

# Limonite – a weathered residual soil heterogeneous at all scales

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Limonite is a residual soil produced by the decomposition of magnesium silicate (olivine) rocks in tropical environments. During weathering most of the original rock is leached away leaving only its iron content, which is precipitated out in the form of iron sesqui-oxides to create a soft and highly porous soil. The predominant mineral present in limonite is goethite, which forms acicular nanoparticles that agglomerate to produce a silty sand with porous particles. The void ratio varies from 2 to 6, with higher values being a consequence of structure-supported voids. An extensive set of laboratory tests have been performed on a limonite soil profile which extends 50 m to rock. These data show that there is no pattern to shear strength with depth, with the shear strength equally likely to be 50 or 200 kPa through much of the profile. It is argued that the shear strength parameters for failure mechanisms, having any significant length, should be based on average values. The letter presents scanning electron microscopy photographs showing the fundamental particles, the results of triaxial tests comparing natural and reconstituted behaviour which show the effects of microstructure on the meso-scale response, and field data to show site variability.

**KEYWORDS:** laboratory tests; microscopy; residual soils

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## INTRODUCTION

Residual soils are directly derived from the parent rock through weathering, without being transported. As a result, the soil particles are not sorted as sedimentary soil deposits, and they tend to be heterogeneous. They also tend not to exhibit any direct relationship between void ratio and stress because the voids arise from the rock being chemically weathered. The process of weathering depends on the nature of the parent rock, the topography, and the climatic conditions (Wesley, 2010; Huat *et al.*, 2012). The chemical weathering process common in sub-tropical and tropical climates leads to the leaching out of silica and the increasing concentration of iron and aluminium sesqui-oxides. This process is called laterisation, and often results in the formation of iron nodules or cemented aggregates, owing to the different polarity of the surface charges of clay and sesqui-oxides. The resulting particle aggregations are often only weakly cemented and are known to break down into finer particles due to thermal and mechanical working, creating difficulties with defining a particle size distribution and soil classification.

This letter discusses a particular residual soil from New Caledonia for which a 50 m deep weathered soil profile has developed, and the process of weathering has resulted in a soil composed almost entirely of the iron oxy-hydroxide, goethite. The microstructure is explored using scanning electron microscopy (SEM). Triaxial tests comparing natural and reconstituted behaviour are used to show the effects of structure on the meso-scale response, and field data are used to show site variability.

## GEOLOGY

The soil specimens investigated were obtained from a nickeliferous lateritic deposit. The nickel lateritic deposits of New Caledonia are derived from a peridotitic oceanic crust. When the peridotite minerals are on the ocean floor, they are serpentinised by interaction with sea water. When the rock is exposed, tropical weathering occurs, which has three major stages (Gidigas, 1976): decomposition, a physico-chemical breakdown of primary minerals; laterisation, the leaching of bases and silica allowing sesqui-oxides to accumulate; and dehydration and desiccation, which ultimately leads to the concentration of crystallised amorphous iron colloids into dense crystalline iron minerals (Golightly, 1981).

Peridotites are magnesium silicate rocks, with the typical chemical formula  $(\text{Mg, Fe})_2\text{SiO}_4$ . In New Caledonia they contain 90% olivine, with very little quartz, 10% iron (III) oxide ( $\text{FeO}_2$ ) and only 0.5% aluminium oxide ( $\text{Al}_2\text{O}_3$ ) (Guilbert & Park, 1986). The olivine and serpentinites of the parent rock are particularly susceptible to weathering because of the absence of quartz and other insoluble material. In the highlands of New Caledonia, the combination of a low water table and high percolation has led to a deep (> 60 m) weathering profile (Golightly, 1981). The general structure of a lateritic weathering profile consists of saprock, saprolite, a transition soil, limonite and an iron oxide (or ferricrete) cap, as the degree of weathering increases towards the surface of the profile (Golightly, 1981).

