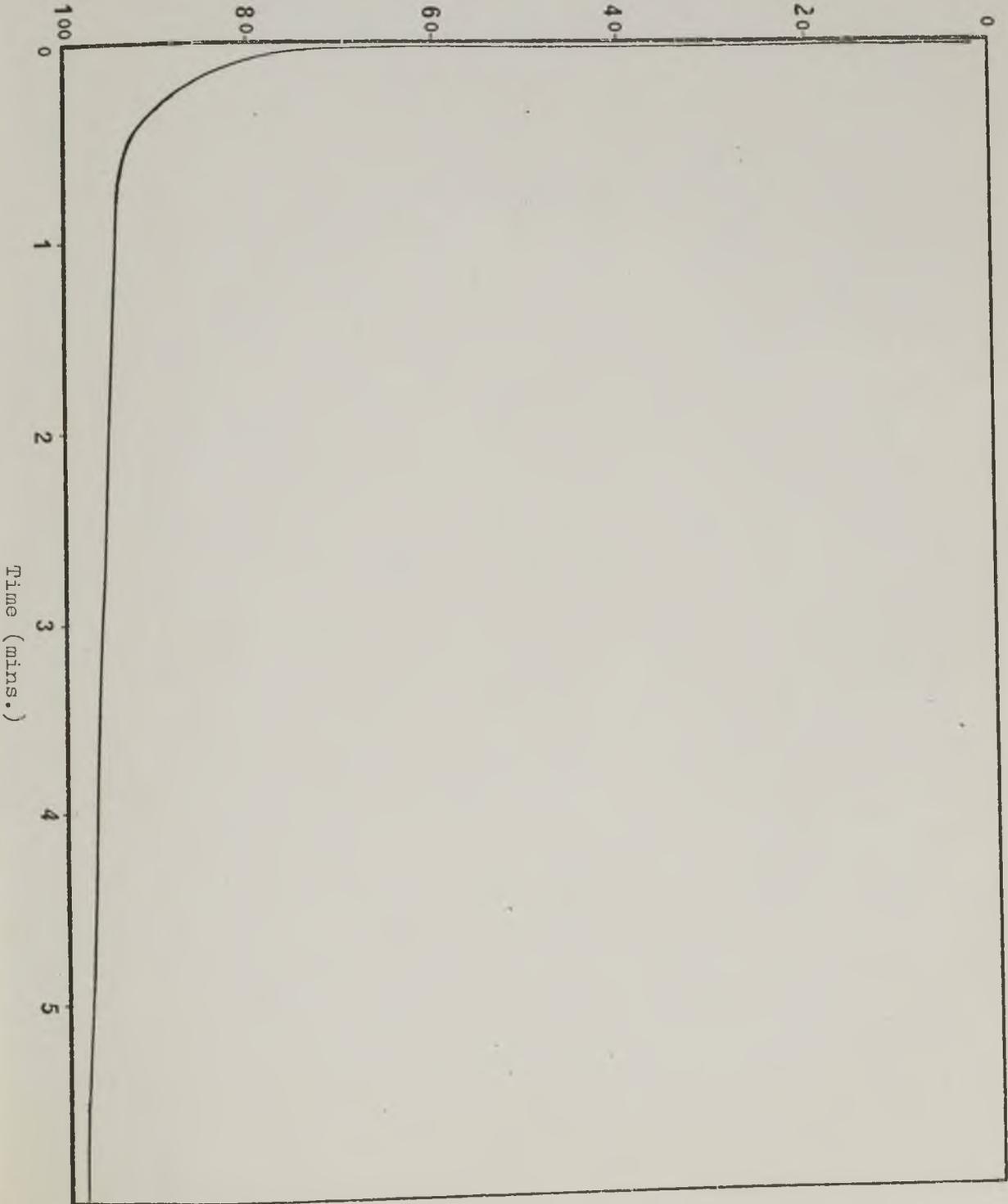


FIG. 7.24 Behaviour of sand under compression (Taylor, 1948)

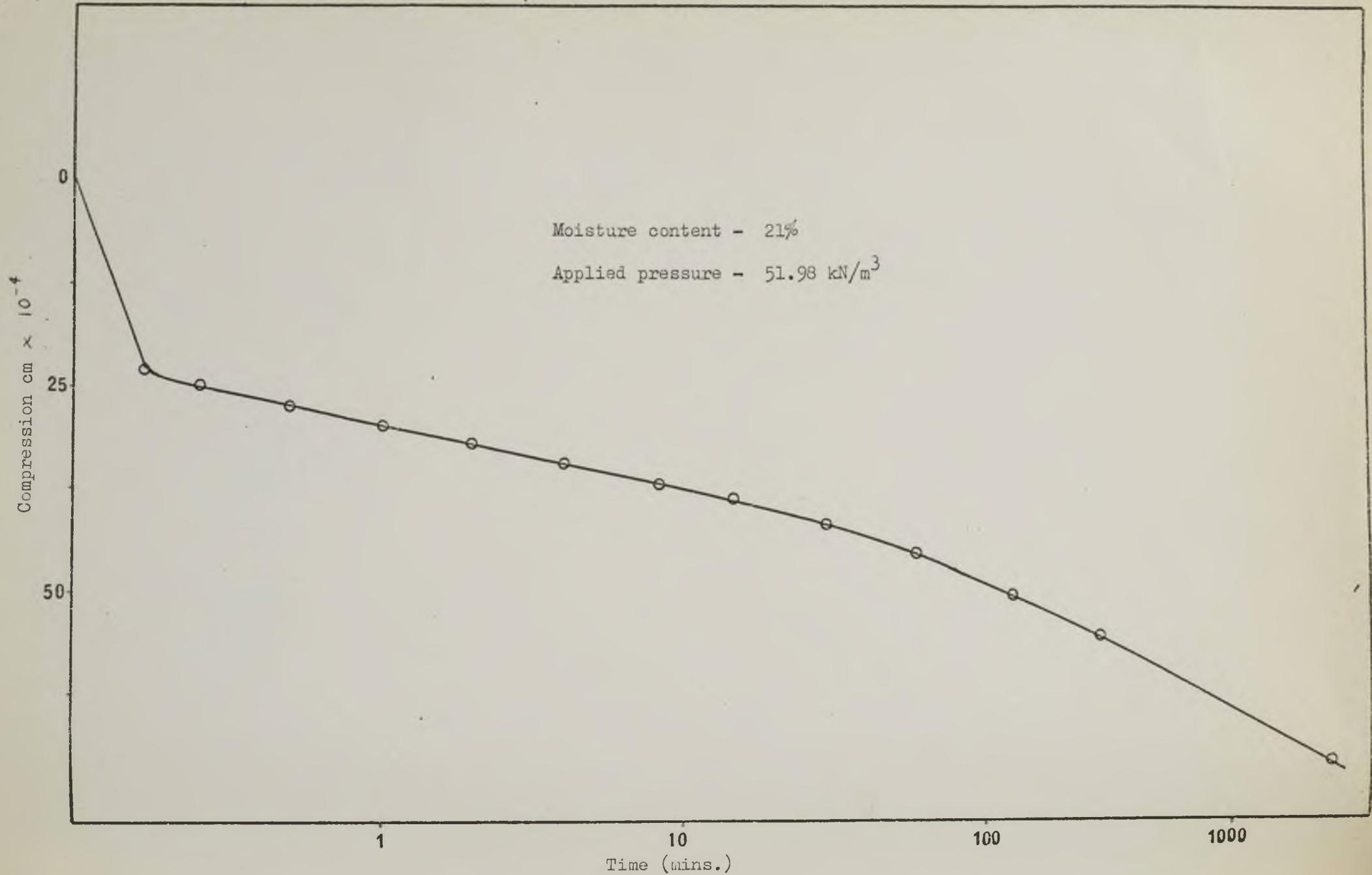


Fig. 7.25 Typical compression - time curve for Sape soil in the oedometer tests

The plot of % 24 hr compression versus time (Figure 7.26) shows that considerable time-lag exists, which can only be attributed to breakage and metastability as all structural rearrangement effects are expected to take place within the first few minutes.

This time lag exists at all applied loads and is found to decrease with increasing load. The plot of % of 24 hr compression that occurs after 1 minute readily shows this (see Figure 7.27). The time lag is least at optimum and highest on the wet side of the moisture density curve. This observation suggests that breakdown is aided by moisture, if the time lag is related to particle breakdown.

The compressibility results therefore also show that there is comminution and breakage of particles, which is similar to those reported by Roberts (1964).

Since comminution exists, the compressibility of the soil is just not dependent on the arrangement of particles (i.e. packing and void ratio) and pressure. The extent of resistance to comminution and the nature of the different voids and packing systems produced after substantial comminution will be important. For the same compactive energy, the soil is most compressible when it is densest. This suggests that greater comminution of particles takes place about optimum moisture content, which is in agreement with observations from the direct shear and triaxial test. The optimum moisture content or just above it, is thus the threshold moisture content for the compactive effort (static mode) used to produce the specimens.

The agreement of the direct shear, with the compressibility results, suggest that there is little difference in the soil behaviour, using different modes of compaction.

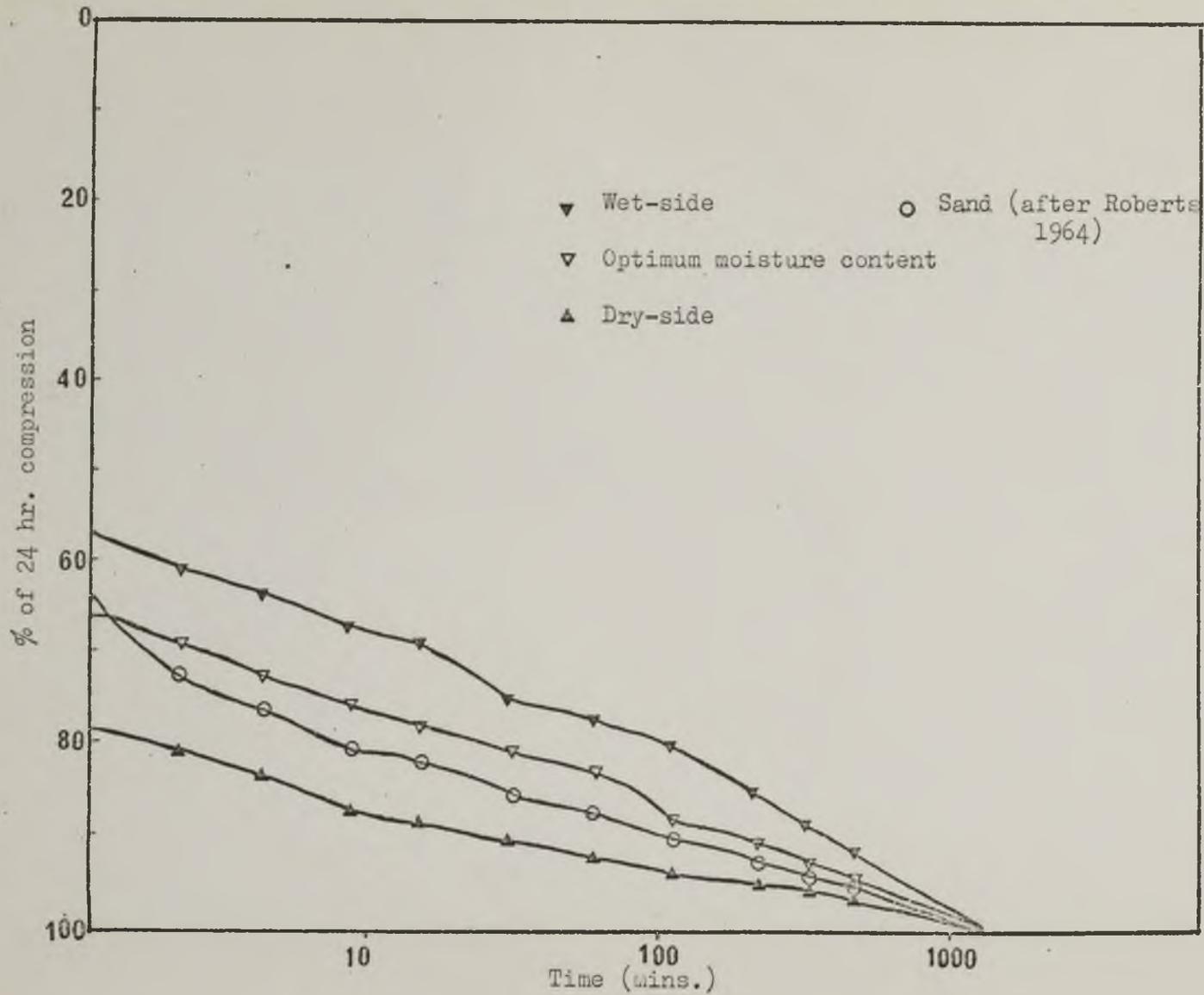


Fig. 7.26 Compression - Time behaviour of Sape soil in the oedometer tests.

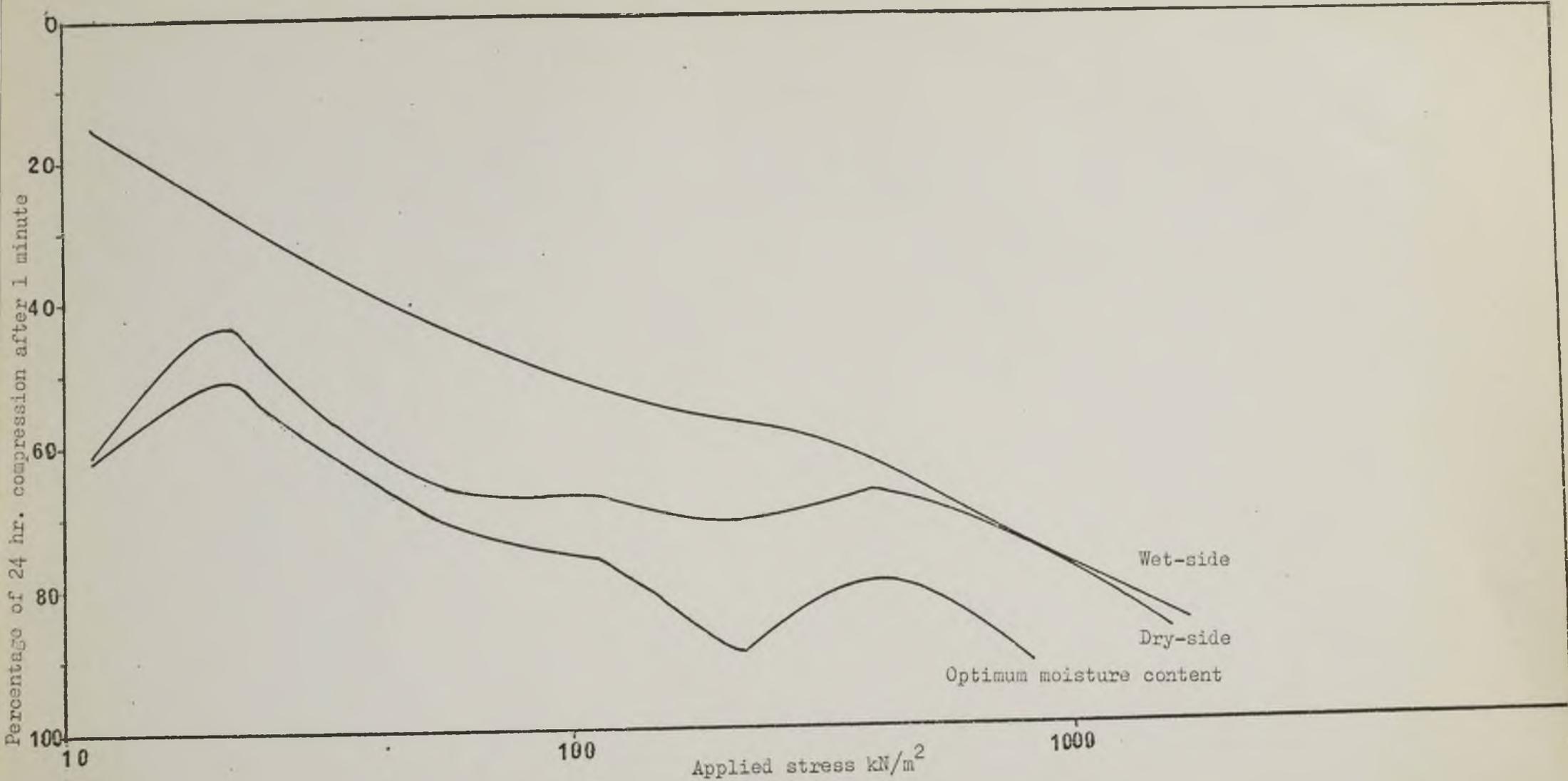


Fig. 7.27 Percentage of 24 hr. compression after 1 minute versus applied stress in the oedometer test.

CHAPTER 8

MICROSCOPIC INVESTIGATION OF STRUCTURE

8.1 Introduction:

The analyses of the form and distribution of kaolinite (Chapter 6) has shown that this clay mineral is present in all the size fractions of the Cuite, Nova Floresta and Sape soils.

It was concluded that the formation of large soil grains, involved the aggregation of the clay minerals and other clay sized particles, e.g. iron oxide into larger sizes. This conclusion was drawn from two observations:

- (i) The existence of alumina only in the form of the alumina-silicate, kaolinite; this was demonstrated by X-ray analysis.
- (ii) The high values of the clay mineral content as determined from mineralogical analysis. These values were far higher than the clay size content obtained from particle size analysis.

Similar conclusions concerning the presence of clay minerals in the large sized fractions can also be reached based purely on the strength behaviour. For example, the compressibility characteristics were shown to be in agreement with those obtained by Lee and Farhoomand (1967); and Roberts (1964). These investigators published the reports on the compressibility characteristics of soils where particle degradation was present. Similar effects were recorded (see Figures 7.25-7.27). The conclusion that follows the similarities shown in the figures, is that degradable soil particles are present in the Sape soil. And, if degradable soil particles are present, they can only be as aggregations, since clay makes up the bulk of the soil.

In the light of these conclusions, discussed in Chapters 6 and 7,

it is desirable that the actual physical appearance of the soil particles be investigated. Microscopic investigations were therefore carried out to reveal the nature of the aggregations and their spatial juxtaposition.

Two forms of microscopy were carried out: Optical Microscopy and Scanning Electron Microscopy (SEM).

The optical microscope used is a Swift polarising microscope that had a Bertrand lens and facilities for light filtering. Facilities for photomicrography were not readily available. An adaptor, designed by Mr. R. Duxbury, technician, Department of Civil Engineering, was used. This adaptor could be screwed on to a single lens reflex camera and to the cylinder holding the eye-piece of the microscope. The procedure adopted for the preparation of sections is given in Appendix A).

The scanning electron microscope employed is a Cambridge Instrument 'Stereoscan' scanning electron microscope. The procedure adopted for the preparation of the specimens is given in Appendix A.

The rigorous analysis of the microstructure of the soil as viewed under the electron microscope is however hampered by the reason of coating of surfaces by iron oxide. In addition to the cementation of clay-sized particles to form larger soil particles, there is massive coating of soil particles by iron oxide. This coating hinders the analysis of the relationship between the cement and the clay-sized particles that are cemented.

Scanning electron micrographs of red soils presented to date, usually show strongly cemented particles that have been massively coated. Examples include those of Townsend and Reed (1971), Lohnes and Demirel (1973).

8.2 General Comparison of Cuite, Nove Floresta and Sape Soils:

8.2.1 Optical Microscopy:

Air drying after exposure of the soil in situ produced some degree of hardness in both concretions and fines of Chapter 5. The pieces used for sectioning were sampled in such a manner that it would include both the concretions and the fines. In the following description, Plates 1-5 refer to Nova Floresta soil, Plates 6-13 Cuite soil and Plates 14, 15 to Sape soil. Many of the features that will be described are represented in the three soils. The micrograph of where they are best represented is, however, presented. Attempts were made to avoid duplication wherever possible. For example, in describing features common to all the soils, Plates 1-5 of sections from Nova Floresta will be used considerably. Features shown in these plates are also present in Cuite and Sape soils, they are not presented for the reason of duplication.

The features common to the three soils are the cementation of clay mineral particles, the coating of the cemented clays and the concentration of iron oxide in certain parts of the soil, best expressed by Plate 4. The concentration leads to the segregation of these parts (central patch Plates 4-5) from other parts that have lower concentration of iron oxides (bodies surrounding central patch Plates 4 and 5).

Under the optical microscope the Cuite, Nova Floresta and Sape soils are seen as round cemented bodies of various sizes. Voids present are also of different sizes, with irregular shapes and orientation (Plates 1-15).

Apart from the size of the bodies, little or no difference exists between the hardened part of soil in situ (concretion) and the earthy matrix (fines). The concretions have a greater proportion of large, round cemented bodies, while small ones predominate in the fines. The larger bodies have a higher concentration of iron oxide than the smaller

PLATE 1 x 0.5

A piece of the hardened part of Nova Floresta soil, showing dark discrete bodies (usually larger than 2mm in diameter). The brownish porous regions between the discrete bodies are iron coated clays that are cemented in place. The light yellow to whitish areas are ochreous fillings of clays with iron oxide stains.

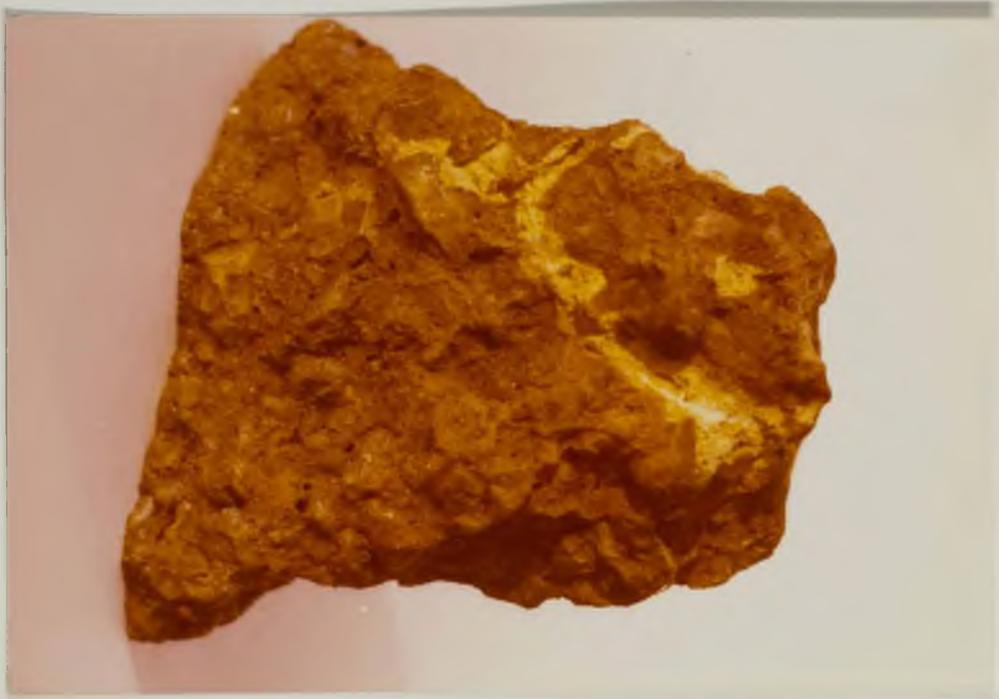


PLATE 2 x 80

Section showing the porous regions of hardened Nova Floresta soil. (This is the general appearance of the porous regions for the three soils investigated).

- (a) - Quartz particle.
- (b) - Quartz-centred clay bodies with iron oxide coating (see text).
- (c) - Iron oxide-centred clay bodies with iron oxide coating.
- (d) - Clay bodies with iron oxide coating.
- (e) - Iron oxide bodies.

The brown material that surrounds and links all the bodies is goethite.

PLATE 3 x 80

Plate 2 under crossed nicols. The different semi-spherical bodies described above become more easily differentiable.

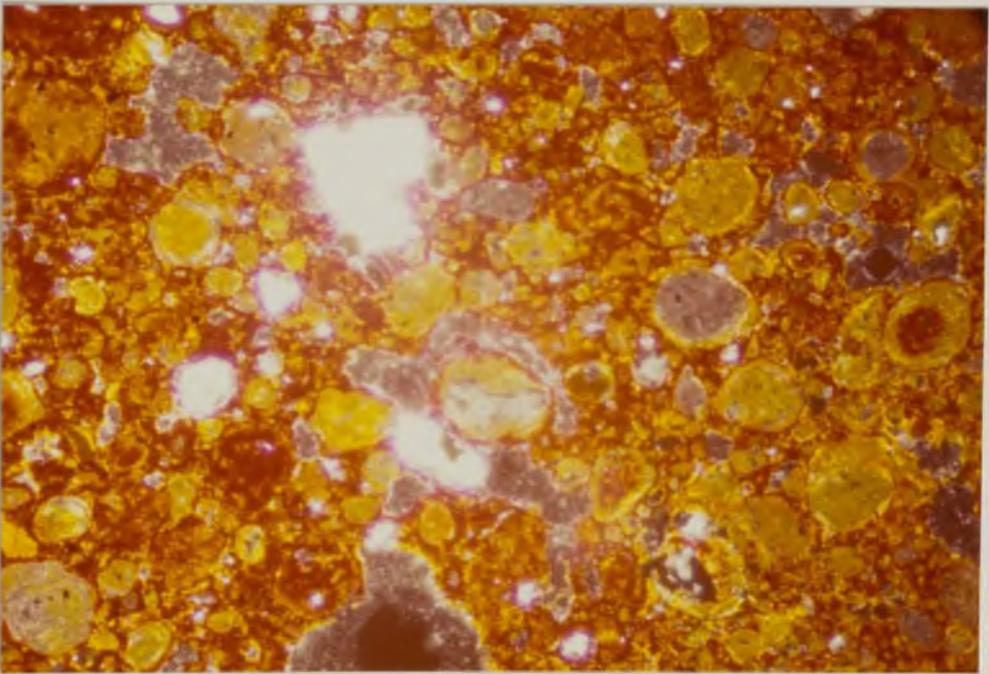
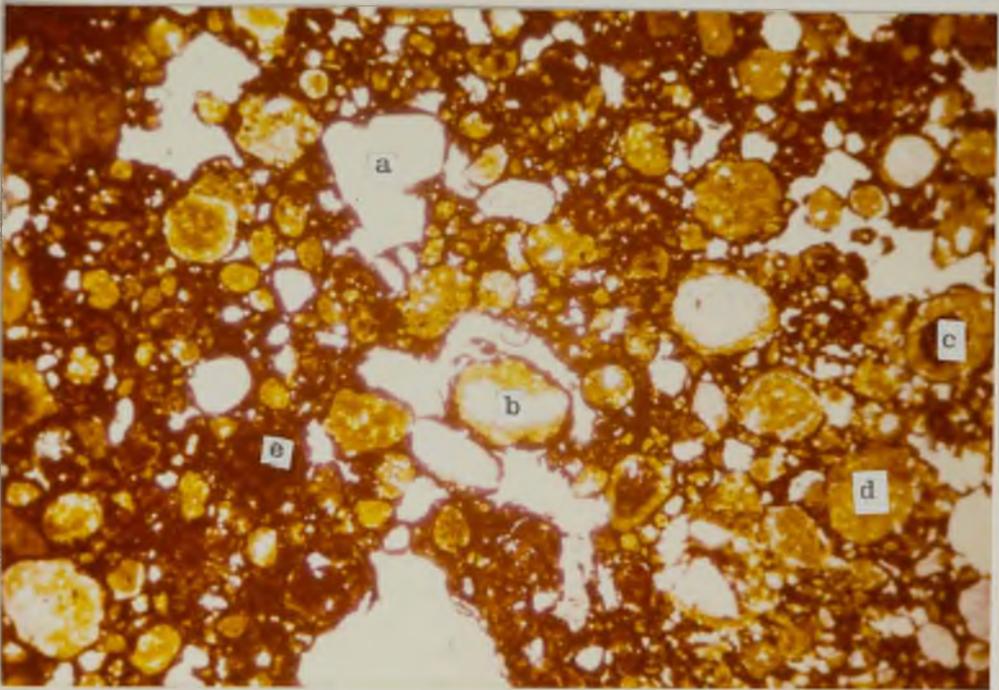


PLATE 4 x 80

Section showing dense area (same as discrete bodies of Plate 1) in Nova Floresta soil. The bell-shaped dense area (central yellow patch) is surrounded by smaller bodies of porous regions.

PLATE 5 x 80

Plate 4 under crossed nicols. The dense area can be seen to consist of cracks filled by yellowish material (identified as goethite).

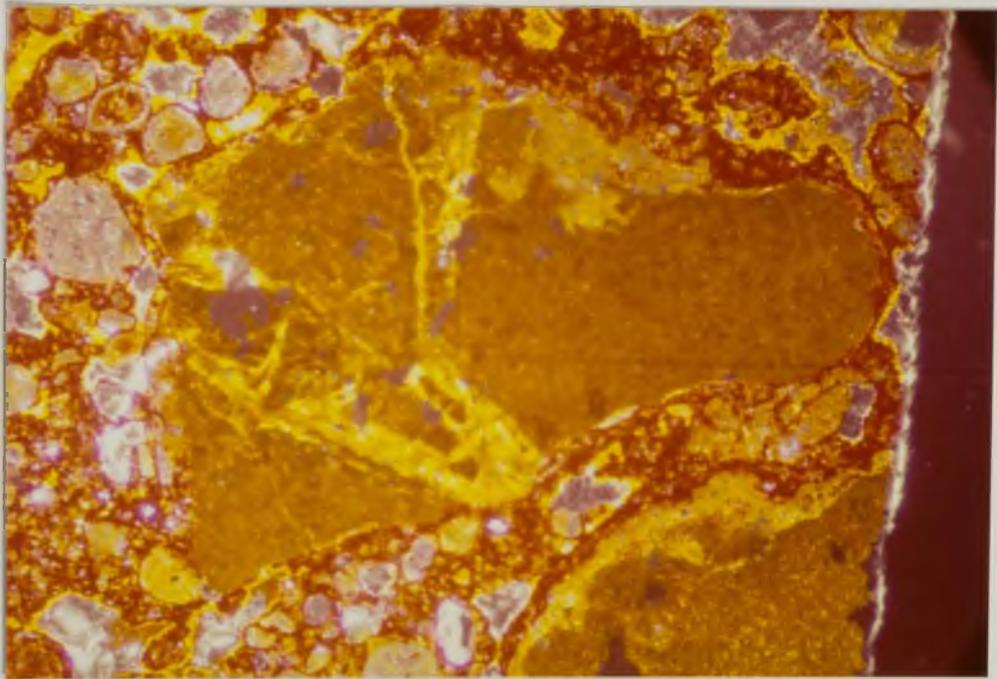
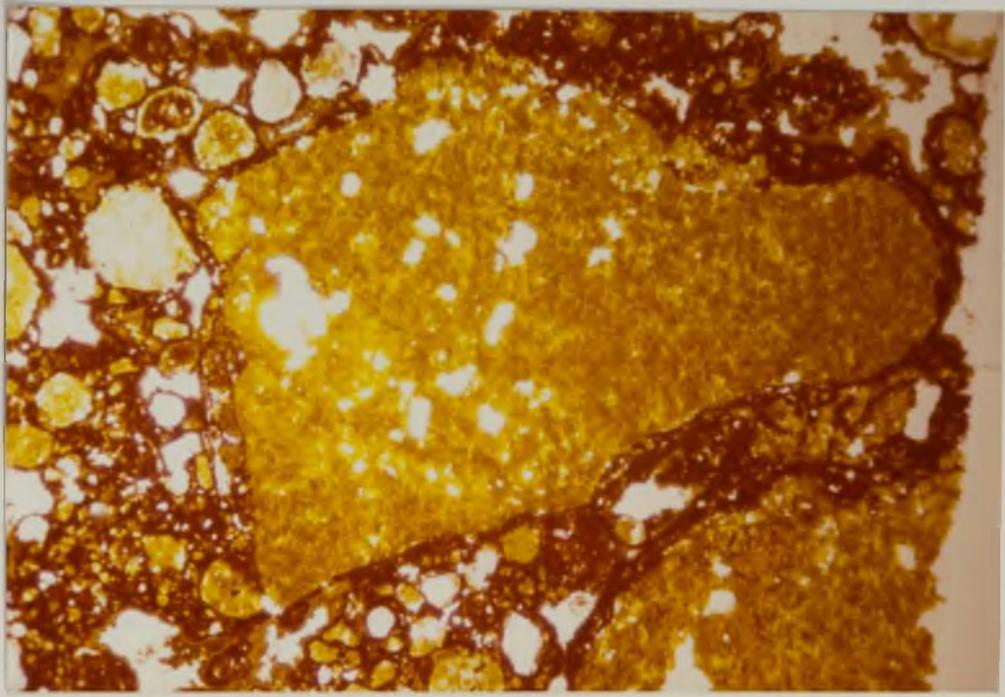


PLATE 6 x 0.5

A piece of hardened Cuite soil showing dark brown discrete bodies, highly porous (vesicular) brown regions surrounding the discrete bodies and light brown to yellow ochreous clay fillings.

PLATE 7 x 160

Micrograph of a dense area in Cuite soil. The Y-shaped feature is a channel partly filled with iron oxide (identified as goethite). Iron oxide is moving from the channel into the dark brown areas. The dark brown areas are clays with iron oxide coating and the white areas with blue edges are quartz particles. Other white areas are voids.

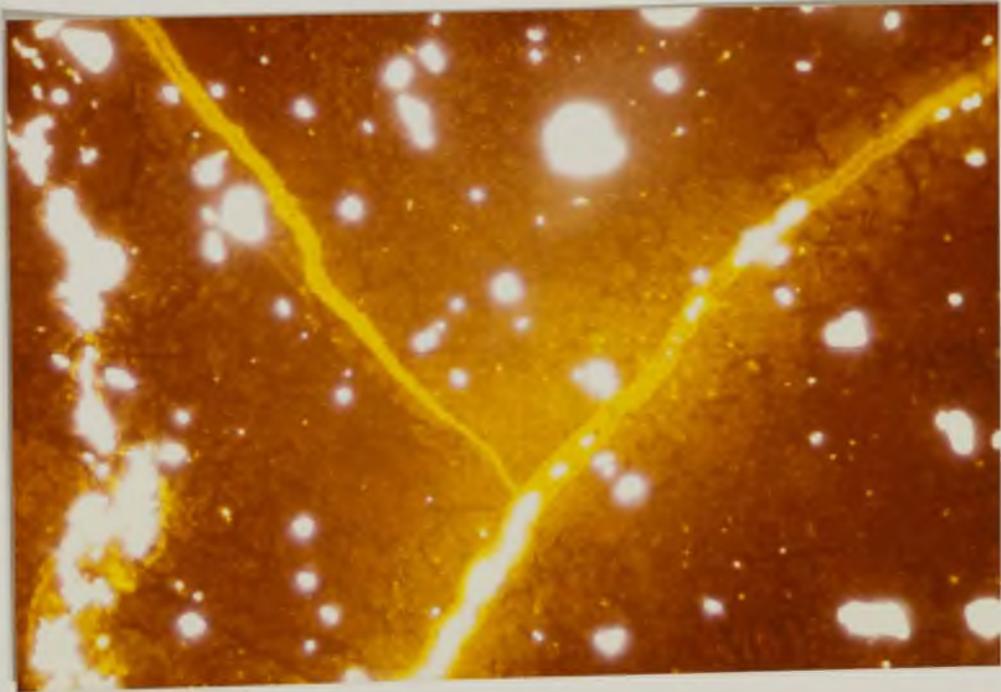
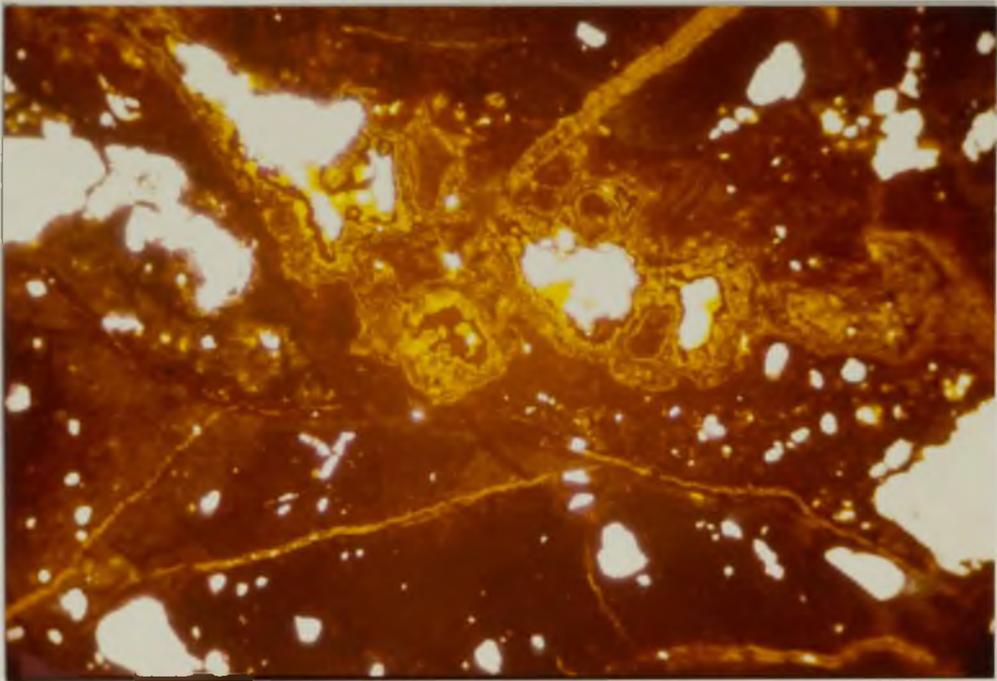


PLATE 8 x 80

Layered deposition and partial filling of a void in Cuite soil. The dark areas are iron coated clays with a high level of coating. The void is the yellow area in the central part of the micrograph. Yellow material is identified as goethite and red material as haematite.

PLATE 9 x 160

Micrograph of dense and porous regions of Cuite soil. The dense region (lower part of the micrograph) is surrounded by porous region (upper right hand corner). Inward movement of iron oxide (towards the centre) in the dense region result in the production of the brownish yellow band (top left-hand corner to bottom right-hand corner). Note the differences in the distribution of voids (white areas without blue edges) between the dense and the porous regions.



— Porous
region

— Dense
region

PLATE 10 x 80

Micrograph showing the incipient formation of a discrete body in Sape soil. Segregation of regions into dense and porous ones is less distinct here. The yellowish brown area at the centre is the incipient discrete body. The white patches within this area are quartz particles, which will probably form nuclei for iron oxide deposition at later stages. The brown area surrounding the yellowish brown area is made up of iron oxide coated clays. Note the cracks that separate the areas.

PLATE 11 x 80

Plate 10 under crossed nicols. The 'uniformity of the incipient discrete body is more clearly seen.

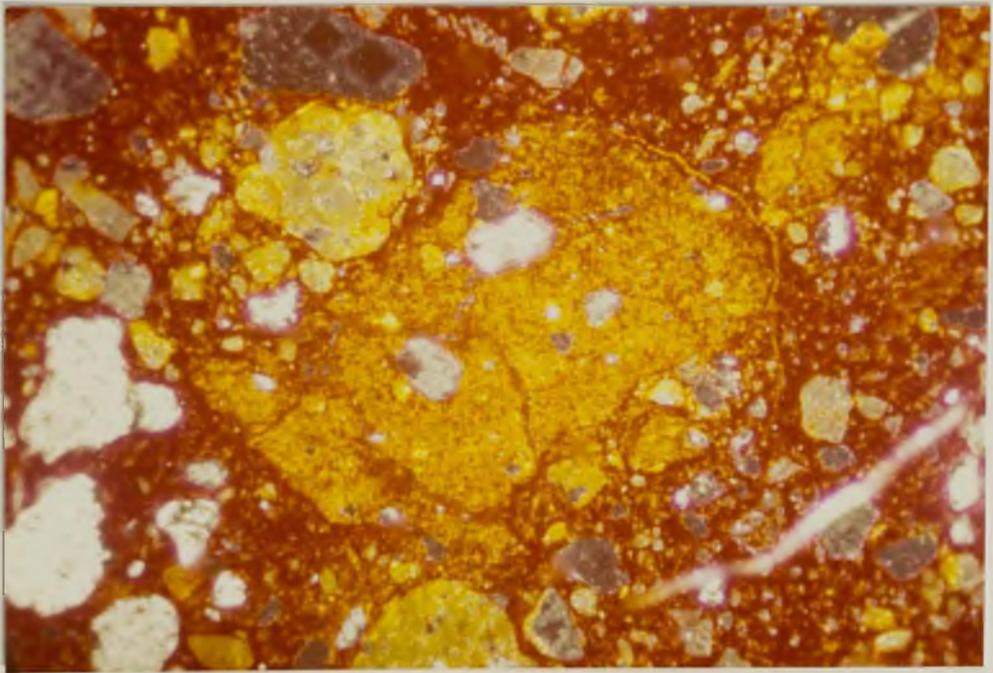
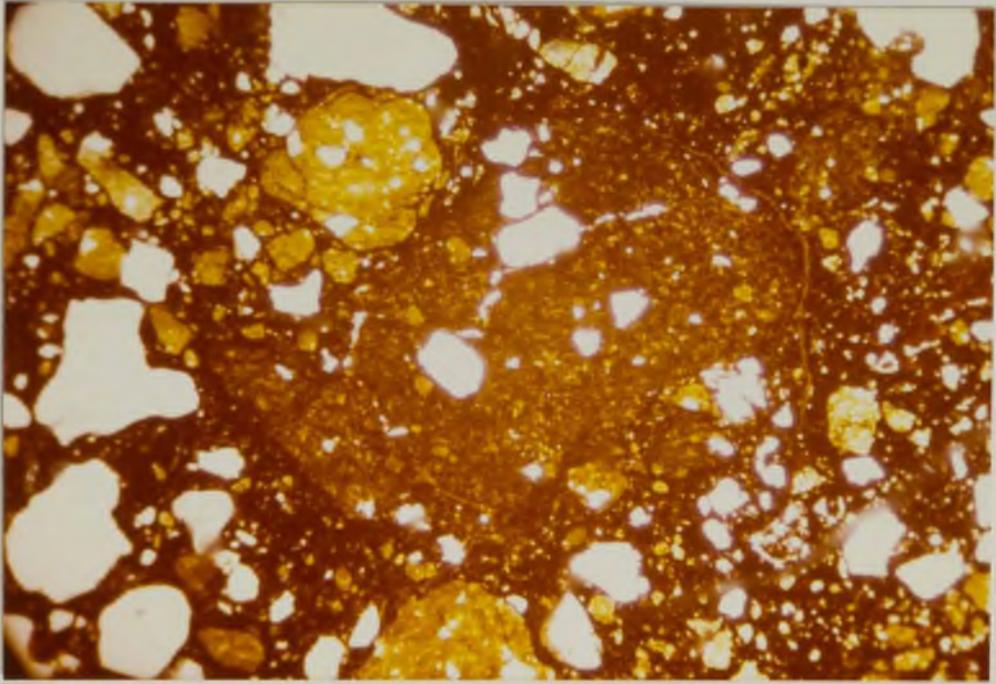


PLATE 12 x 80

Newly formed quartz, wholly occupying a void in Cuite soil (dense region). The quartz particle is formed by inward growth from silica deposited on the surface of the void. The dark areas are iron coated clays.

(q) - Newly formed quartz particle.

PLATE 13 x 80

Plate 12 under crossed nicols. The different mineral grains within the particle can be seen. Note the cracks that are concentric about the quartz particle. These cracks are due to stresses accompanying the total filling of the void.