The parent rock is known as the saprock. The saprolite consists of partly decomposed parent rock. In this zone, significant changes in chemical and physical parameters occur. The transition soil is much like the saprolite, except that smectite minerals and goethite have replaced the parent minerals. The limonite comprises fine-grained goethite and amorphous ferric hydroxides, with rare gibbsite. These soils are also known as oxisols or latosols (Brady, 1974). For the site considered in this letter, layers of saprolite, transition zone and limonite are identified based on their colour; however, the differences in mineralogy and physical parameters are relatively minor. In all

Manuscript received 5 May 2012; first decision 2 August 2012; accepted 31 August 2012

Published online at [www.geotechniqueletters.com](http://www.geotechniqueletters.com) on 28 September 2012.

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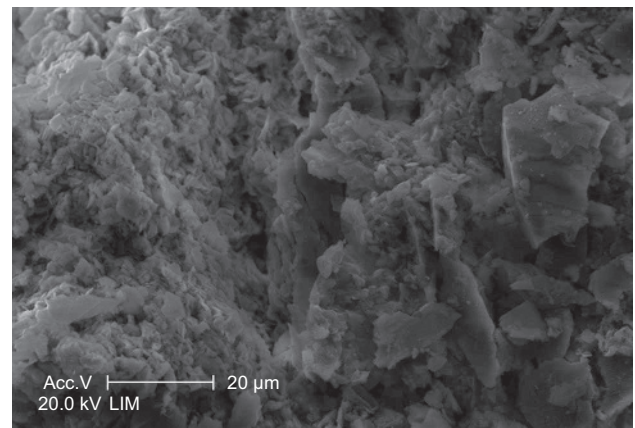
three layers, goethite is the predominant mineral, with increasing (but always very small) amounts of talc, talc soapstone and aluminium chromite with depth. It has also been suggested that the proportion of crystalline goethite, as opposed to amorphous iron hydroxides increases closer to the surface, and in the upper 2 m some hard iron nodules are present. During the weathering process, the rock is completely broken down, accompanied by the precipitation of iron sesqui-oxides and the dissolution of the magnesium and silica. As a result, none of the original parent rock structure survives.

### MICROSTRUCTURE

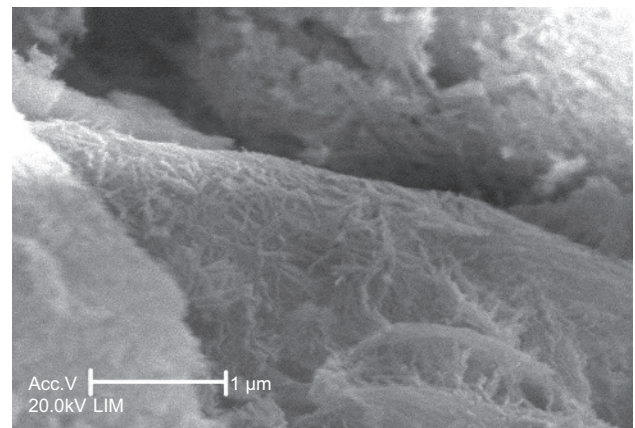
SEM photographs showing the microstructure are presented in Fig. 1. At a magnification of 2000 times, Fig. 1(a) shows the soil to be composed of platey particles, which have typical sizes in the range from 20 to 75  $\mu\text{m}$ . However, on further magnification to 40 000 times (Fig. 1(b)) and 100 000 times (Fig. 1(c)) it can be seen that the plates are composed of a network of interlocked crystals. In some cases, the crystallinity is poorly developed, but the majority of the soil comprises these microscopic goethite ferric oxyhydroxide ( $\text{FeO}(\text{OH})$ ) crystals, which are typically less than 0.05  $\mu\text{m}$  in diameter and approximately 0.5  $\mu\text{m}$  in length. Importantly, the high magnification also shows that the majority of the apparently plate-like particles are in fact empty space. The goethite needles appear to be randomly orientated, generating a very rough particle surface. This rough particle surface probably contributes to the high friction angle of these soils, which is approximately  $42^\circ$ .