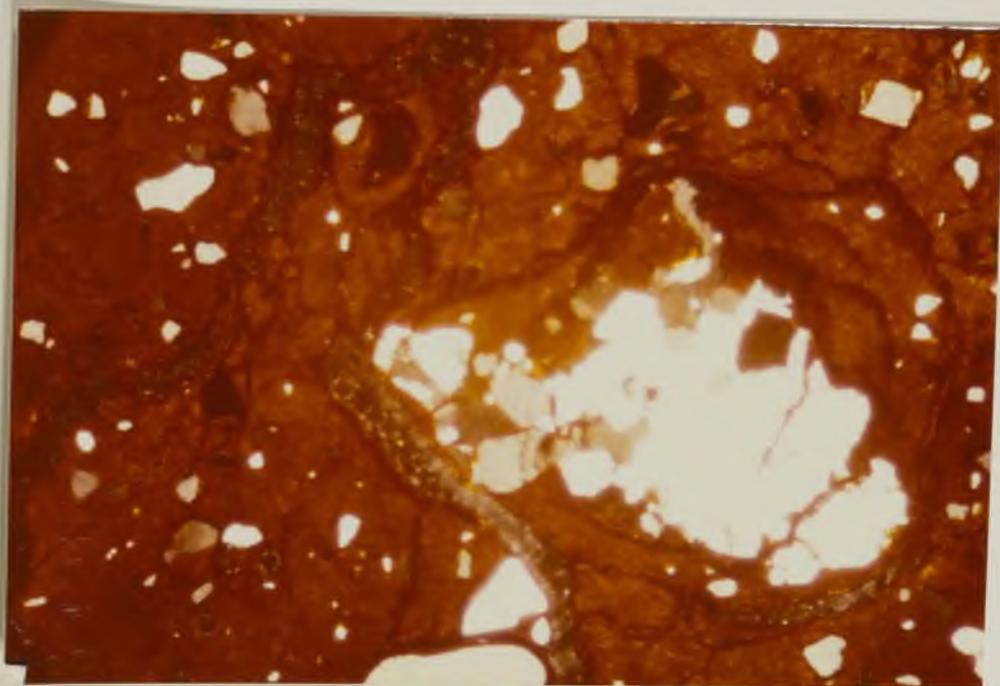
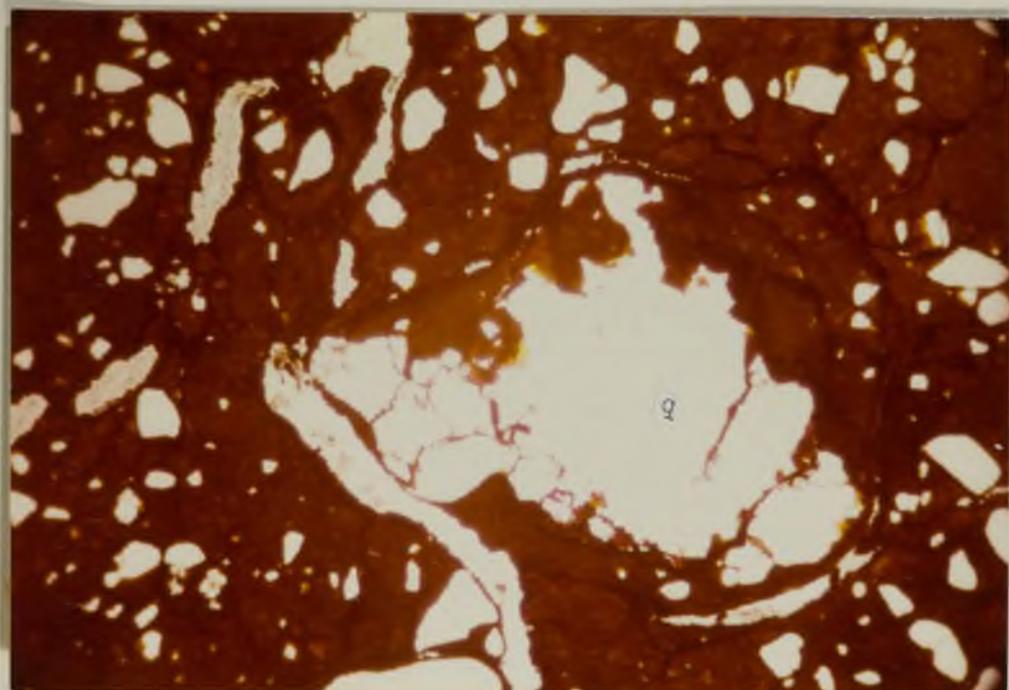


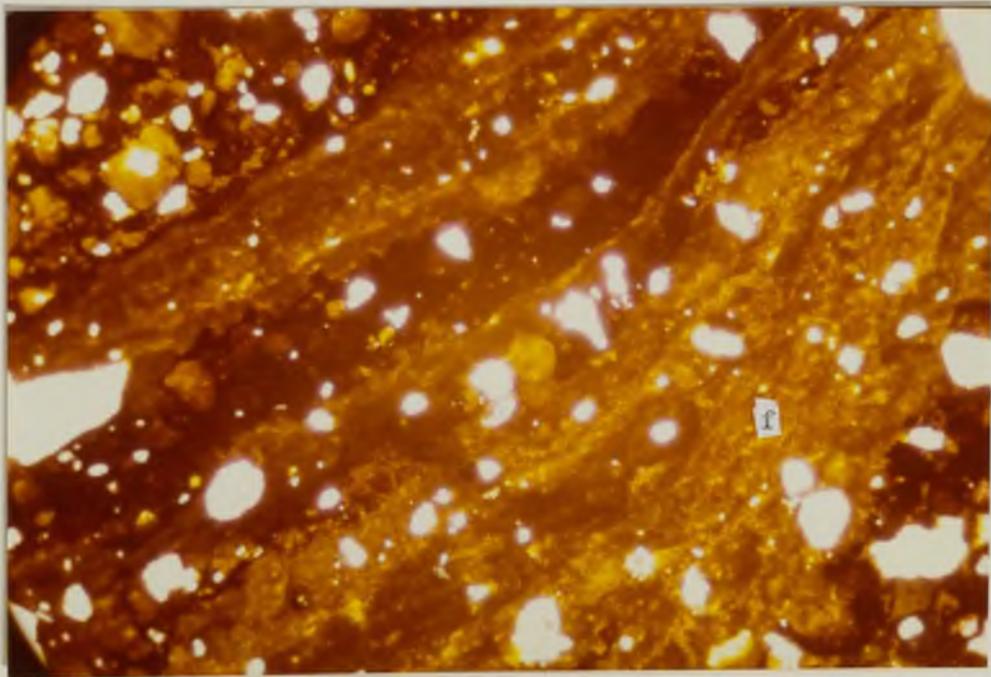
PLATE 14 x 0.5

A piece of hardened Sape soil. Note the porous nature and the lack of the discrete bodies found in Nova Floresta (Plate 1) and Cuite soils (Plate 6). Note the flow structure.

PLATE 15 x 80

Flow structure of Plate 15 as seen under the microscope. Coalesced channels have spilled over and have obliterated some of the soil features. Brown areas are iron coated clays, yellow areas are amorphous iron oxide, white areas with light blue edges are quartz and the light areas without light blue edges are voids.

(f) - The flow structure.



ones, this implies a difference in the chemical composition,

The bodies are mostly round and have different amounts of iron oxide impregnation. They are all products of cementation of soil particles (clay minerals, quartz, iron oxides etc.). In addition to the cementation role within the bodies, iron oxide also provides a continuous link between the bodies. The link was identified to consist mostly of goethite. Haematite was identified in a few places, which presumably results from the transformation of goethite.

The difference between the three soils is basically the degree of segregation of the aggregated bodies. The Nova Floresta soil (Plates 1-5) is one in which the soil particles having aggregated, almost entirely, into rounded bodies. The Cuite soil (Plates 6-13) is aggregated to a lesser extent and the Sape soil (Plates 14 and 15) is least aggregated. The segregation of certain parts involves the enrichment of the parts in iron oxide (see large central patch in Figures 4 and 5 and compare with incipient similar patch Figures 10,11). Under the microscope, the thin sections can be separated into two main regions. This is based on the presence or absence of voids and the concentration of iron oxide.

- (i) A dense iron-rich region 2mm to 6mm in diameter (central patch in Plates 4 and 5; lower section of Plate 9). It is yellowish brown to dark brown under plain light. It is separated from the other regions in such a manner its borders with the other regions become progressively well-defined with concentration of iron oxide. This body is the same as the nodule of Bryan (1952); pisolith of Ollier (1959), McFarlane (1976, p.66) and pisolite of Alexander and Cady 1962.
- (ii) A porous region, that has a lower amount of iron oxide. This region is also aggregated into bodies smaller than the dense,

iron-rich region. Sizes range from $25\mu\text{m}$ to $250\mu\text{m}$ in diameter. Average diameter is about $100\mu\text{m}$ (see dark brown areas surrounding yellowish central patch in Plates 4 and 5. This is the same as the top right-hand section of Plate 9).

The segregations are best exemplified by the Nova Floresta soil. The discussion of the segregated areas will therefore be discussed with reference mostly to the micrographs from the soil.

Dense regions: An example is the yellowish brown central patch of Plates 4 and 5. They are iron coated clays with a high degree of coating. It is significantly lacking in voids. The voids having been filled by periodic deposition of iron oxide (yellow areas within the brown central patch of Plate 5), The boundaries between the regions and the surrounding region are distinct in the Nova Floresta soil and faint in the Sape soil.

Porous regions: These are made of small bodies (Plates 2, 3 and 9). They are highly separated into discrete entities in the Nova Floresta soil and less separated in the Sape soil. Four types can be identified:

- (i) Iron oxide bodies.
- (ii) Clay bodies with iron oxide coating
- (iii) Iron oxide-centred clay bodies with iron oxide coating
- (iv) Quartz-centred clay bodies with iron oxide coating.

Each one varies in diameter between $10\mu\text{m}$ and $250\mu\text{m}$. Deposition of iron oxide exists at their boundaries and serve as links between the different bodies. The iron oxides also link these bodies to the dense regions.

The separation carried out above on the absence or presence of voids and amount of iron oxide could also be made using size as a basis. The dense bodies are usually in excess of 2mm while the bodies in the porous regions sizes lower than $250\mu\text{m}$ (See Plates 2-5 and 9). This observation

PLATE 16

Electron micrograph of porous region of Cuite soil. Clay particles have been cemented to form semi-rounded bodies. The void in the central part is partly filled by iron oxide deposited in an amorphous state. Crystallisation of the iron oxide produces spherical humps which grow outwards from the deposition surface, towards the centre of the void. Some form of internal ordering is present as seen in the fibrous nature of the interior.

PLATE 17

Plate 16 at a higher magnification showing the layering of the deposited iron oxide. Layering of the iron oxide deposited is evidence of periodic deposition.



PLATE 18

Plate 16 at a higher magnification showing the relationship between the iron oxide deposited and the surface onto which it was deposited. Note the smooth nature of the surface of deposition.

PLATE 19

Plate 16 at a higher magnification showing the fibrous nature of goethite that has crystallised from deposited amorphous iron oxide.



PLATE 20

Electron micrograph of porous region of Cuite showing surfaces of clay grains cemented by iron oxide. There is continuous orientation of clay grains around semi-spherical voids. The voids were probably filled with spherical bodies similar to the ones shown in Plate 2. The bodies were probably dislodged during the preparation of the section. Newly formed quartz particles can be seen with linear cracks.

PLATE 21

Porous surface of cemented clay grains and quartz particles. Note the attachment of cemented clay onto a fairly detached quartz particle. The attachment suggests that the quartz formed a nucleus for the deposition of iron-coated clays. Cuite soil.

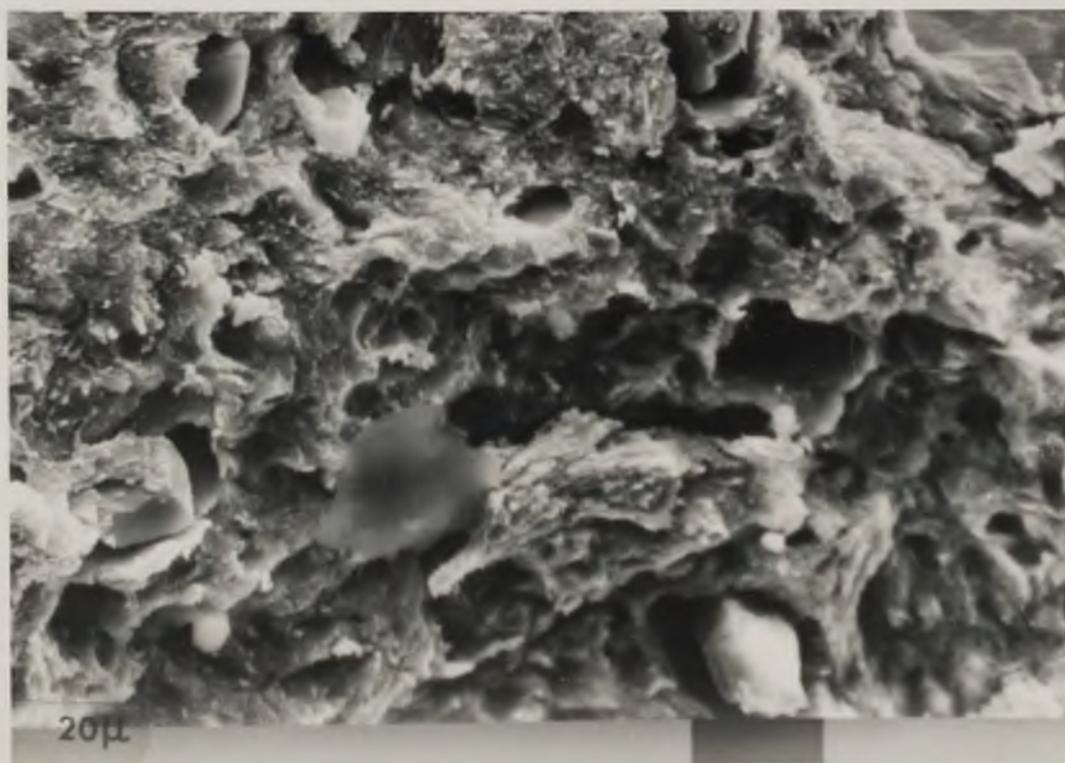
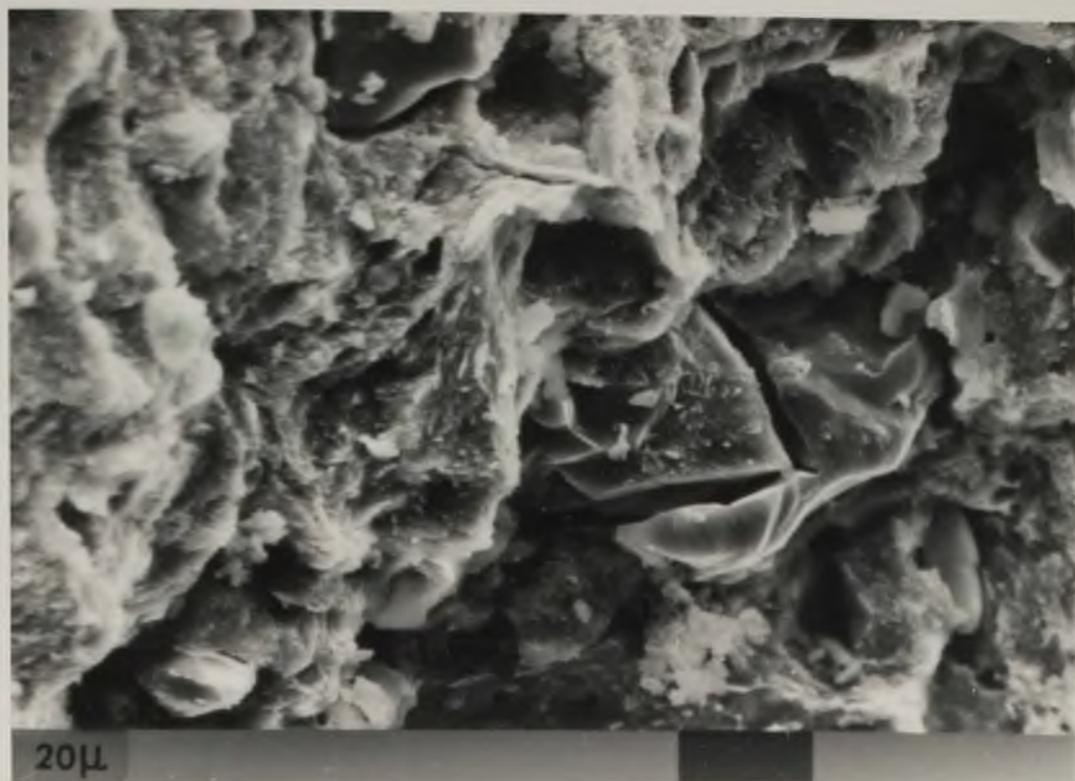


PLATE 22

Electron micrograph of dense region of Cuite soil showing strongly cemented surfaces. The lower right-hand corner has deposition of amorphous iron oxide. The centre of the micrograph is occupied by a near-completely filled void. Note the continuous orientation of clay grains parallel to the side of the void. The iron oxide deposited in the void has fibrous structure and has been involved in crystallisation.

PLATE 23

Plate 22 at higher magnification. Note the worm-like surfaces of the humps, developing inside the void, on the surface of the deposited material.



PLATE 24

Subrounded, abraded quartz particle attached to a strongly cemented surface of Sape soil. The surface is fairly porous.

PLATE 25

Plate 24 at higher magnification. Note the smooth surface and sphericity. These are evidence of transportation and agrees well with the colluvial nature formulated for the soil on the basis of mineralogical and chemical composition of particle sizes in the soil. (see Chapter 6).



PLATE 26 and PLATE 27

Irregular voids and strongly cemented areas in the Sape soil.



1000x 1000x 1000x

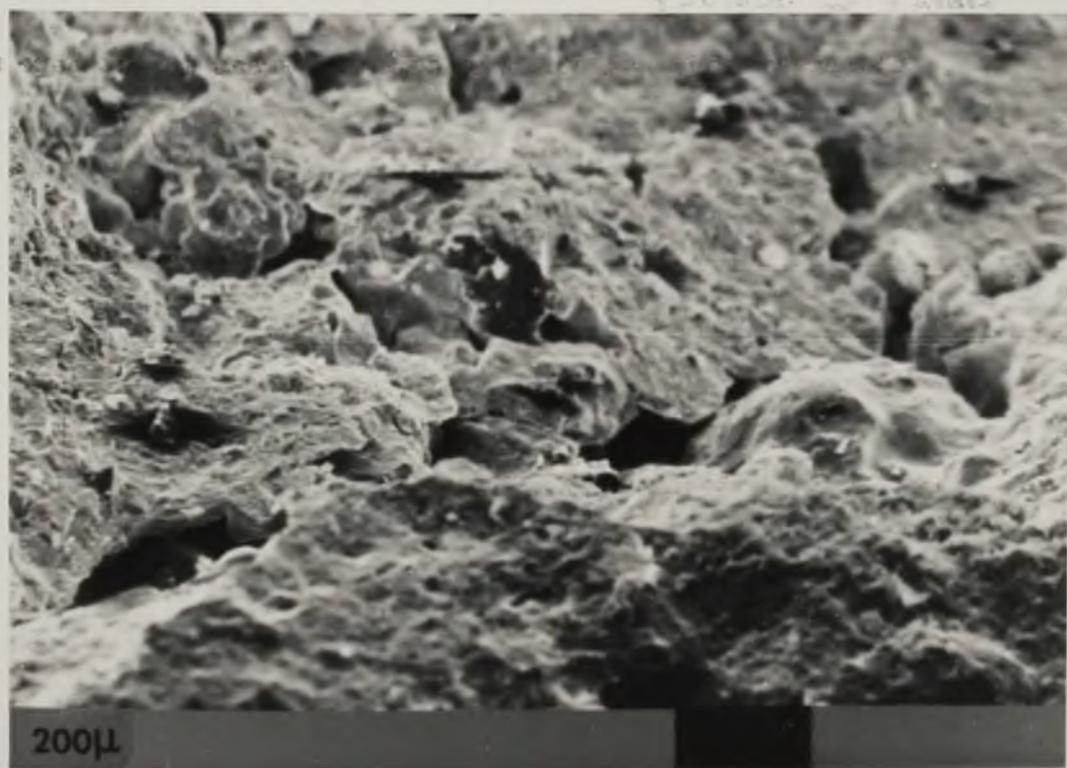


PLATE 28

Cemented clay surfaces of porous regions in Cuite soil. Note the surface texture of the quartz particles and their angular nature. (Compare with Plates 24, 25, from Sape soil).

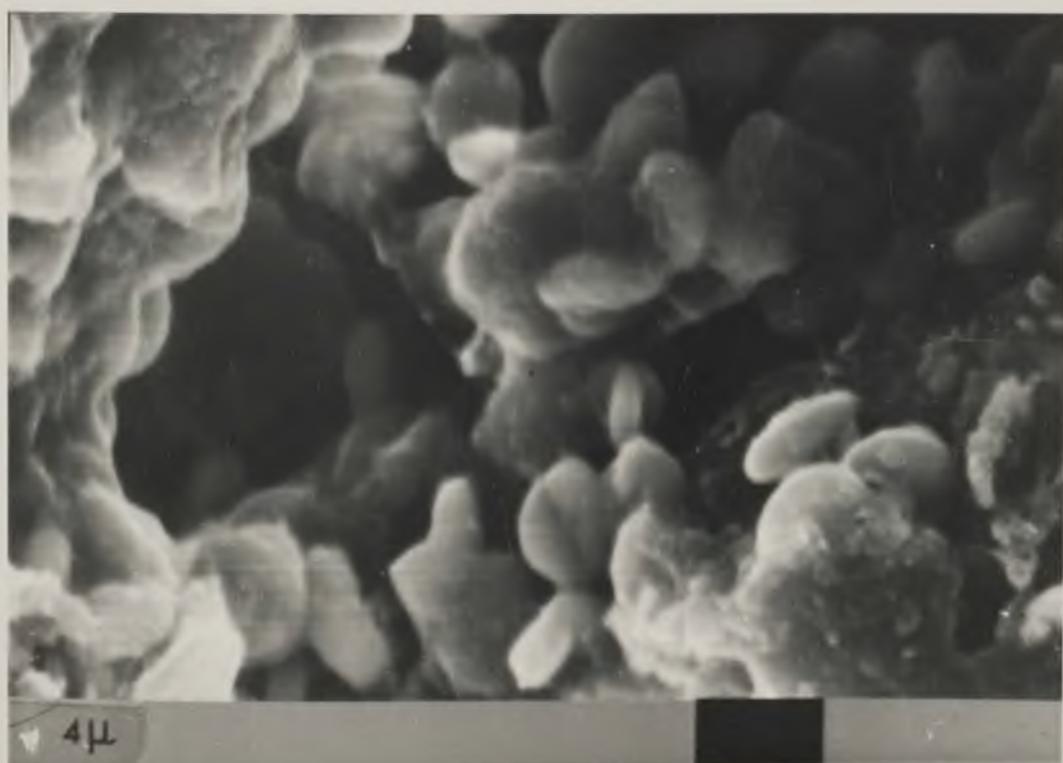
PLATE 29

Plate 28 at higher magnification showing the attachment of clay particles to surfaces of quartz particles.



PLATES 30, 31

Plate 28 at high magnification. Note the clustering of clay particles by iron oxide. The clusters are iron-stained clays. (The ochreous clay fillings of Plate 6).



validates the classification of red soils into oolites and pisolites as suggested by Twenhofel (du Preez, 1954). This is the same as ooliths and pisoliths of Pullan (1967), Ollier (1959) and McFarlane (1976, p.66).

8.2.2 Electron Microscopy:

The surfaces of hardened soils (concretions and fines) that had been fractured were examined using a Cambridge Instrument 'Stereoscan' scanning electron microscope. The method adopted for the preparation of the specimens is given in Appendix A.

As already mentioned, the coating and cementation by iron oxide does not allow the distinction of the different components with any degree of clarity.

The interaction between single clay particles (kaolinite) is impossible to see. However, some interesting observations of void fillings are present. The growth of the implantation in the voids and for the first time the internal structure of the implantation, can be discerned. These are described along with the micrographs (Plates 16-31).

8.3 The Formation of Aggregations in red soils:

The different elements of structure described from observations in the optical microscope will influence strength in different ways. An appreciation of their origin and paths for the transformations of clay-sized particles to larger-sized ones is considered desirable.

A mention has been made of the paths leading to the transformation of primary minerals in rocks, into clays and secondary products in soils. The conditions necessary for the accumulation of iron oxide in soils have been discussed. The interaction between clay particles and iron oxides leading to the cementation of the clay particles has also been mentioned. No further elaboration need therefore be made, concerning the source of

iron oxides for the formation of aggregations.

Two conditions have to be satisfied for the formation of aggregations in red soils. These are:

(1) The presence of a alternation of wetting and drying climatic phases.

(2) The matching up of the zone of intermittent saturation by groundwater with the zone in which accumulation is to take place (i.e. zone receptive to the accumulation of iron oxide.

The zone of intermittent saturation is the zone in the ground between wet and dry season water levels. This zone must coincide with the zone in which accumulation is to take place so that the effect of the alternate wet and dry cycles can be continuous and significant. The zone of accumulation has to contain some amount of clay, though not too much is required (see Chapter 3).

A theory for the mechanism of segregation and rearrangement can be formulated as follows: Iron is mobilised in solution and is carried through the soil during the wet season. The groundwater moving through the soil, leaves iron bearing solution in random pockets. These pockets are likely to be areas in which small pores predominate, which will have high moisture-retention capabilities.

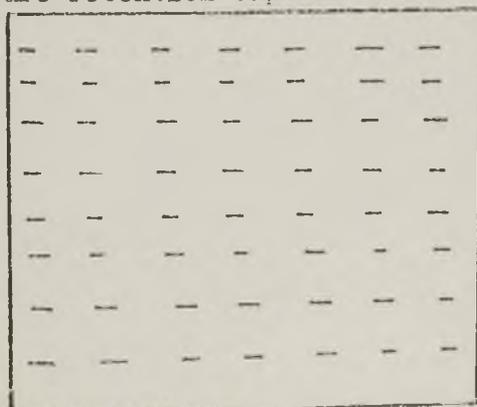


Figure 8.1(a)

I - Clayey soil. Already formed or in statu nascendi. First introduction of iron oxide will serve to saturate the clay surfaces. Aggregations will not form until the clay surfaces are saturated (Fripiat et al. 1954).

If a dry phase follows the wet-phase, evaporation occurs when the concentration of the iron solute reaches a stage where the solubility product of the iron compound is exceeded (Ojanuga & Lee 1973). The iron is deposited from solution onto the surfaces of soil particles (usually clay) in random areas.

The deposition of iron oxide onto the surfaces of soil particles does not only have to be from evaporating solutions. The retention of iron oxide may be from a silica-protected soil which loses its protective silica in a fairly dehydrating environment (Demoion and Batisse, 1944), e.g. at the water-table. Oxygen-rich regions can also give spot oxidation of ferrous iron or ferrous complexes carried in solution, in which case a dry period is not essential. Whatever the mechanism, the oxidation and dehydration that occurs during the dry phase is necessary for the fixing of iron oxide retained from the groundwater.

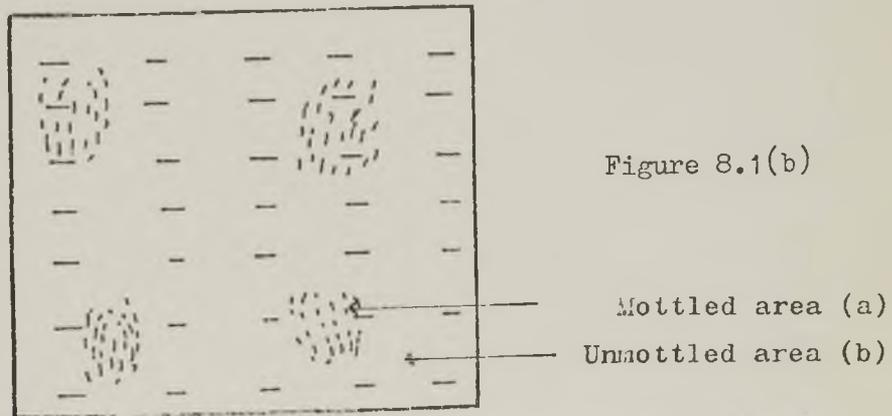


Figure 8.1(b)

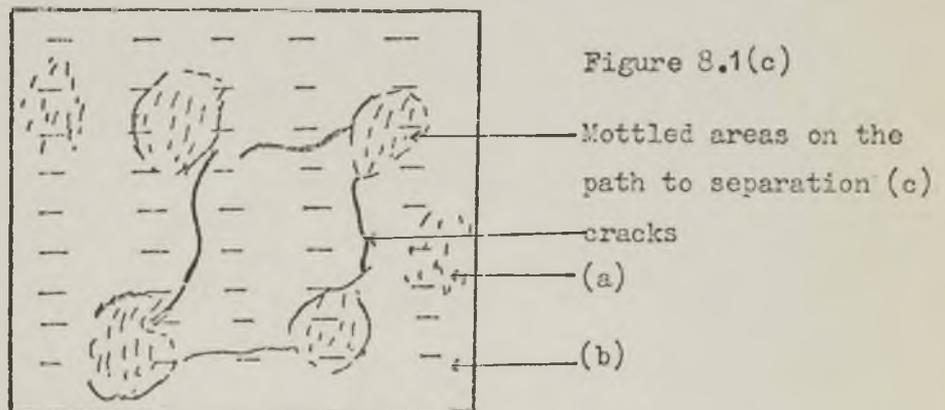
II - Iron coating of clay surfaces and concentration in random areas (Mottled stage).

The soil is now in a mottled state and contains random areas of iron oxide concentration (see stage II Figure 8.1(b)). The mottled areas are the ones that will subsequently form aggregations. That the mottled regions are precursor to hardened discrete bodies has been suggested (Pullan, 1967, Ojanuga and Lee, 1973). The transformation of the mottled areas into aggregations requires the continual cycle of

wetting and drying. Soils in regions with no marked dry seasons will not develop aggregations and the tendency is for groundwater to move iron oxide into other areas until it becomes uniformly distributed. This can be achieved by dissolution from mottled areas or by new precipitation in unmottled areas.

The deposition of oxide in the unmottled areas will mostly be in the form of amorphous iron oxides (hydrated) which will change into more crystalline forms with age and continuation of the cycle. The deposition of iron oxide in the mottled areas and the consequent dehydration leads to the cementation of soil particles in these areas.

The retention of iron-bearing solution during subsequent periods of wetting will be higher in the cemented areas, because of the presence of smaller voids in them. The large voids in these areas have become reduced by deposition and cementation. The deposition and cementation in turn, leads to an increase in the density of the mottled areas and consequently, a decrease in the density of the unmottled areas. The density difference is similar to the one shown in Plates 10 and 11. This corresponds to stage III Figure 8.1c



III - Mottled areas with fairly distinct boundaries and the initiation of cracks around and between them.

The evaporation during a dry phase following a wet phase generally leads to the removal of pore-water and therefore shrinkage. As a result of the different volumes of water present in the mottled and unmottled areas the amount of shrinkage in the two areas will be different. The mottled areas shrinking less than the unmottled areas.

The differential shrinkage between the mottled and unmottled areas leads to the formation of cracks around the mottled areas. The formation of the shrinkage cracks initiates the process of separation of the entities in the mottled areas from those of the unmottled areas (Figure 8.1(c) Stage III). Plate 1 shows these cracks around segregated bodies. They are mostly filled because the stage is more advanced in the sample.

The separation of mottled from the unmottled areas can also be due to inherent discontinuities. For example, relict joints inherited from the parent rock or microjoints and microfaults produced by such physical processes as mass movement, attendant during soil formation can lead to separation.

The soil grains in the unmottled areas become readily moveable as a consequence of the decreased density. The clay can be readily moved because of decreased clay-water affinity due to iron oxide deposits on the surfaces created by differential shrinkage. Yellowish white areas of Plate 1 are such cracks which contain ochreous clay fillings.

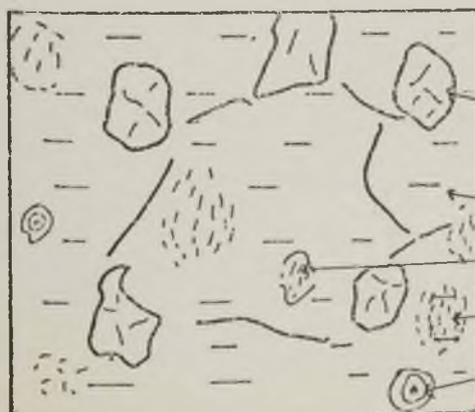


Figure 8.1(d)

Segregated mottled area with cemented particles (d)

(b)

(c)

(a)

Deposition around a nucleus (e)

IV - The first mottled area is now segregated and further deposition takes place in the voids within it. Deposition around nuclei takes place leading to the formation of concentric concretions.

The process described is an ever continuing one, unmottled areas can become centres of deposition leading to cementation and the formation of aggregations. As a result of the continual deposition, the soil particles in the unmottled areas will have been coated and cemented by iron oxide (Fig8.1(d) stage IV).

Further deposition of iron oxide takes place mainly in the voids and channels of the mottled areas and to a lesser extent in the unmottled areas. Plates, 4, 5, 7 and 8 show channel and voids filling within such areas. The coating of iron oxide in the pores or the surface of particles may absorb ferrous iron present in solution. Bloomfield (1951) showed that ferric oxide can absorb ferrous iron.



Figure 8.1(e)

Deposition on the surface of a segregated body (f).

V - Deposition on the surface of segregated bodies lead to outward growth.

The deposited iron oxide will initially be in the form of amorphous oxides which will change into crystalline forms with continuation of the process. When channels and voids in the separated bodies (mottled areas) are filled, the deposition takes place around the bodies and they start to grow outwards due to periodic deposition (Fig.8.1(e) stage V). See Plate 5. Evidence of periodic deposition is seen in the dense region, the bottom left corner.

Certain other bodies besides those derived from the cementation of soil particles in-place can be present, particularly in the previously unmottled areas. Many of these are formed by the periodic deposition

of iron oxide around a nuclei. These nuclei can be of quartz, other primary mineral particles or small iron oxide particles precipitated from solution. The bodies formed by periodic deposition will be generally denser than those formed by cementation in-place. (Plates 2 and 3 show deposition of iron-coated clay around quartz and iron oxide bodies in the originally unmottled area).

Some of the bodies observed in the microscope were seen to have undergone some form of diffusion process whereby iron oxide was concentrated at the centres of semi-spherical bodies at the expense of the periphery (see Plate 9, dense region). The mechanism for this movement is not very clear. A possible explanation is the dissolution of clays (kaolinite) given by Sivarajasingham et al. (1962). The dissolution is supposedly by groundwater moving around the bodies and allows the movement of iron towards the centre. This dissolution, as suggested by Sivarajasingham et al., will lead to the formation of gibbsite, but no form of alumina was observed in the soil. Instead, an excess of silica, over the amount required for the formation of kaolinite was recorded. It is possible that the dissolution occurred, the iron moved to the centre and gibbsite was formed. Subsequent introduction of silica will transform the gibbsite back into kaolinite and this can explain the absence of alumina. The evidence of addition of silica is presented in Plates 12 and 13. This will, however, mean that the accumulation of iron oxide leading to the formation of concretions and the accumulation of silica are not contemporaneous. The accumulation of silica occurring at a later stage than the accumulation of iron oxide. Plates 12 and 13 show the growth of quartz from a filled void which had been defined. This shows that silica was added at a much later stage.

The material deposited in the voids and the surfaces of the bodies

does not have to be only iron oxide. Alumina, silica or even clays can be moved and deposited as coat on walls of voids and channels. Plates 12 and 13 show neoformation quartz resulting from deposited silica.

At this stage, the accumulation horizon now appears as indurated bodies in a loosely cemented matrix.

The outward growth of aggregated bodies leads to their cementation with other bodies and even the assimilation of some of the initially unmottled areas which now are aggregated bodies but of smaller sizes. The difference between different aggregations, due to the iron oxide present and the time taken for their development (Figure 8.1(f) Stage VI).



Figure 8.1(f)

VI - Dense cemented segregated bodies, the bodies are linked by aggregated jumble of smaller bodies.

Further deposition and continuation of the alternate wet and dry cycles leads to the linking together of aggregated bodies by iron oxide on their surfaces leading to the formation of a continuous network of iron oxide that connects all the bodies (see Plate 4). A stage further will be the formation of a hard, continuous crust in which all the bodies are completely linked and strongly cemented. Some authors are of the opinion that this only occurs when the soils in the previous stages are exposed to a long, continuous dry phase (King, 1967, p.176).

It is worth mentioning that the system is a dynamic one. Some

bodies exhibiting features of an earlier stage will be found while other areas present an advanced stage or vice versa. On the average, the materials within a particular horizon can be ascribed to a particular stage.

It can be seen that the mechanism described above is applicable to soil development by iron enrichment within the profile or from an outside source. The important factors are the existence of an alternate wet/dry cycle and the requirement that the zone of intermittent saturation by groundwater falls within the zone where deposition is to take place.

8.4 The need for the investigation of microstructure in the study of Red Soils:

The examination of the microstructure of the soils can be shown to provide support for many of the conclusions reached earlier. These conclusions are based entirely on studies carried out relating to chemistry (Chapter 6), mineralogy (Chapter 6) and strength (Chapter 7).

For example, the flow structure shown in Plate 15 that occurs only in the Sape soils, has been examined by previous authors. Alexander and Cady were of the opinion that such a feature imparts rigidity to soils and that they are found only in hard red soils. Hamilton (1964, p.272) suggested that the feature is only observable in young stages of red soils formed by impregnation of slope deposits. Such a colluvial nature has already been suggested for the Sape soil (Chapter 6) based on the study of profile characteristics and Chemical/Mineralogical content. The writer, however, disagrees with Hamilton in the question of age of the soil, since the feature is only indicative of recent mobility. It presents only evidence of continuity of iron oxide accumulation. It would apply to any age as long as the process is continuing. McFarlane (1976, p.34) has discussed the inversion

of relief associated with red soils. An old red soil which came to occupy an interfluvial position will possess the feature, if it receives iron accumulation from outside sources.

Another example concerns the presence of degradable soil particles. This concept was invoked to explain many of the unusual engineering behaviours (Chapter 7). The actual physical nature of the aggregations can be seen in Plates 1-15 and the friable nature of the porous regions (e.g. Plate 2) attest to the presence of degradable particles.

A third example is the presence of amorphous silica in the concretions. A concept which was invoked to explain the high molecular ratio of silica to alumina in the clay fractions of the three soils. Newly formed quartz can be seen in Plates 12 and 13 as a void filling. Its occurrence as a void filling shows that later additions of silica occurred after the void had been defined by other soil elements (including iron oxide) and also points to the mobility of silica. If silica was mobile some of it could easily have been deposited in the amorphous form.

The observations demonstrate the need for a comprehensive approach to the study of red soils. Particularly needed is the actual observation of microstructure, which has not been given considerable attention. Mitchell (1976, p.198) has made reference to the scarcity of previous investigators. Terzaghi (1958), Townsend et al. (1969) have made reference to the influence of structure on engineering behaviour without actually observing this structure. Others, Coleman et al. (1964), Wallace (1973), have suggested the nature of the structure based on their observations of physical properties. This is surprising considering the widespread availability of microscopes today for observations. It is widely accepted that indirect approaches, though important, are of limited value. Complex systems exist on soils and

physical measurements can be attributed to a variety of reasons. Coleman et al. (1964) suggested that iron oxide was present as thin films on clay surfaces of Nyeri soil. This suggestion was based on the reflectivity of monochromatic light. Smart (1973) carried out electron microscope observations of the same soil and failed to find any films.

The direct approach may hold the key to the understanding of the engineering properties of soils. For example, based on this observation of microstructure the writer intends (Chapter 9) to suggest the range of particle sizes that are critical to the engineering behaviour.

CHAPTER 9

THE RELATION BETWEEN THE PROPERTIES INVESTIGATED

9.1 General:

Modern ideas on the engineering characteristics of soils accept the concept that, the values obtained from the physical parameters used in the evaluation of soils for engineering purposes, reflects the properties that operate on lower levels, (i.e. microscopic). The present work puts great emphasis on the investigation of the intrinsic and fundamental properties because it is considered to hold the key to the interpretation of the engineering behaviour of red soils.

The use of red soils as engineering materials has been undertaken by engineers, who, by and large, have been instructed in the behaviour of soils found elsewhere. The non-conformity of these soils to the classical soil behaviour approach necessitate the consideration of other approaches.

The investigation of engineering properties of tropical soils carried out in the last few years have concentrated mostly on the use of physical properties to explain other physical properties. A specific case is the investigations carried out by Lyons Associates/B.R.R.I. (1971) Ghana for soils of Africa. Innumerable statistical correlations were sought between related and obviously unrelated properties. The result of the findings suggested a distinction between three groups of soils previously separated on the basis of soil-forming process D'Hoore's (1964) Classification for soils of Africa. The inadequacy of the approach is seen in the considerable overlap of the physical properties of the three soils; soils that the study aimed at differentiating. The very fact that D'Hoore's original classification is based entirely

on soil-forming processes necessitate the use of the properties derived from these processes for the assessment of engineering behaviour.

The analysis of the characteristics of the soil profile, the engineering properties and considerations of chemical and mineralogical composition places the Cuite, Nova Floresta and Sape soils among the ferruginous soils group - D'Hoore's (1964) classification.

Ferruginous soils fall into the thermally stable group, i.e. one in which no substantial change in property occurs after drying at 100°C. From the mineralogical analyses, the components present which are susceptible to change on drying are amorphous iron oxides. These are however, not present in considerable amounts and have been immobilised to goethite and haematite. Hence the thermal stability (see Table 7.1).

The second type of peculiar problem, i.e. mechanical instability has been shown to be true, (Chapter 7). Mechanical instability is manifest as particle breakdown in the direct shear, triaxial and compressibility tests (see 7.3). The particle breakdown has been found, from the direct shear tests, to be dependent on the level of stress and the moisture content (see 7.3.1 and Figure 7.22).

The compressibility tests have also shown that the breakdown is a progressive phenomenon and continues with time. Figure 7.26 shows that the comminution of particles is time dependent and if the soil were to be used in construction, gradual production of finer particles would result. This could bring loss of strength and other problems not anticipated during construction.

The problem of strength of the soil is therefore not only one of determining the safety of the structure during construction, but also to determine if post-construction changes that will happen within the soil, will allow it to bear the structure.

The compressibility result also shows that the Sape soil is more

compressible at optimum moisture content than either the dry or the wet side of the moisture density curve (Figures 7.19-7.21). The interplay of energy and moisture content seems to produce the most compressive specimen at this moisture content. (Using the mode and amount of energy described). The influence of this finding on method of further handling of the Sape soil (already used as base course for the Sape-Guarabira road(see Figure 5.3), is important.

The unusual strength behaviour, the higher compressibility at optimum moisture contents, the overall moisture-strength relationship have all been explained by the mechanical instability of the aggregations and the continuous link of iron oxide (Chapter 7.3 and 8.1). The strong dependency of strength on the stress-level and moisture compaction, makes the investigation of the strength behaviour over a wide range of moisture content, for the construction of embankments and earth dams important. Simple compaction at optimum moisture content as is the usual practise (Ritter and Paquette 1960, p.220) may produce a higher compressible material. This may not be advisable. The strength of the compacted material is also important as the cohesion is a minimum at or just above this range (Figure 7.10, 7.11, 7.13). It is therefore desirable to investigate the soil properties over a range of possible conditions, that the designed structure will be subjected to, before the initiation of a major project.

9.2 Relationship between mineralogy, structure and strength:

The preceding chapters involve the separate investigations and discussions of the content and distribution of mineral/chemical components, the actual form, shape and presence of these components in the soil as observed under the microscope at various magnifications. Also carried out is the investigation and discussion of the response of the soil to

loading using standard physical tests.

In the following part of this Chapter, based on the investigations and their discussions carried out in the preceding chapters, a discussion of the relationship between the different properties investigated and the control exercised by one upon the other, will be carried out.

9.2.1 Mineralogy and Engineering Properties:

Kaolinite, goethite, haematite and quartz are the important minerals identified in the soils (Chapter 6). These have been found to interact in such a manner that individual particles are rarely present (Chapter 8). The particles are cemented together to produce various spherical bodies observed in the microscope (Chapter 8).

The minerals that have the most important influence on the engineering behaviour are the iron oxide minerals, which have been found to coat clay mineral particles, cementing and aggregating them (Chapter 8).