As with many residual soils, the characterisation of the soil presents many challenges as conventional procedures for determining particle size, field density and so on are not appropriate (Wesley & Irfan, 1997; Suchowerska & Airey, 2010; Williams & Stocker, 2010). Standard methods of soil preparation using conventional soil dispersants classify the soil as a silty sand; however, with mechanical reworking it is possible to break down the particle aggregates so that all the soil will pass through a 75  $\mu\text{m}$  sieve. Depending on the amount of mechanical reworking, increasing amounts of fine particles are created. After mechanical reworking and dispersion in a solution of lecithin in iso-octane, the particles were found to all be of fine silt size with a  $d_{50} = 5 \mu\text{m}$ . This is consistent with the results of Ding & Pacey (2008), who suggested that this is the size of particles that results from the fracture of the large aggregates. The tendency for aggregation of nanoparticles, such as with goethite, due to Van der Waals forces, is well known, as is the difficulty of their dispersion, which requires significant energy input (Ding & Pacey, 2008).

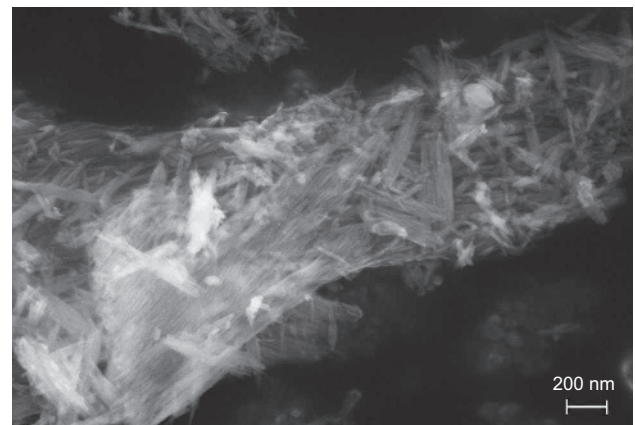
A second aspect of the microstructure concerns the arrangement of the particles. There is evidence of significant structure-supported voids in some SEM photographs indicating the presence of some large voids. Physically, these manifest themselves by water flowing out of the specimens during trimming. Figure 2 shows a comparison of the compressibility of the natural ( $2.6 < C_c < 3.4$ ) and reworked reconstituted specimens ( $C_c \approx 0.5$ ). The natural material can have very high void ratios and high compressibility. In part, the high void ratios are a consequence of the inter-particle voids (see Fig. 1(c)), and this is reflected in the high void ratios of the reconstituted specimens. The difference between the natural and reconstituted soil can be explained by the loss of large structure-supported voids and by the mechanical breakdown of the particle aggregates themselves. Because the process of remoulding reduces particle size, there is a possibility that



(a)



(b)

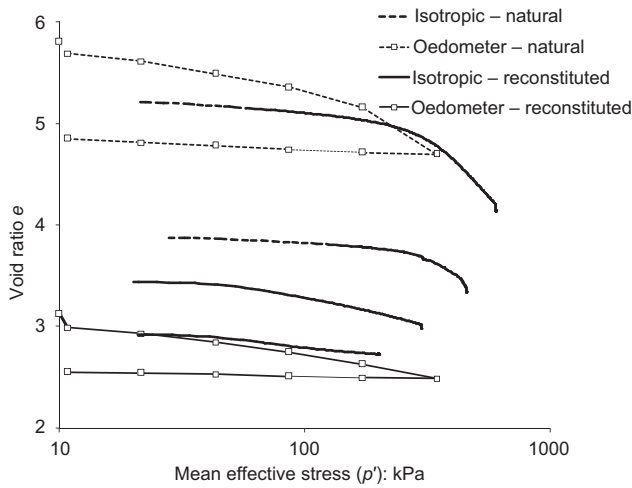


(c)

**Fig. 1.** SEM photographs of limonite (a) at magnification of 2000 $\times$ , (b) at magnification 40 000 $\times$  and (c) 100 000 $\times$