The clay-size contents of the three soils based on granulometry are less than 18% (Figure 7.1a). The kaolinite content determined from thermogravimetric analysis is generally in excess of 35% Table 6.5. The difference in the values shows the extent to which cementation by iron oxides has led to the upgrading of finer particles to larger sizes. The resultant change in grain size distribution will affect the engineering properties. The grain size distribution is an important parameter in the evaluation of engineering soils.

The iron oxide-clay interaction results in the coating and cementation of clay particles by iron oxide. The mineralogical analyses have shown that the iron oxide exist mostly in the crystalline state and therefore will suppress the normal interaction expected to exist between clay and water. The suppression of this interaction results in the lowering of

the clay affinity for water and therefore the Atterberg limits. Atterberg limits are another important evaluating parameter for soils. Low values of liquid and plastic limits were recorded. This is a result of the aggregation and cementation of the clay and the high extent of crystallisation that the iron oxides have undergone. The expansive property of the soils is low for the same reason.

The aggregation and cementation of clay as well as the high degree of crystallisation of the iron oxide results in the high values of ϕ , the angle of friction measured. The unusual stress-strain behaviour and pore pressure characteristics can be related to the effects of cementation by iron oxides (Chapter 7.3).

The individual minerals do not seem to have much direct influence on the engineering properties. The form of the minerals present and the nature of interaction between them appears to be very significant. The influence of mineralogy on the strength is via the interacted minerals rather than individual ones.

9.2.2 Structure and Engineering Properties:

The major properties of mineralogy, chemistry, strength and structure are all interrelated. The type and nature of the minerals present affects their spatial distribution and arrangement in the soil. The spatial distribution and arrangement in turn influences the engineering properties.

The most important factor that influences the engineering behaviour of the red soils investigated is the structure. Its influence is recorded on simple classification tests as grain-size and Atterberg limits and also on physical tests as triaxial strength, direct shear strength and compressibility (Chapter 7).

The Cuite, Nova Floresta and Sape soils all belong to an advanced stage in the aggregation process described in Chapter 4. At this stage, the clay minerals, iron oxides and other soil particles are all involved

in aggregation to produce spherical bodies with different degrees of cementation (see 8.2).

The response to applied load will essentially depend on the complex arrangement of the soil particles. It will also depend on the relationship between them and the iron oxide network that surrounds them and acts as linkages between them.

For soils loaded in the undisturbed state, the overall strength will be controlled by the strength of the iron oxide network linking the various bodies in the porous regions (see Plate 2). The strength of this link is likely to be weaker than the strength of the aggregated bodies. Ingles (1962) has described the nature of failure of stabilised materials. The failure theory for such systems is applicable in the case of these soils. Failure within the cementing links will take place by propagation of fracture through many of its small cracks. In which case, tensile rupture of the cementing links is the first stage in the fracture process.

Analysis of the strength of a cemented soil should consider where failure is to take place. This could be in the cementing network, in the particle or in the cement.

Since the cementing material of the link will be weaker than the aggregated bodies, the propagation of fractures takes place readily. If the cementing network had been stronger than the cemented bodies, the propagation of fracture is suppressed, until the general stress level is high enough to rupture the cemented bodies.

In the normal use of the soils for road building, the indurated elements are pulverised. The strength of pulverised soil will not only depend on the strength of the link between the bodies, but also on the strength of the bodies. The link which had been continuous between the bodies would now be present within a few particles. The

strength of the various small bodies in the porous regions and of the dense region will now influence the strength of the soil.

The situation here is more complex, for the different aggregated bodies, which may comminute have different resistances, as a result of the different amount of iron oxides present and the different degrees of crystallisation they had undergone. The particles from areas within the porous region containing the links will fail at about the same level of stress, since the link is likely to be of the same nature from place to place. The comminution of particles recorded in the compressibility test is therefore likely to be from the particles of the porous regions observed in the investigation of structure.

To finalise the discussion of the relationship between structure and engineering properties, it is worth reiterating that the work carried out has shown metastability to be a very active problem in red soils. This metastability will be more prominent in particles from the porous regions, and since the biggest "uniform" entity, here is about $120\mu m$ one can conclude that metastability will be more important in silt and fine sand size grains than any other particle sizes. There is need for differentiation of sizes that will be stable in the long run, from those that will be not. It is suggested, that soil evaluation should include stability of silt and sand sizes, these are the sizes susceptible to degradation.

CHAPTER 10

CONCLUSIONS

In addition to the general conclusions reached in the discussions of the mineralogy, condition/mechanism of formation and the engineering properties of these soils, the following specific conclusions can be mentioned:

1. The red soils of Paraiba which are highly aggregated concretionary red soils, are thermally stable but show mechanical instability.
2. This mechanical instability is as a result of particle breakdown which results from failure of the iron oxide links that cement the particles together.
3. The effects of particle breakdown on the strength of the soils have been demonstrated. This effect can be seen:
 - (a) The stress-strain behaviour which was found to depend on the level of stress and moisture content.
 - (b) The unusual nature of the plot of the drained cohesion parameter C' , determined from drained triaxial test on compacted specimen, versus the moisture of compaction.
 - (c) The compressibility characteristics of a compacted soil.
 - (d) The development of pore pressure in a loaded compacted soil.
4. The mechanical instability is due to aggregation of soil particles by iron oxides. This is the basic difference between these soils and those found in the temperate zones. The effect of the instability in a loaded soil is to produce finer materials continually. Long-term behaviour under stress is a factor that should be taken into consideration when these soils are used for civil engineering purposes.

5. The mineralogical composition of the soils is relatively simple, consisting of kaolinite, quartz, goethite, haematite, amorphous silica.
6. The kaolinite exists in a less ordered form. The lower crystalline nature probably affects the nature and amount of hydroxyl water in the crystal structure.
7. Iron oxide has cemented clay, quartz and other iron oxide particles into small spherical bodies. The actual clay mineral content is much higher than standard particle size distribution analysis would give. The aggregating effect of iron oxide is seen in its concentration in silt-size particles which results in the elevation of clay minerals to silt-sizes.
8. The propagation of cementation from microscopic clay/iron oxide interaction takes place as a result of continual deposition of iron oxide during alternating cycles of wet and dry periods. The alternating cycle not only moves and deposits iron oxides, it imposes certain physical conditions on the soil which enhances the formation of separated bodies, e.g. differential shrinkage.
9. The formation of aggregations in red soils necessitates two major physical processes. The presence of a fluctuating water table and alternating wet and dry periods. When these processes are present in addition to a source of iron, aggregation is expected to form.
10. The optical microscope is a very useful tool for the study of the structure of the soils, particularly in cases where most of fine materials have been cemented and aggregated.

CHAPTER 11SUGGESTIONS FOR FUTURE RESEACH

1. The effect of breakdown of particles under stress, observed in this study, should be examined for other engineering tests, besides the ones investigated.
2. The long-term behaviour under stress and its effect on engineering behaviour should be investigated.
3. The possibility of the identification of the size ranges most susceptible to degradation should be examined. The investigation of soil behaviour with and without these size ranges is desirable.
4. The extent to which the degree of crystallinity of kaolinite, affects the nature and amount of hydroxyl (OH) ions present in its structure, requires further investigation.
5. The origin, role and influence of amorphous silica on the geotechnical properties of a red soil, requires examination.
6. The effect of the degree of crystallinity of kaolinite and the absence or presence of amorphous materials on the reactivity of red soils to stabilisers, needs investigation.
7. The soils of Paraiba investigated have been involved in the accumulation of sesquioxides to a great extent. A similar investigation on soils with a lower degree of accumulation is desirable.

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APPENDIX A: Some Experimental Methods

- (i) Specimen preparation for optical microscopy.
- (ii) Specimen preparation for scanning electron microscopy.

Specimen preparation for Optical Microscope study

To facilitate cutting without the disturbance of structure, water in the pores of the soil samples were replaced with a suitable resin. Araldite CY 219 (Resin) and Versamid 140 (Hardener) were used. These were mixed in a ratio of 1 : 1 giving a refractive index of 1.54 (same as Canada balsam). The sections were prepared in accordance with the recommendations of Brewer (1964, p.403). The cementation of the cut sample onto a glass plate and of the cover glass onto the polished surface was done with Canada balsam. Specimen thickness was about 25 .

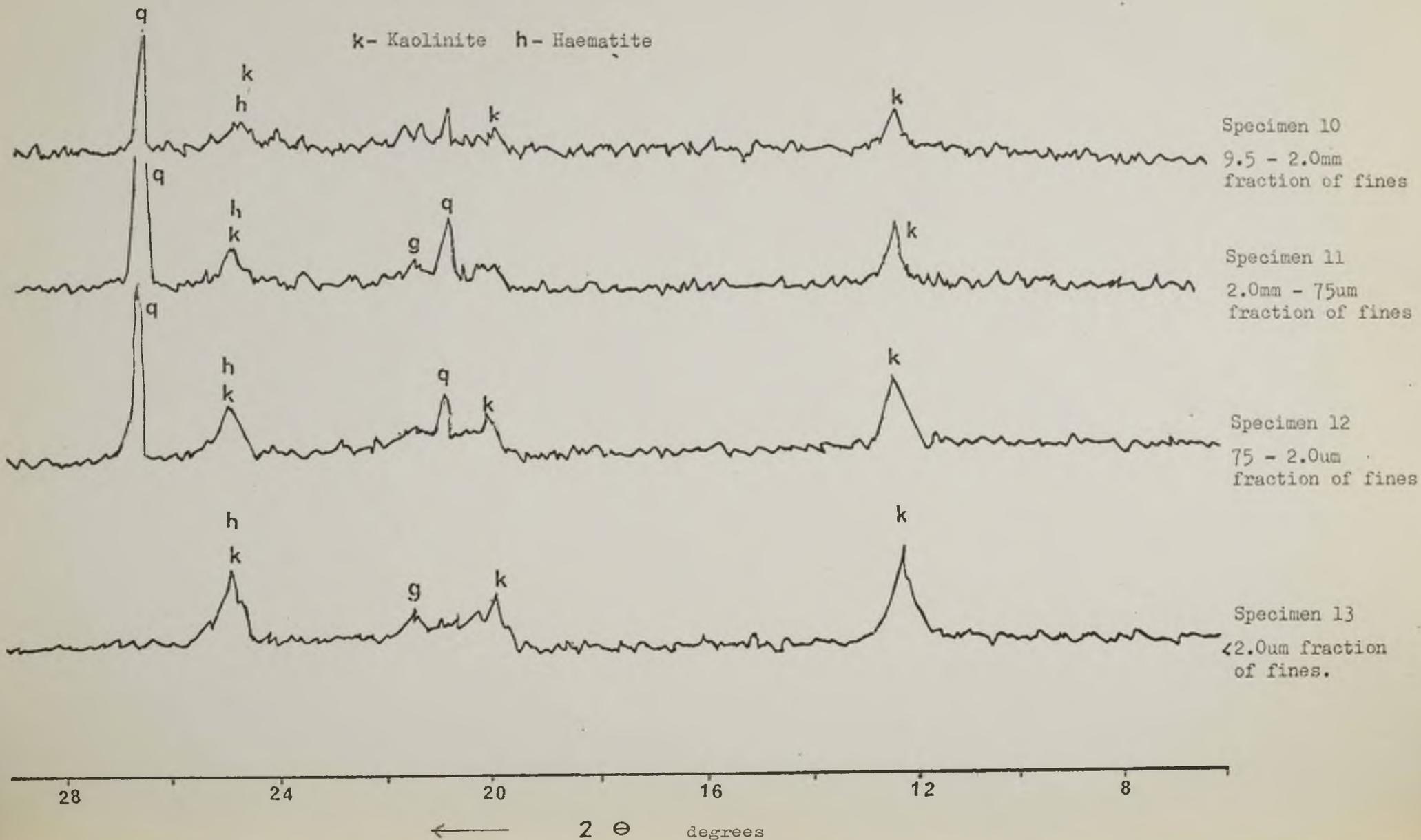
Preparation of sample for SEM study

Samples of undurated soils were air-dried and fractured. The side remote from the fractured surface was then ground flat. The flat side was fixed to specimen mounts suitable for use in the scanning electron microscope with Araldite. The fractured surfaces were treated with 60 applications and removals (peeling) of adhesive tape (sellotape) as suggested by Barden and Sides (1971). The surfaces were then coated with about 100\AA ⁰ thick coating of silver in a vacuum evaporating chamber. The coated surfaces were examined at different magnifications using a Cambridge Instrument company 'stereoscan' SEM.

APPENDIX B: Some Experimental Results

q - Quartz g - Goethite

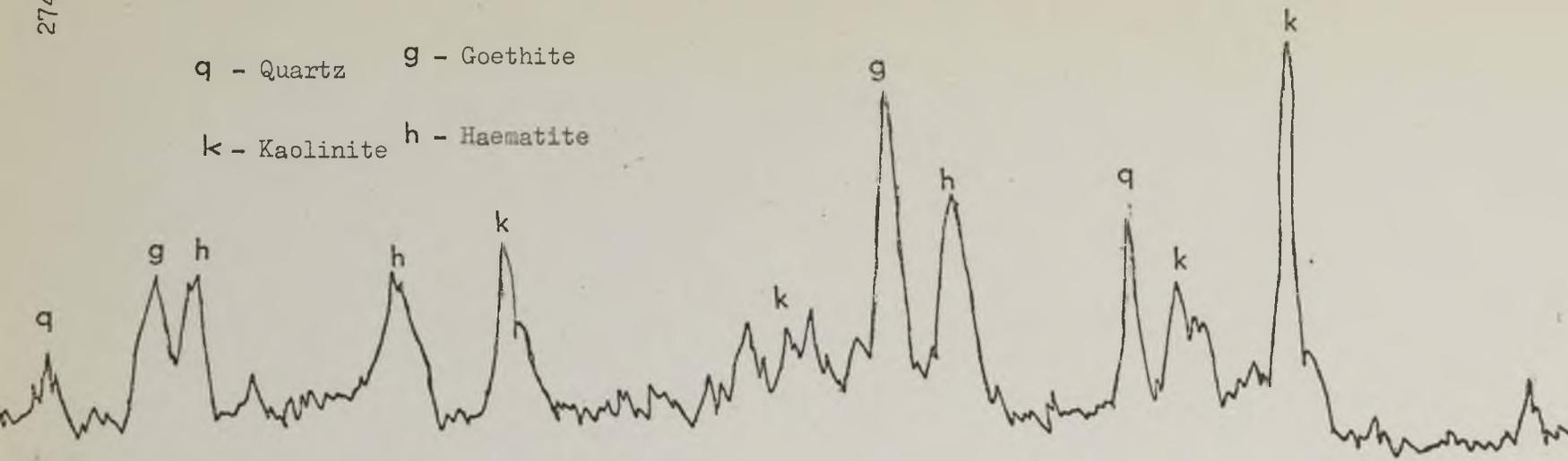
k - Kaolinite h - Haematite



X-ray Diffractograms of samples from the same horizon in Cuite soil. 1.0 m depth, left side. Cu $K\alpha$ radiation.

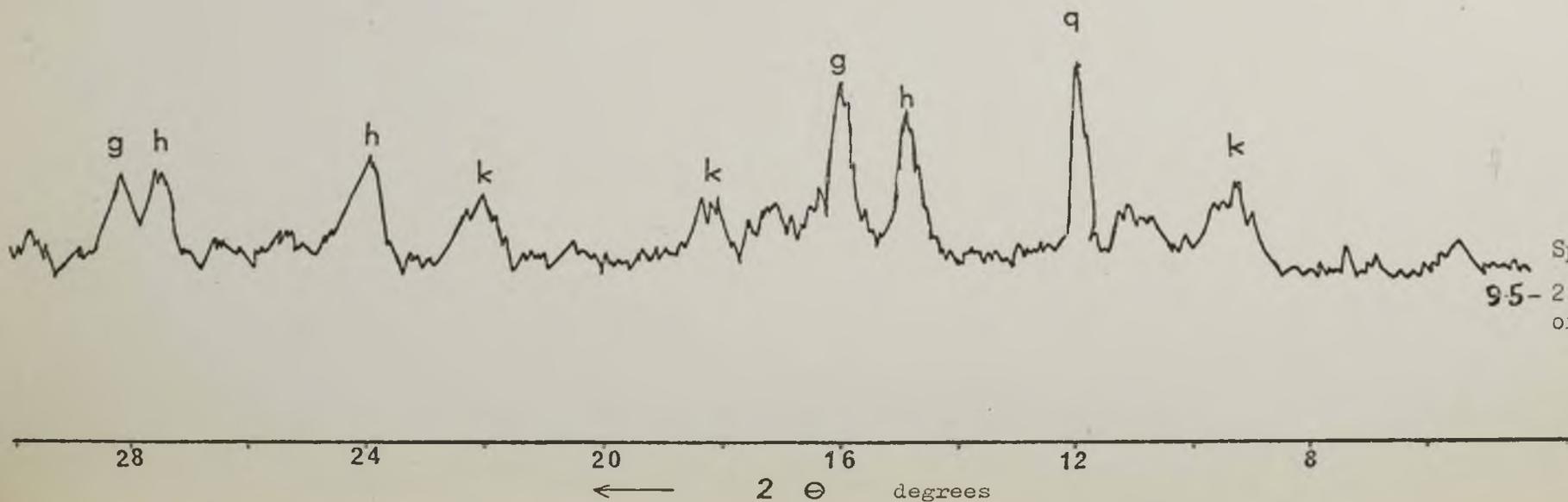
q - Quartz g - Goethite

k - Kaolinite h - Haematite



Specimen 34

75 - 2.0um
fraction of concretions



Specimen 30

9.5 - 2.0um fraction
of fines

28

24

20

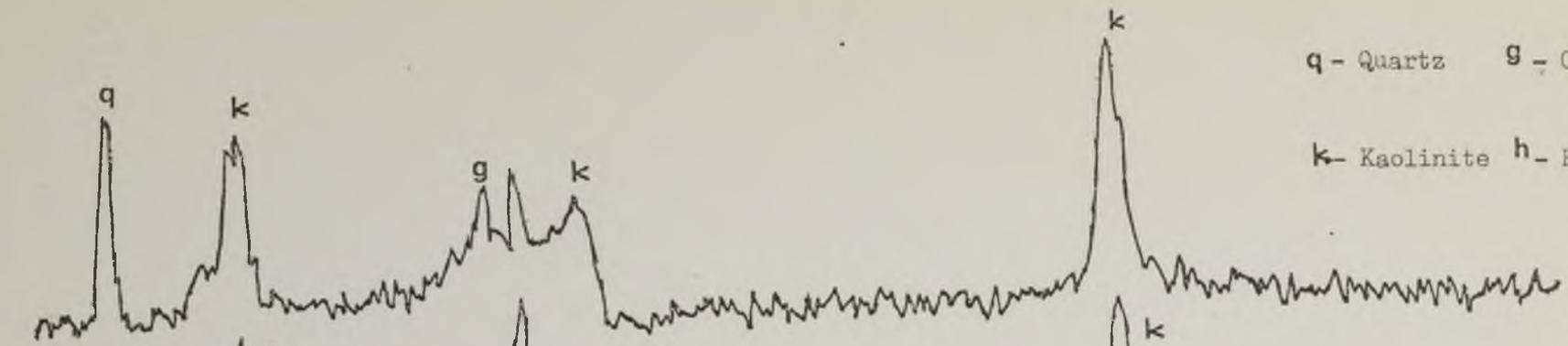
← 2 θ degrees

16

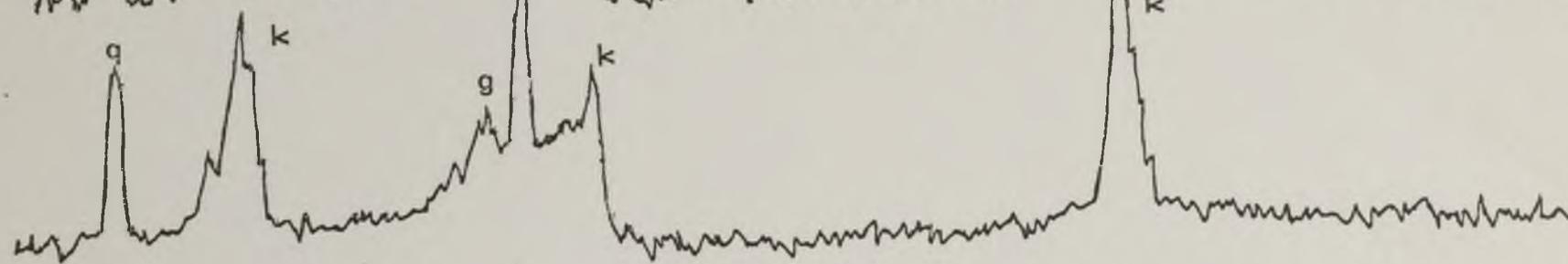
12

8

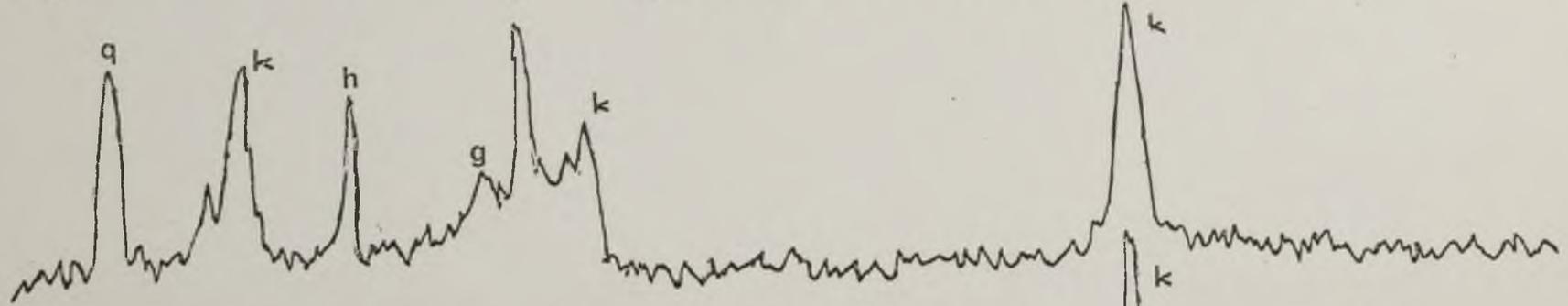
q - Quartz g - Goethite
 k - Kaolinite h - Haematite



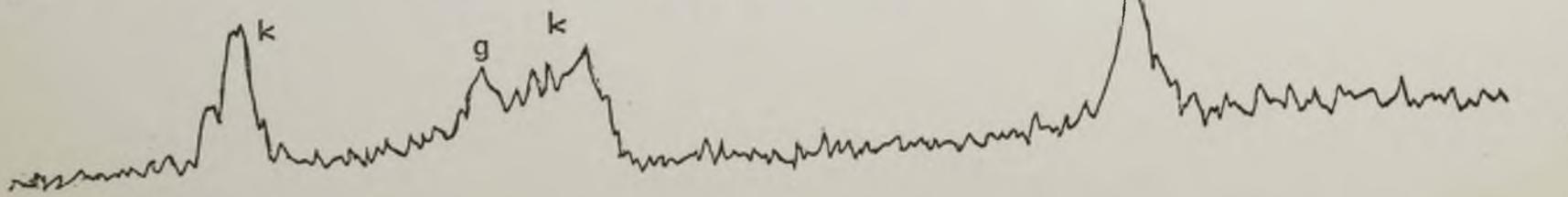
Specimen 40
 9.5 - 2.0mm
 fraction of fines



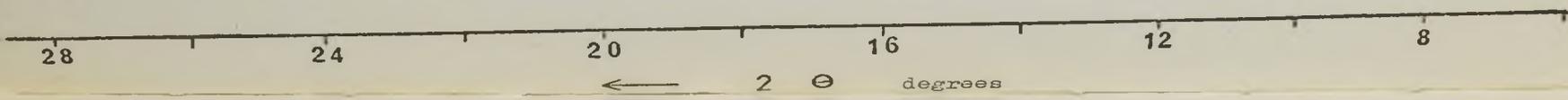
Specimen 41
 2.0mm - 75um
 fraction of fines



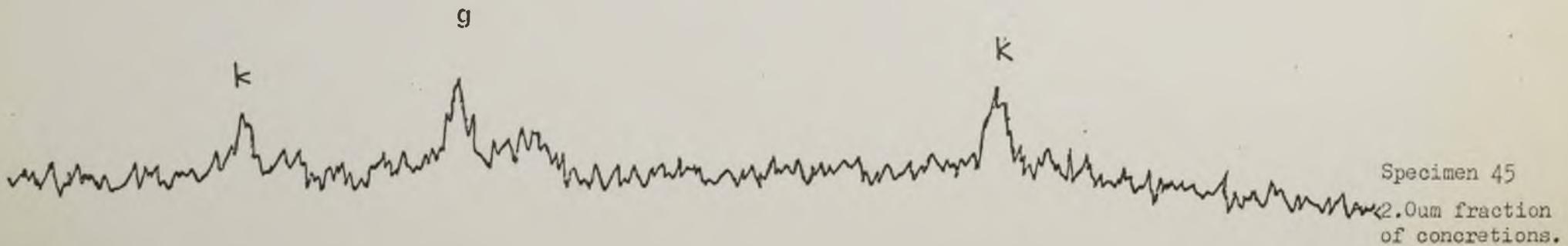
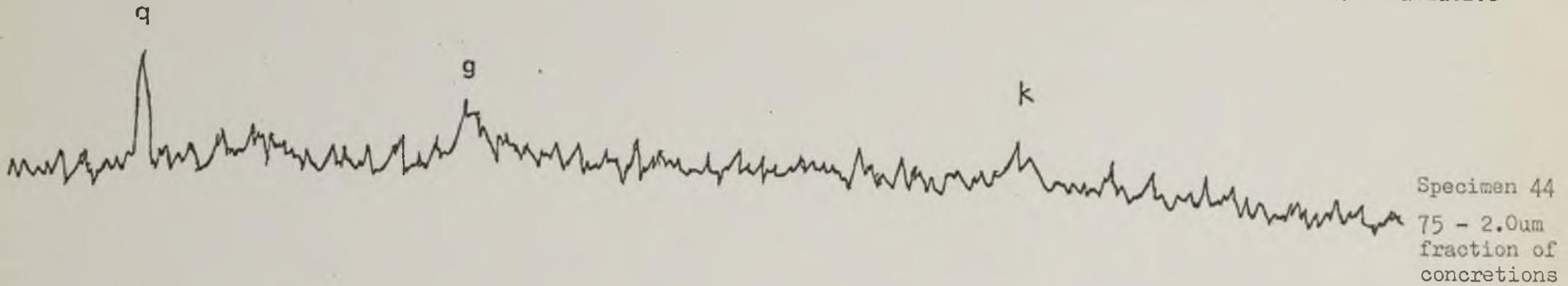
Specimen 42
 75 - 2.0um
 fraction of fines



Specimen 43
 < 2.0um
 fraction of fines



q - Quartz g - Goethite
 K - Kaolinite h - Haematite



28

24

20

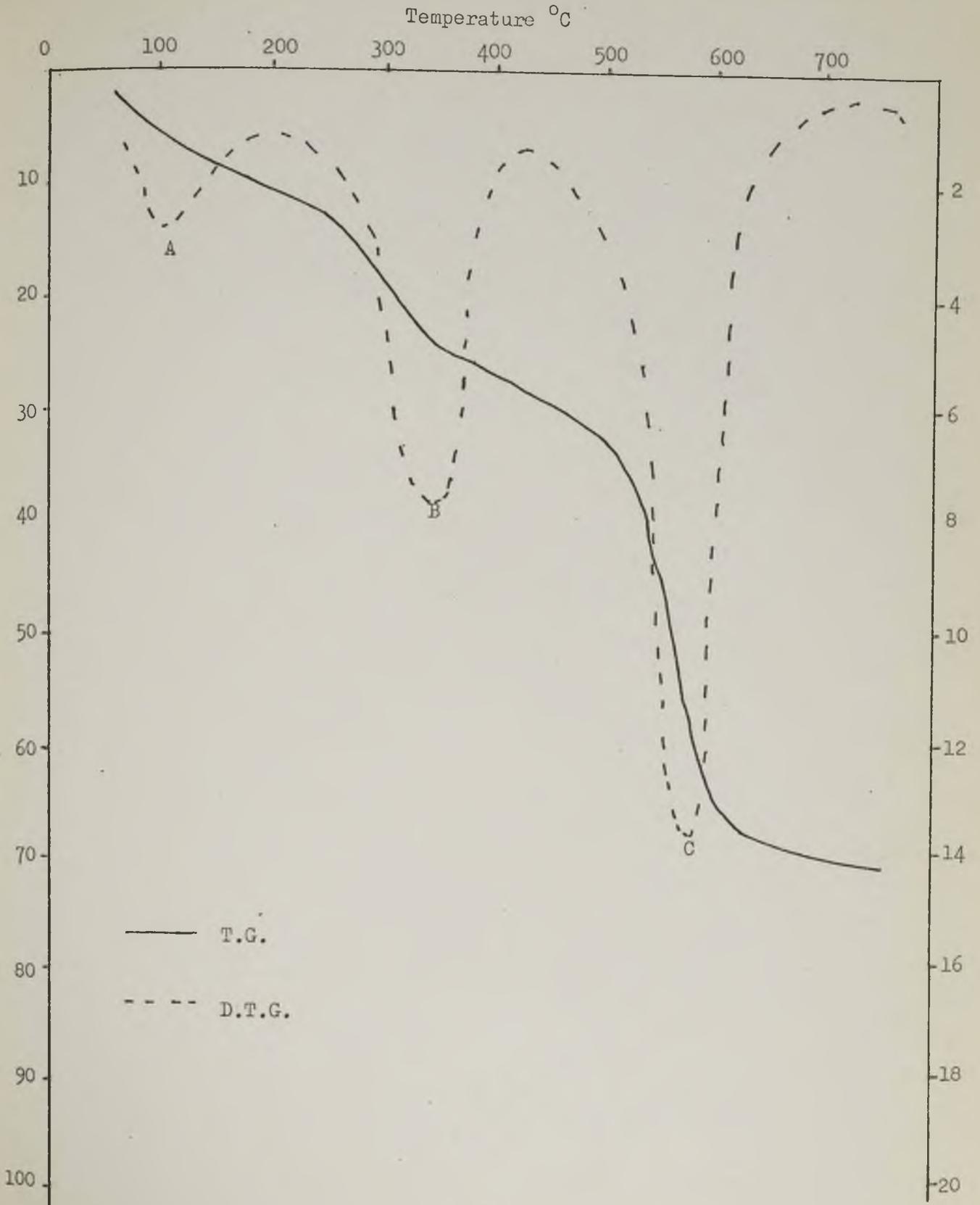
16

12

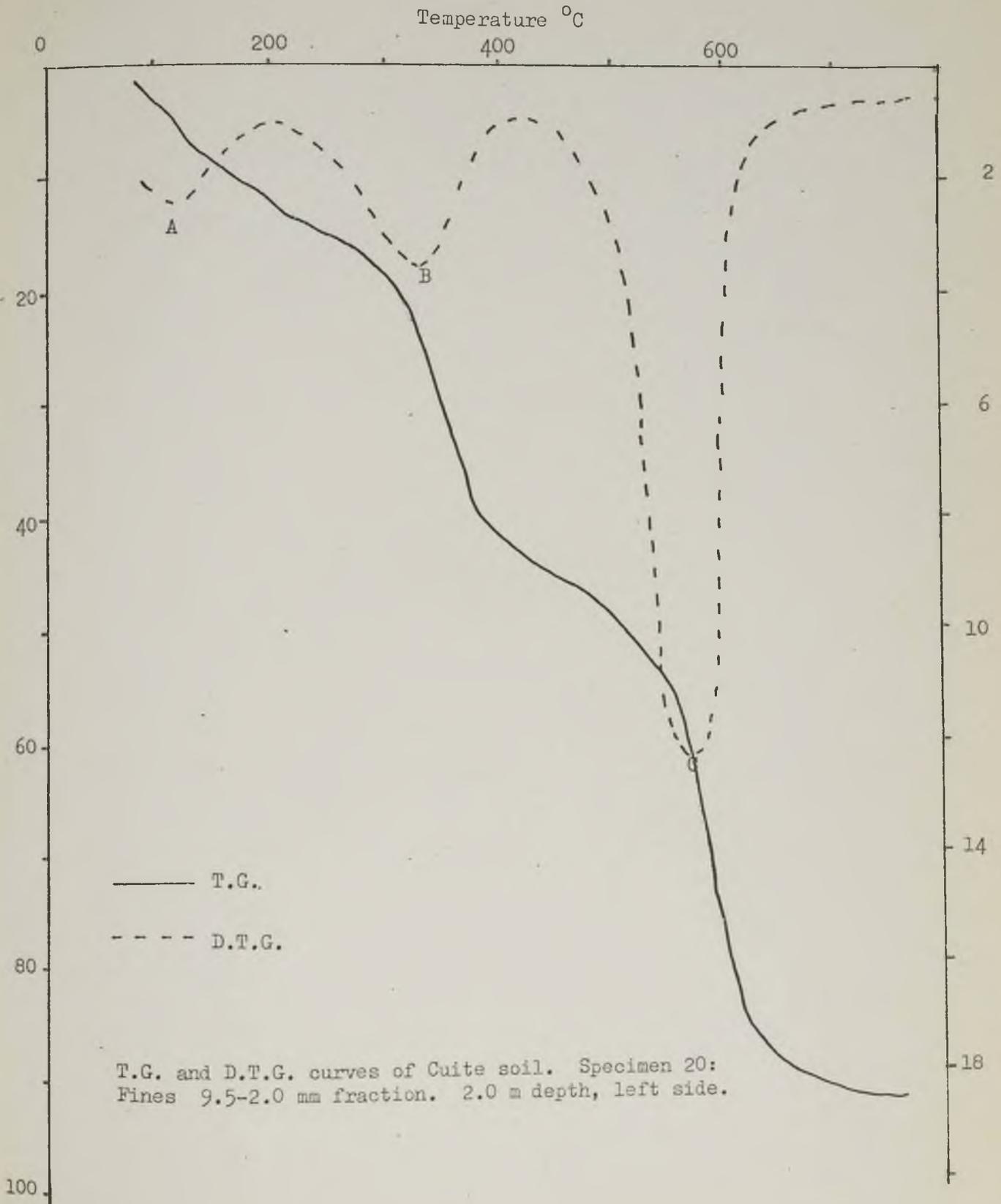
8

← 2 θ degrees

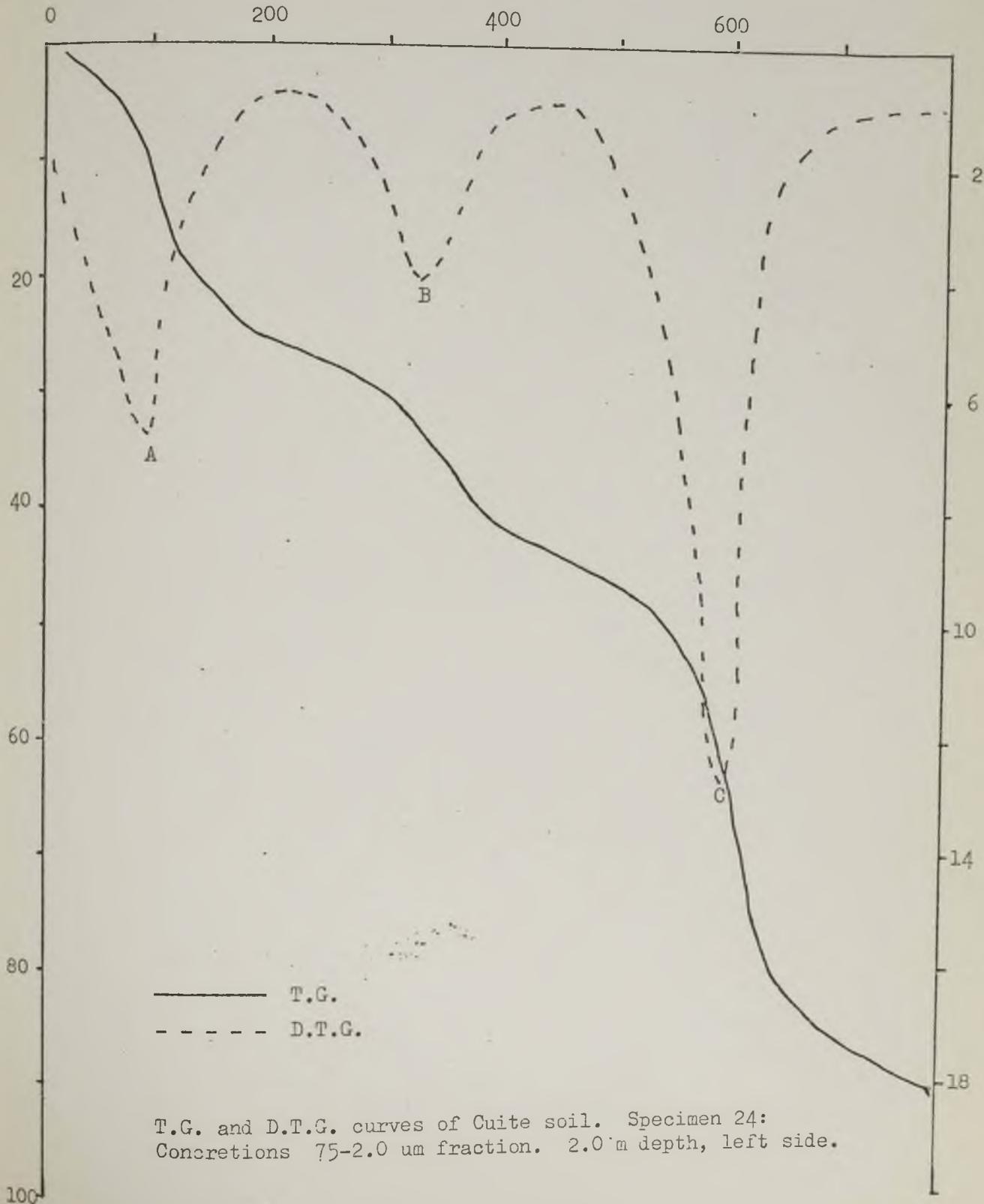
X-ray Diffractogram of samples from the same horizon in Nova Floresta soil. 1.2 m depth. Cu $k\alpha$ radiation.



T.G. and D.T.G. curves of Quite soil. Specimen 10:
Fines 9.5-2.0 mm fraction. 1.0 m depth, left side.

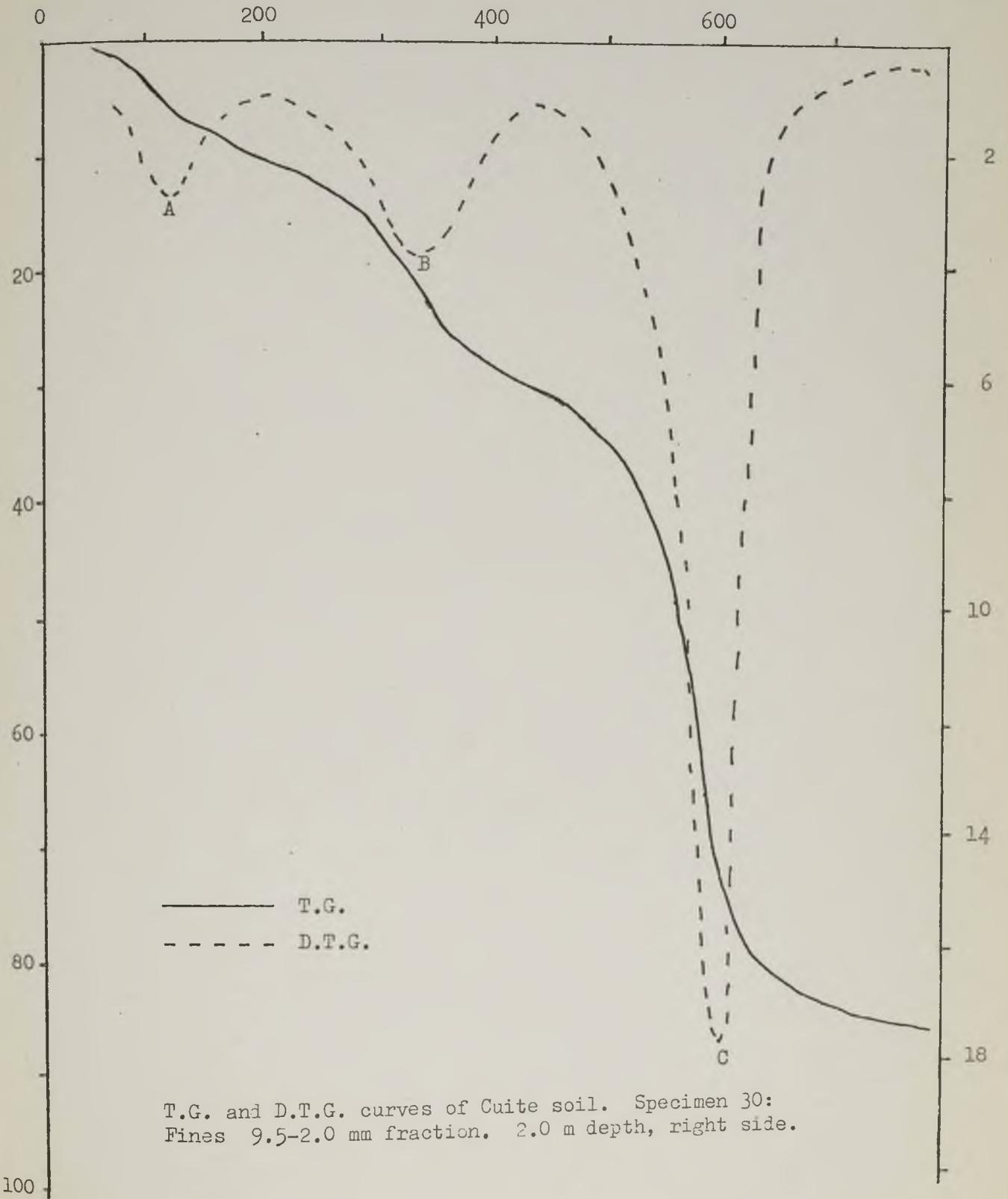


Temperature °C



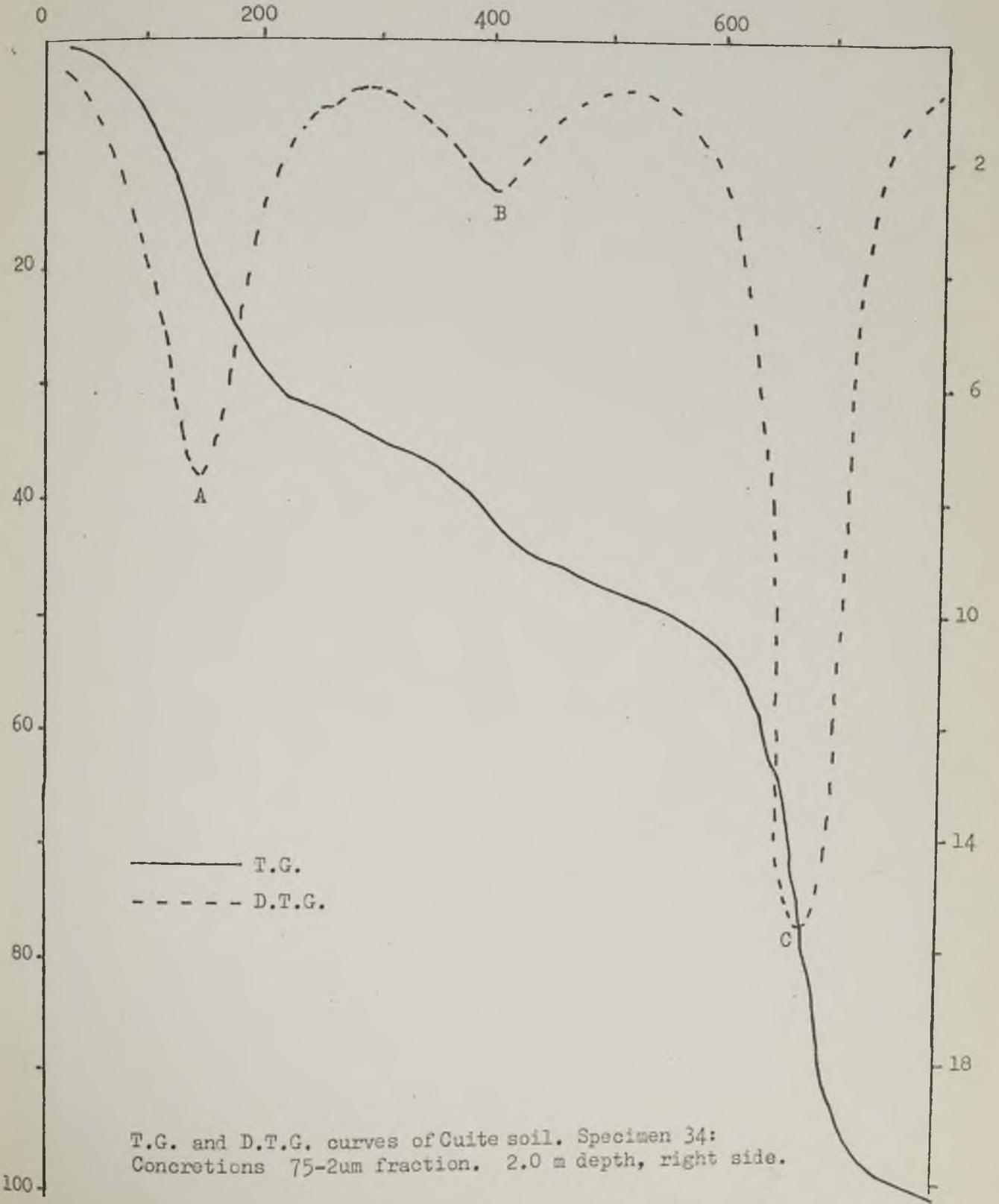
T.G. and D.T.G. curves of Cuite soil. Specimen 24:
Concretions 75-2.0 um fraction. 2.0 m depth, left side.