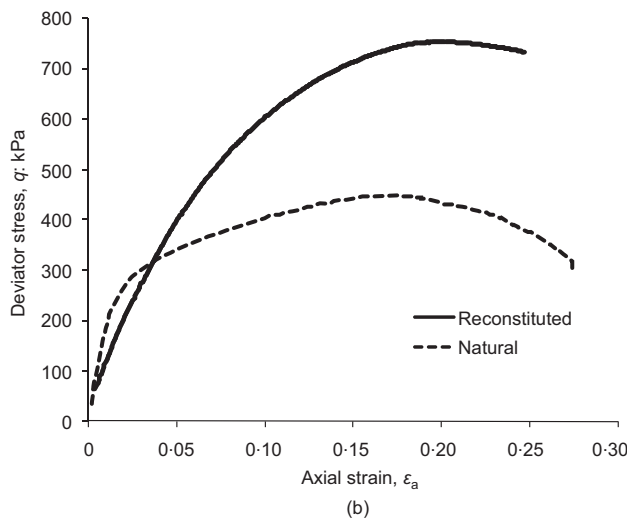
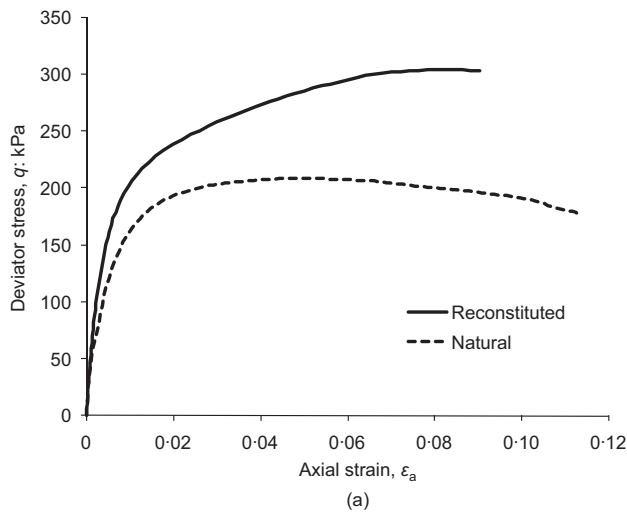
comparisons between natural and remoulded residual soils may overestimate the effects of structure.

In the most extreme situation, the limonite contained only 15% solids, a consequence of the high void ratio and high specific gravity  $G_s$ . Specific gravities of between 3.4 and 4.15 were measured for the soil specimens tested. The upper value represents pure goethite, whereas lower numbers reflect the presence of other minerals. Because of the weathering profile, a pattern of reducing  $G_s$  with depth might be expected; however, this was not observed. The natural soil was fully saturated and void ratios of between 2 and 5.8 were determined, corresponding to



**Fig. 2.** Compressibility of natural and reconstituted limonite

moisture contents of between 60 and 140%. Liquid limits (LL) of between 40 and 120% were measured following mechanical remoulding for up to 30 min, until no further change in LL occurred. In some cases LL was lower than the in situ gravimetric moisture content, which probably



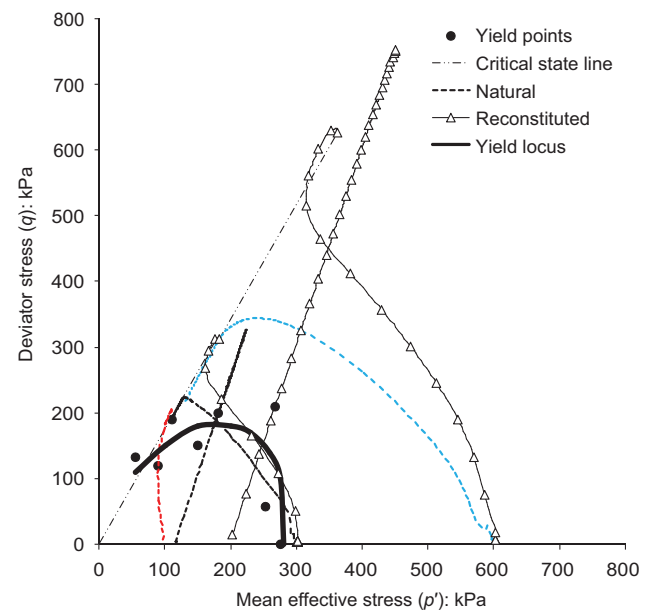
**Fig. 3.** Triaxial stress–strain responses from (a) drained tests with  $p_c = 200$  kPa, (b) undrained tests with  $p_c = 300$  kPa

reflected the difficulty of uniformly breaking down the goethite aggregations, as X-ray diffraction (XRD) studies indicated goethite was the predominant mineral in all specimens.