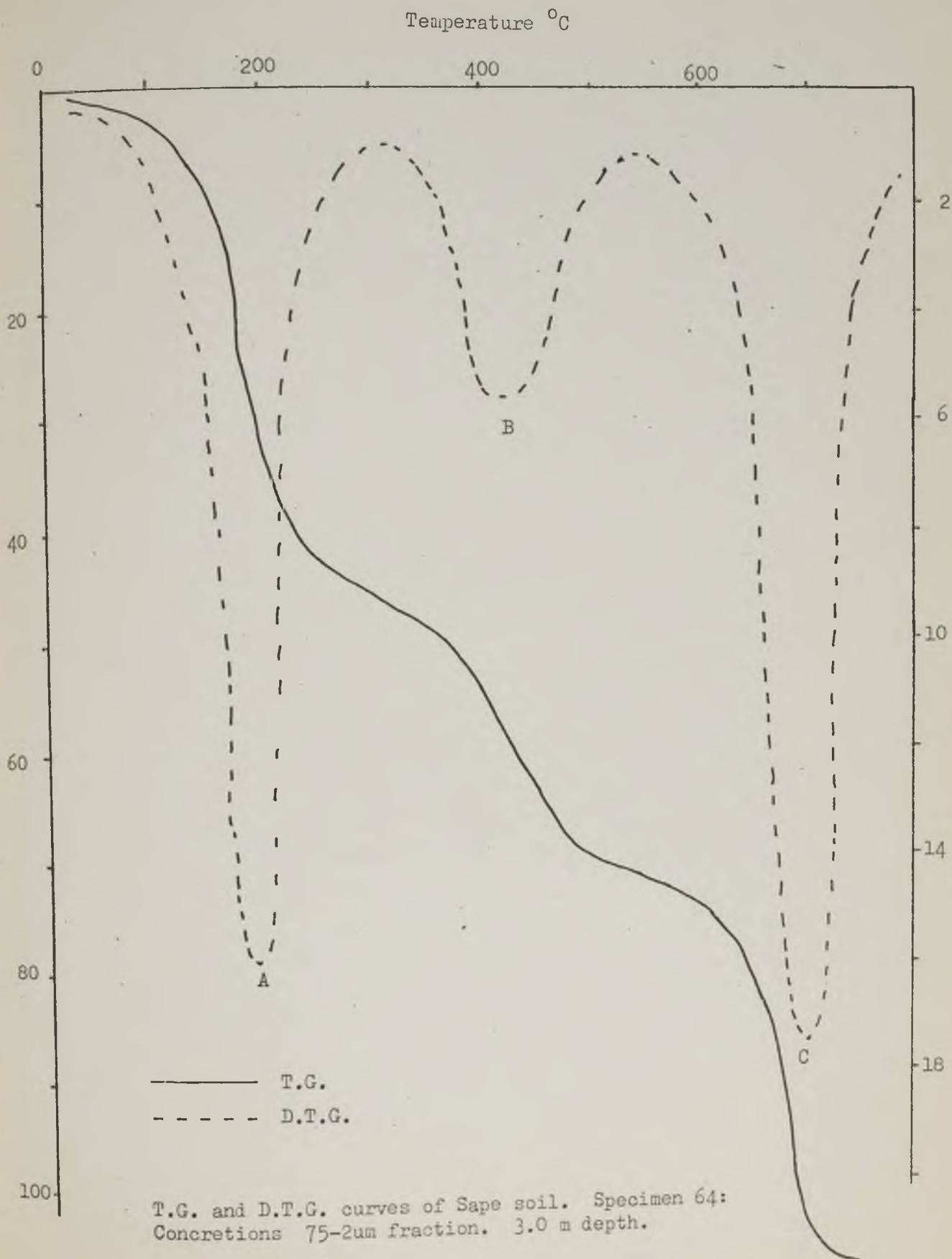
Temperature °C

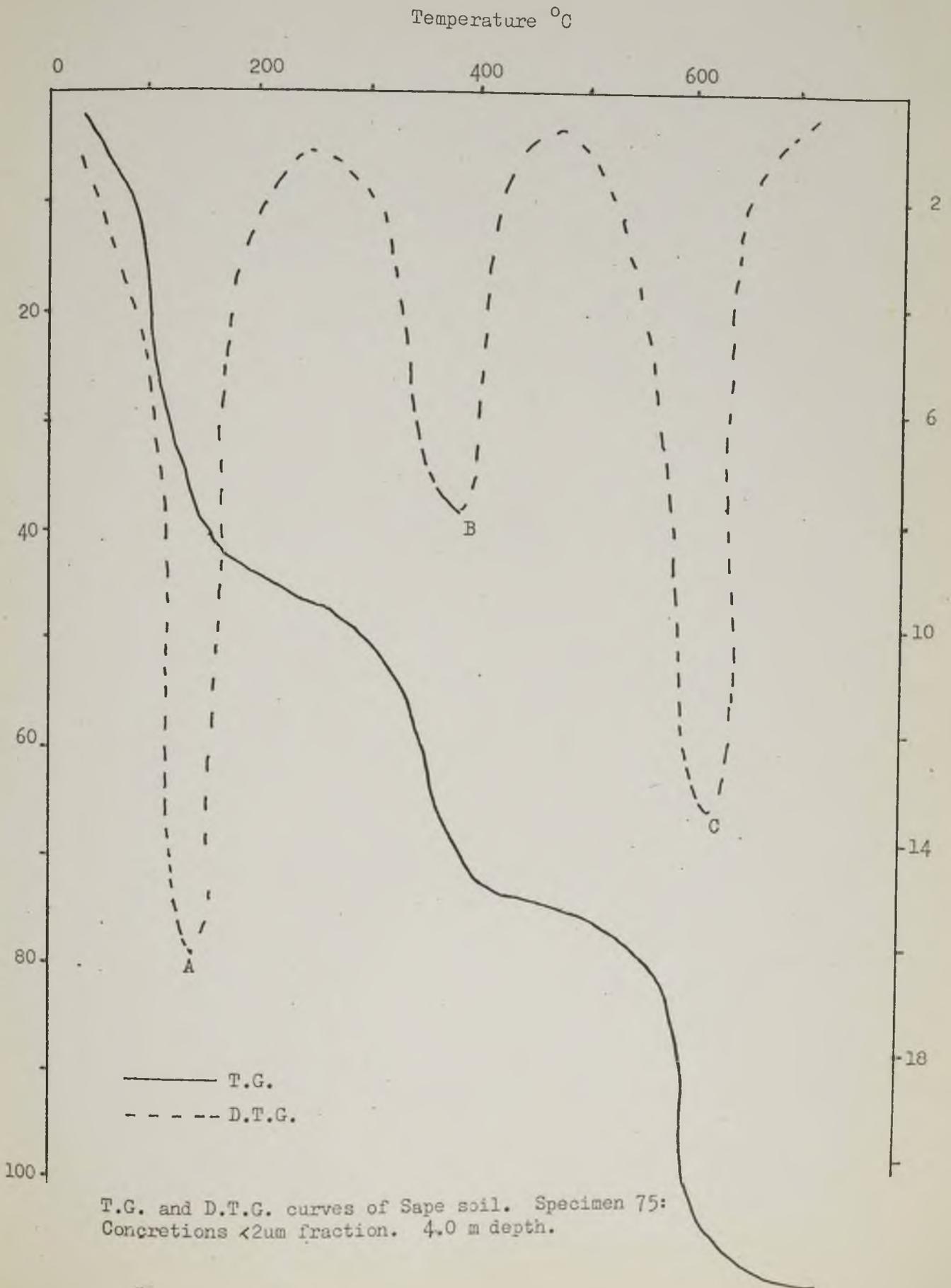


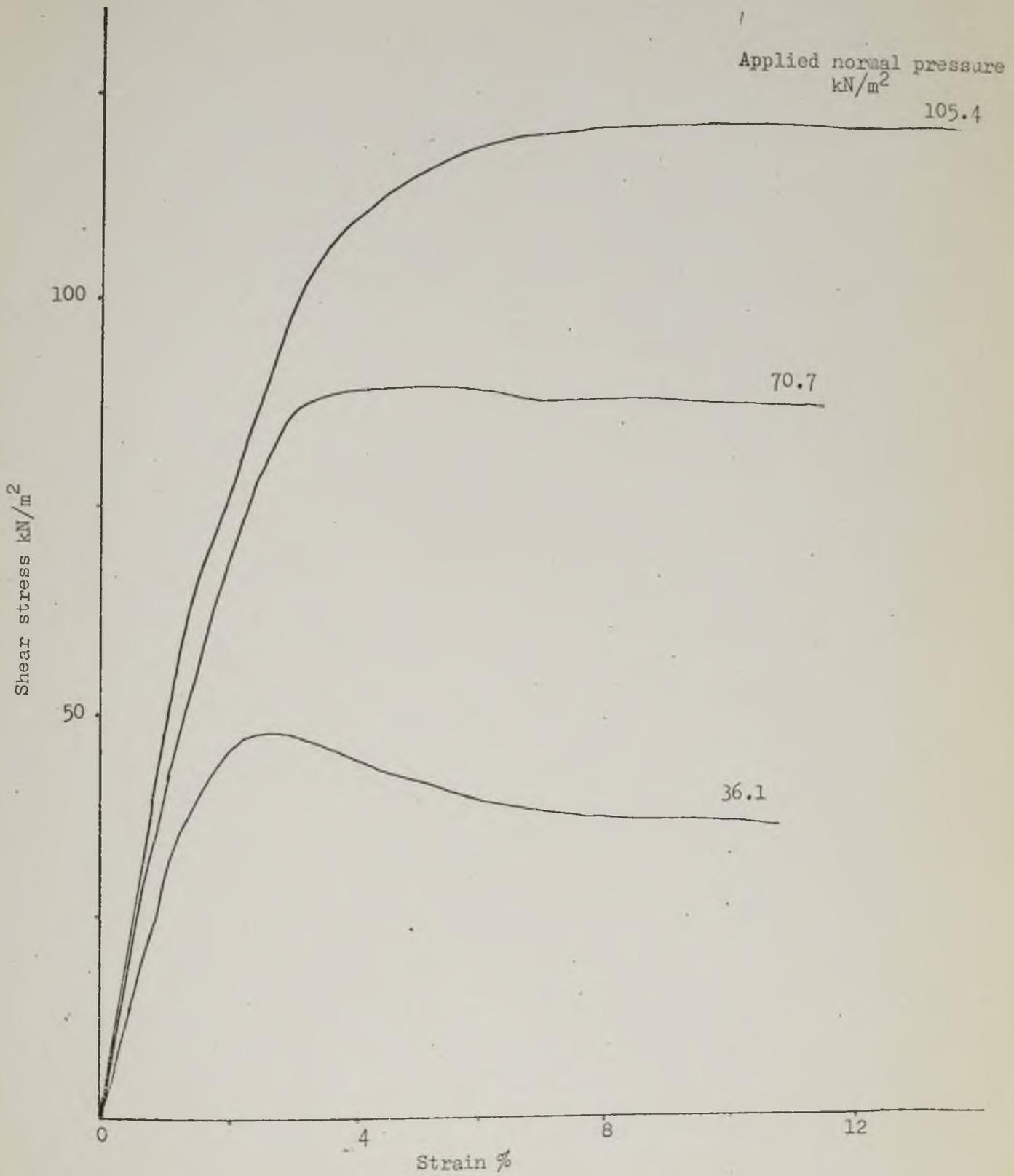
T.G. and D.T.G. curves of Cuite soil. Specimen 30:
 Fines 9.5-2.0 mm fraction. 2.0 m depth, right side.

Temperature °C

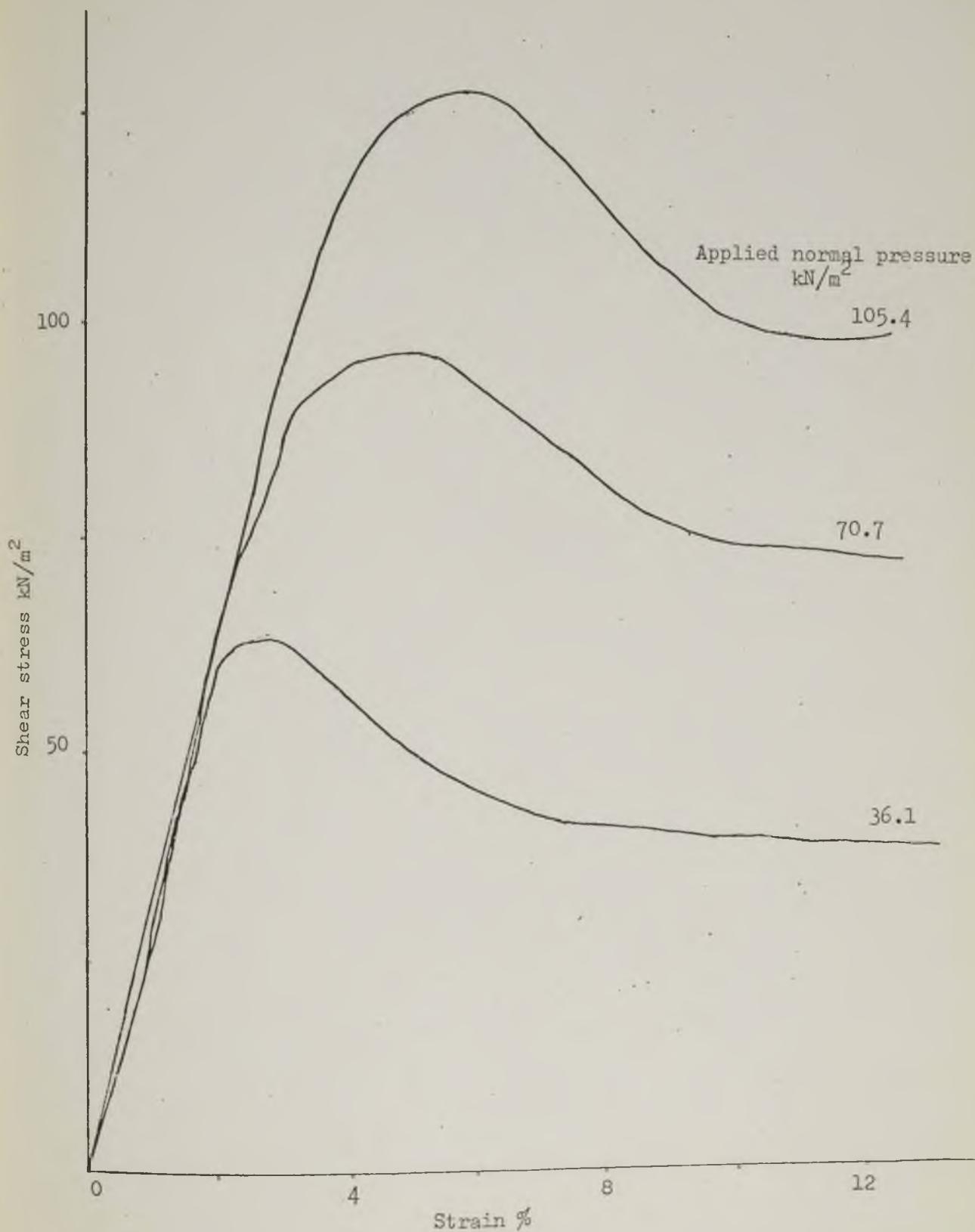




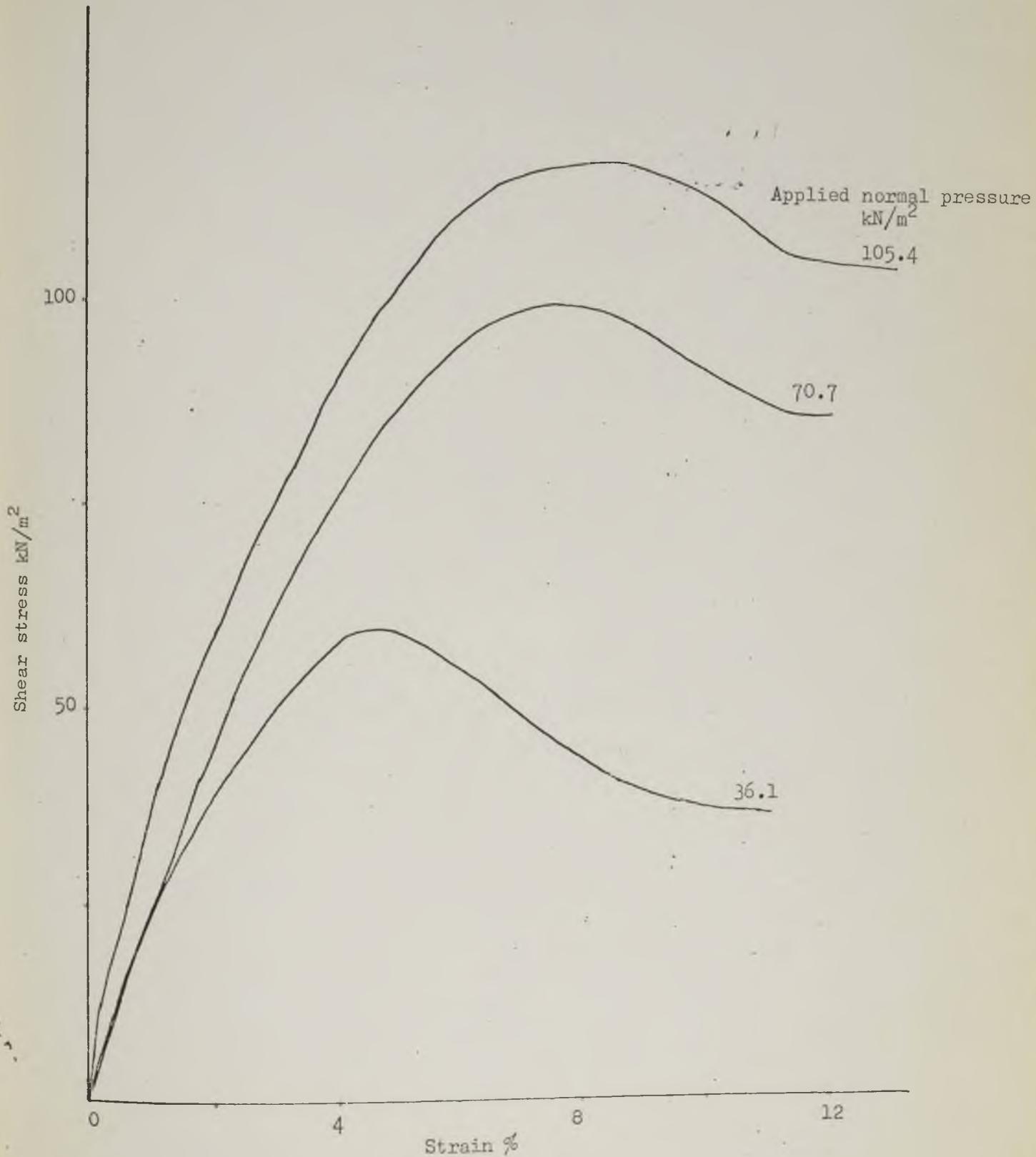




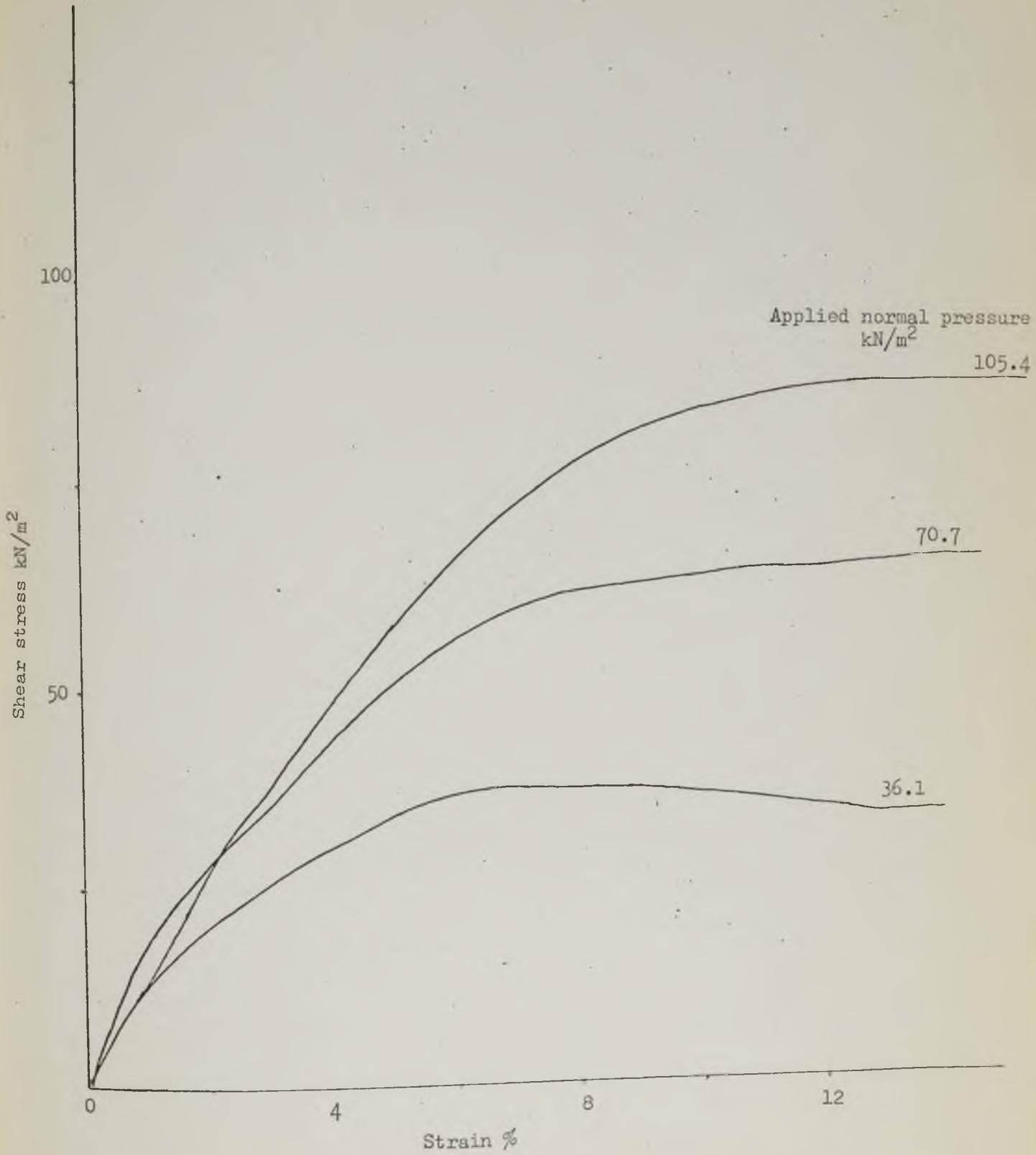
Stress-strain curves for Cuite soil in the direct shear stress at three normal pressures. (Moisture content 12%)



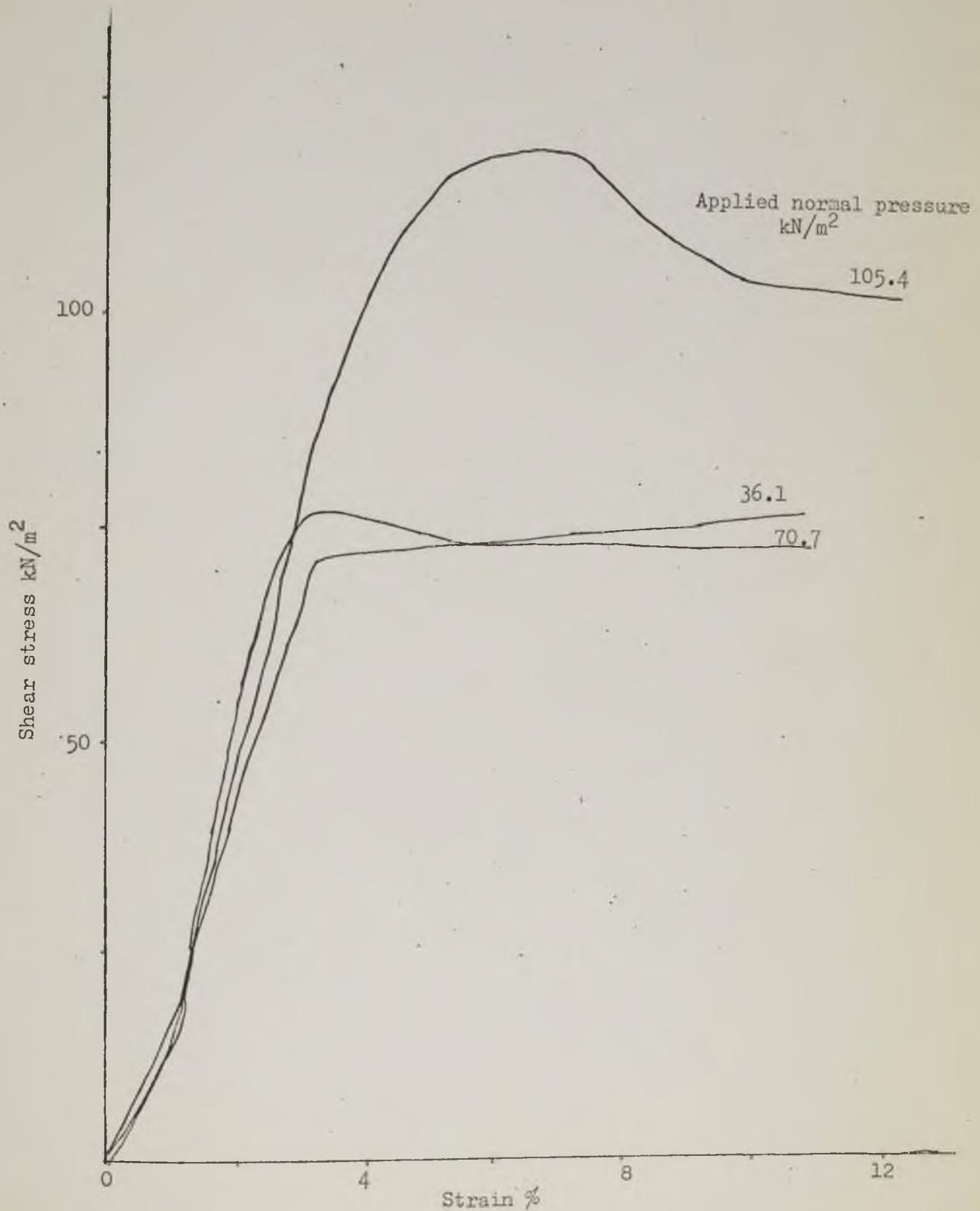
Stress-strain curves for Cuite soil in the direct shear stress at three normal pressures. (Moisture content 13.2%)



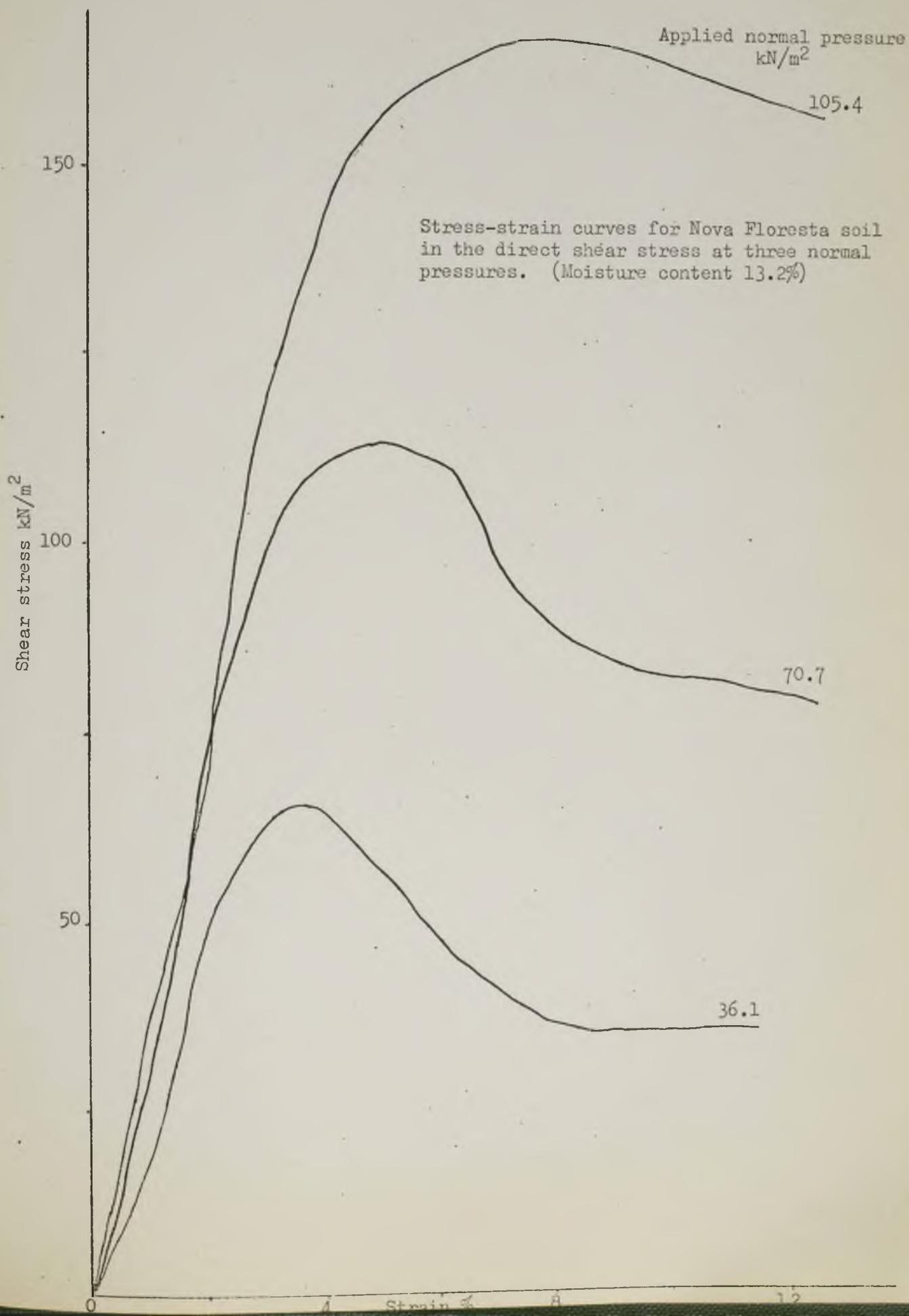
Stress-strain curves for Cuite soil in the direct shear stress at three normal pressures. (Moisture content 14.6%)

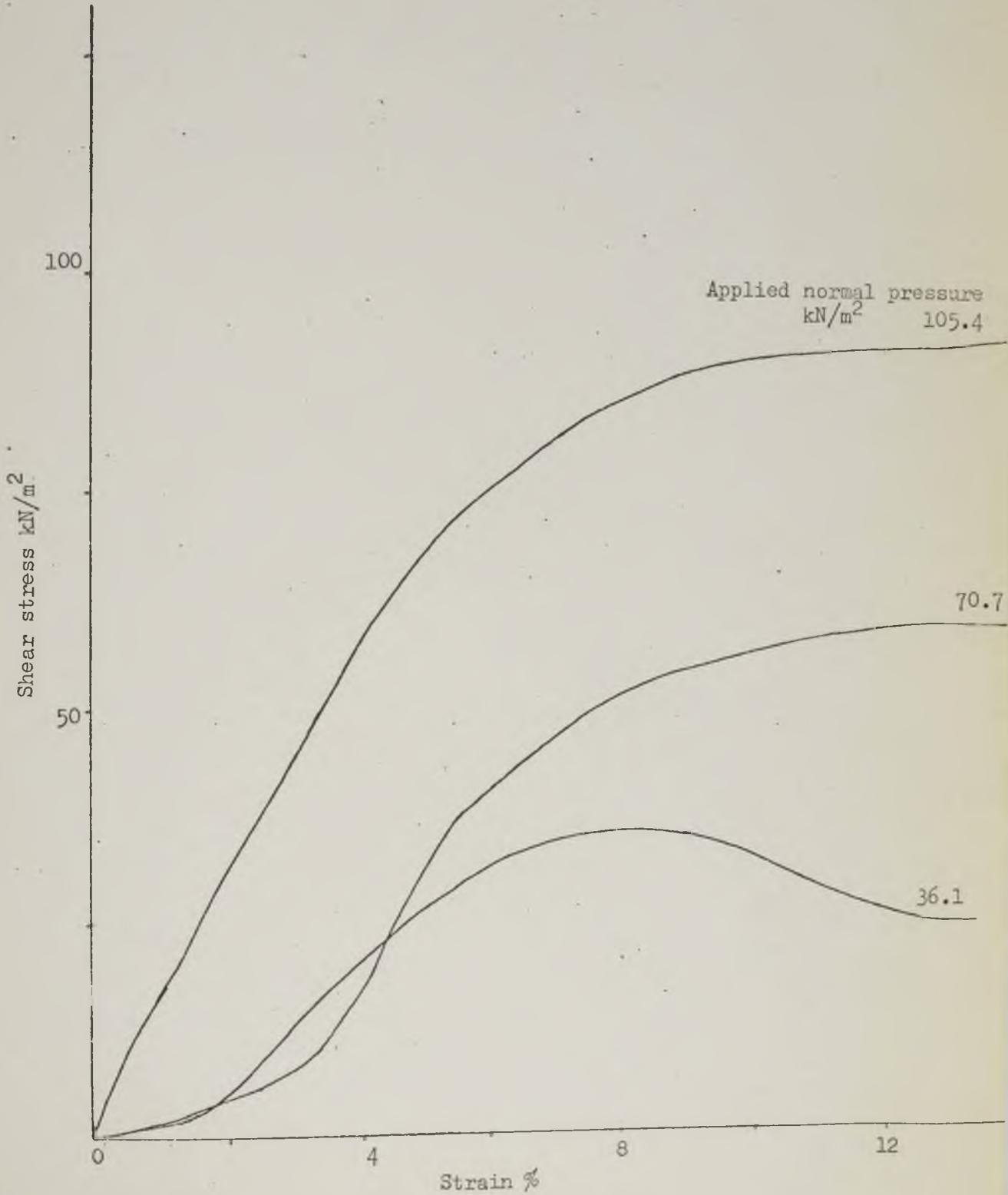


Stress-strain curves for Cuite soil in the direct shear stress at three normal pressures. (Moisture content 17.1%)

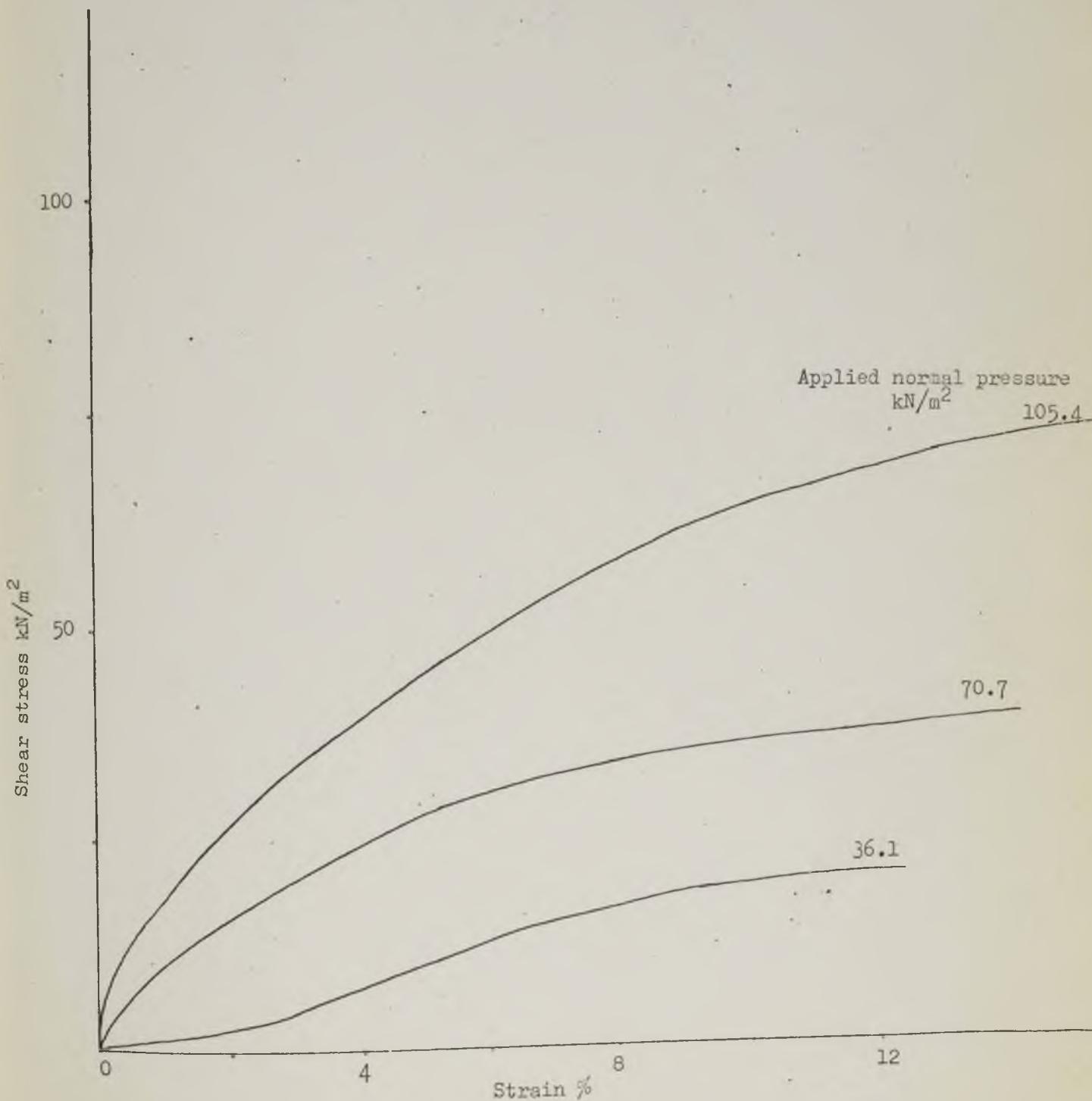


Stress-strain curves for Nova Floresta soil in the direct shear stress at three normal pressures. (Moisture content 12.2%)

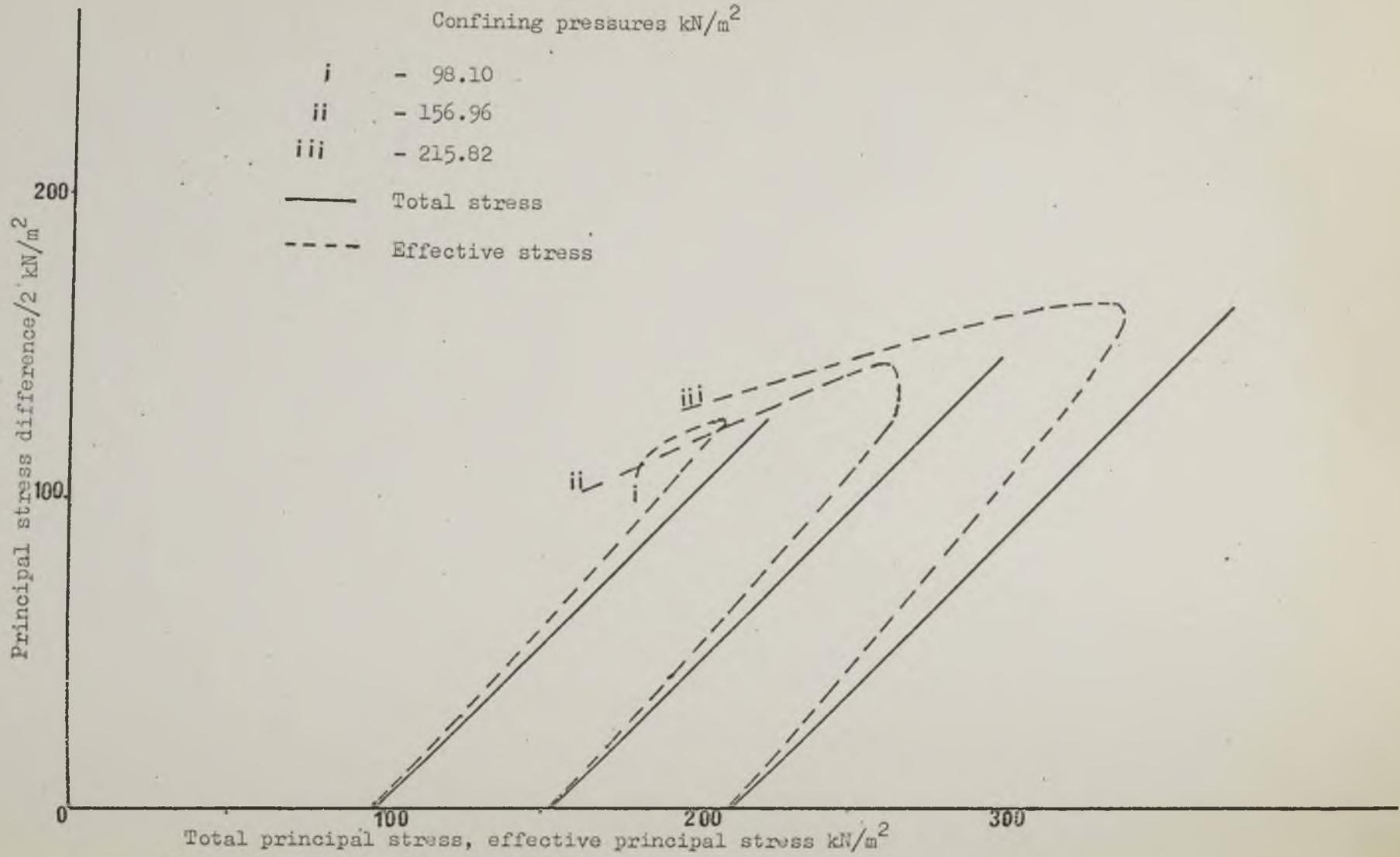




Stress-strain curves for Nova Floresta soil in the direct shear stress at three normal pressures. (Moisture content 14.6%)



Stress-strain curves for Nova Floresta soil in the direct shear stress at three normal pressures. (Moisture content 17.4%)



Stress paths for Consolidated-Undrained triaxial test on Sape soil. / Moisture content 18.1%