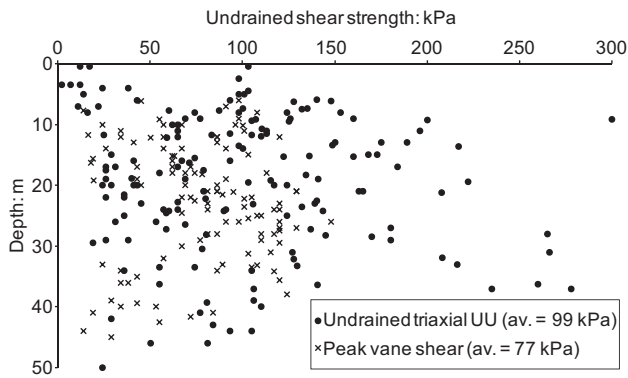
**TRIAXIAL TEST RESULTS**

Specimens for the triaxial and oedometer tests for which data are shown in Figs 2 to 4 were obtained from a single 350 mm cube block sample obtained from a depth of 11 m. The block sample was predominantly red in colour, but it contained a couple of intersecting thin, 2 mm thick, planar features consisting of darker material. On excavation the soil tends to split into blocks along these planes which occur throughout the limonite profile at a scale of hundreds of millimetres, and as a consequence these features are believed to have an important influence on the macroscale response. They are believed to be the result of shear-induced fissuring. The triaxial specimens were selected so that they did not intersect any of the dark veins observed in the block sample. Additional tests were performed on reconstituted specimens which were prepared by crushing oven-dried material with a mortar and pestle until a fine powder with maximum particle size of 75  $\mu$ m was produced. This powder was mixed with water to form a slurry with 100% moisture content and compressed one-dimensionally in a mould to form cylindrical triaxial specimens. Figure 3 shows comparisons between the deviator stress plotted against axial strain responses for undisturbed and reconstituted specimens in drained (Fig. 3(a)) and undrained (Fig. 3(b)) tests. The associated effective stress paths of these and other triaxial tests (Suchowska, 2008) taken from the same block sample are shown in Fig. 4.

Figure 2 shows the compression responses of natural and reconstituted specimens with the greatest and least values of void ratio. For the natural material the void ratio varied from 3.8 to 5.7. This large difference in the void ratio did not produce dramatic differences in the shear response, with all specimens approaching the same ultimate frictional resistance with  $\phi'$  of 42°. This is similar to the friction angle reported by Williams & Stocker (2010) for tests from all depths throughout the limonite profile, and indicates that



**Fig. 4.** Effective stress paths for limonite



**Fig. 5.** Variation of limonite undrained shear strength with depth (after Williams & Stocker, 2010)

the frictional shear strength is everywhere controlled by the agglomerated goethite particles. Most of the natural specimens subjected to drained shearing failed to reach the ultimate frictional state (c.f. Fig. 3(a)) because of heterogeneity within the natural specimens that led to them bowing out. The natural soil showed a relatively stiff response within the yield locus, which is shown in Fig. 4 based on yield points for all the specimens tested from the block sample (Suchowerska, 2008). As the in situ vertical effective stress is approximately 110 kPa, the in situ stress state lies well within the yield locus and the soil is apparently over-consolidated, which is probably related to the structure of the soil. The response of the natural soil at the in situ stress level is consistent with an over-consolidated soil showing a higher undrained shear strength ( $s_u \approx 100$  kPa) than that estimated, assuming  $s_u/p'_c$  constant for normally consolidated reconstituted soil, at the same stress level ( $s_u \approx 50$  kPa), even though the natural soil has a much higher void ratio. However, at higher stresses beyond yield as the structure breaks down, the more compressible natural soil gives lower undrained shear strengths than the reconstituted soil as shown in Fig. 3(b), as would be expected because of its higher void ratio.

As the gravimetric moisture contents of the reconstituted specimens are similar to the minimum values measured in situ, and as the mineralogy does not vary significantly throughout the soil profile, it is believed that the natural and reconstituted specimens are indicative of the range in behaviour that can be expected. However, the site data showing the variation of undrained strength with depth (Fig. 5) show a far greater variation than might be expected from the tests discussed above for which the minimum undrained strength, associated with the highest void ratio, was about 75 kPa. This suggests that the chemical weathering process has produced a soil with highly variable structure and void ratio.

## CONCLUSIONS

This letter presents data on a residual soil, consisting almost entirely of an iron oxy-hydroxide, goethite, formed

by the weathering of a magnesium silicate rock in the subtropical climate of New Caledonia. The goethite is composed of acicular nanoparticles that bond together to form porous particle aggregates, that appear plate-like and have silt to sand sizes. Owing to the mode of soil formation, large structure-supported voids are present in some specimens. The result is a soil with very high void ratios and highly variable voids, so that gravimetric moisture contents vary randomly between 60 and 140% throughout the 50 m deep soil profile. The undrained shear strength also varies randomly between 20 and 300 kPa, and is not well correlated with gravimetric moisture content. However, the effective stress shear strength parameters show remarkable consistency, with the undisturbed and remoulded specimens tested all approaching the same ultimate friction angle of  $42^\circ$ , despite the significant differences in void ratio. It has been suggested that the structure and engineering parameters of this residual soil are heterogeneous at all scales and, as a consequence, average values should be applicable to any failure plane of significant length. However, because of the site variability the average value for any failure plane may vary across the site, and a statistical method would be appropriate for estimating appropriate values of strength for use in design.

The residual soil described in this letter provides challenges at many scales that include: what the factors are controlling the agglomeration of the goethite nanoparticles and how they respond to stress; how to describe the variability of structure-supported voids; is void ratio (moisture content) a useful parameter; and what processes lead to the development of fissures and macrostructure.

## REFERENCES

- Brady, N. (1974). *The nature and properties of soils*, 8th edn. Macmillan Publishing.
- Ding, P. & Pacey, A. W. (2008). De-agglomeration of goethite nano-particles using ultrasonic comminution device. *Powder Technol* **18**, No. 1, 1–10.
- Gidigas, M. D. (1976). *Lateritic soil engineering*. Amsterdam: Elsevier Scientific Publishing.
- Golightly, J. P. (1981). Nickeliferous laterite deposits. In *Economic geology, 75th anniversary volume*. El Paso, Texas: Economic Geology Publishing Co., pp. 710–735.
- Guilbert, J. & Park C. Jr., (1986). *The geology of ore deposits*, New York: W. H. Freeman and Company
- Huat, B. K., Toll, D. G. & Prasad, A. (2012). *Handbook of tropical residual soils engineering*. New York: CRC Press.
- Suchowerska, A. M. (2008). *Nickeliferous lateritic soils: engineering characteristics*. Honours thesis, University of Sydney.
- Suchowerska, A. M. & Airey, D. W. (2010). Characterisation and classification of nickeliferous lateritic soils. *Proc. 3rd Int. Conf. Problematic Soils, Adelaide, Australia*, pp. 319–328.
- Wesley, L. D. (2010). *Fundamentals of soil mechanics for sedimentary and residual soils*. Chichester: Wiley.
- Wesley, L. D. & Irfan T. Y. (1997). Classification of residual soils. *Mechanics of residual soils* (Blight, G. E. (ed.)). Rotterdam: A. A. Balkema.
- Williams, D. J. & Stocker, P. T. (2010). Geotechnical parameters and behaviour of limonite. *Proc. 3rd Int. Conf. Problematic Soils, Adelaide, Australia*, pp. 353–360.

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