



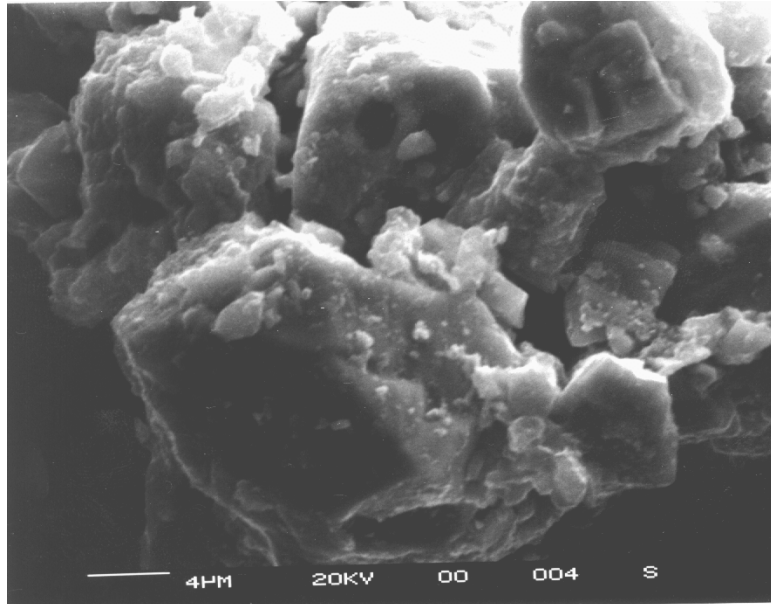
**British
Geological Survey**

NATURAL ENVIRONMENT RESEARCH COUNCIL

REPORT CR/01/096N

A MINERALOGICAL EXAMINATION OF A MAGNETIC TROPICAL BROWN SOIL FROM CAMBODIA AND A POSSIBLE SYNTHETIC ANALOGUE MADE WITH SOIL FROM DEVON

**M T Styles, D C Entwisle, D A Gunn, E Hodgkinson, P D Jackson,
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Front cover illustration: Aggregated grains in clay soil from Cambodia

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BRITISH GEOLOGICAL SURVEY

Commissioned Report CR/01/096N

A MINERALOGICAL EXAMINATION OF A MAGNETIC TROPICAL BROWN SOIL FROM CAMBODIA AND A POSSIBLE SYNTHETIC ANALOGUE MADE WITH SOIL FROM DEVON

1. INTRODUCTION

DERA is carrying out research and developments of land-mine detectors to be used for mine clearance in countries such as Cambodia. Many mine detectors used in Cambodia produce numerous 'false alarms' from soil that appears to contain no obvious source for the spurious or high background signal. A previous study (Styles et al 2001) showed that the response in the Cambodian soils appeared to be caused by very fine-grained magnetite. The response was always present in the finest size fraction ($<63\mu\text{m}$) and in some cases in coarser fractions which is probably due to incomplete disaggregation during sieving. The Devon soils also produced a response from the mine detectors but in contrast this was largely in the coarser fraction due to pebbles of weathered volcanic rock.

Following discussions with DERA it was agreed to carry out further research to gain a better understanding of the causes of the response by the tropical soil and explore the possibility of producing a 'tropical soil' analogue by mixing fine-grained magnetite with soil from Devon.

A range of tasks were agreed to achieve these ends and are listed below.

1. Test mine detector response on sample 11 from Five Elms Lane Exeter, 10 cms depth, as per other samples in previous report
2. Kappa meter magnetic susceptibility measurements on all samples
3. Detailed mineralogy of sample 7 and either sample 10 or 11 depending on results of 1. To include XRD and SEM analysis
4. Prepare simulated magnetic tropical soil from a UK soil with added magnetite powder so that it has similar kappameter response to the Cambodian soil.
5. Produce test cell to check variation of conductivity with moisture content, carry out comparative tests on

- a) Cambodian soil,
 - b) Simulated tropical soil
6. Provide purchasing information for kappameter and conductivity meters
 Check existing reports on tropical soils for information relevant to mine detector performance
 Check regulation etc. on soil import

The samples examined during both studies are listed below with those studied in detail in this report highlighted.

Table 1. Locations of samples

No.	Sample location	MPG Code
1	Wat Takoat	G169
2	Wat Takoat, roadside laterite soil	G170
3	Krisela soil	G171
4	Krisela laterite	G172
5	Boengkrasararea	G173
6	Samlot	G174
7	Battambang	G175
8	Posbury Clump Surface sample	G176
9	Five Elms Lane Newton St Cyres	G177
10	Posbury Clump 10cm depth	G178
11	Five Elms Lane Newton St Cyres, 10 cm depth	G432

2. GENERAL COMMENTS ON TROPICAL CLAY SOILS PERTINENT TO THE INVESTIGATIONS UNDERTAKEN IN THIS REPORT.

2.1 Formation of tropical clay soils (pedogenesis)

In humid tropical climates such as those found in South East Asia, residual soils dominate the landscape. Tropical residual soils are those formed 'in place' by intense weathering/alteration of the underlying parent rock in tropical and sub-tropical climatic environments. The parent rocks may be both igneous (e.g. basalt lavas, tuffs, granites, etc) and sedimentary (e.g. sandstones, limestones, etc.).

Two very broad categories of residual soils may be considered: tropical '*black*' soils and tropical '*red*' soils. The former category, which includes those soils termed 'black cotton soils' or 'black swelling clays', comprise a relatively distinct group of soils, rich in

smectite clay minerals, whose behaviour is dominated by volume changes (ie. shrinking and swelling) when they are subjected to changes in natural moisture content. These soils invariably form in areas of poor drainage. However, there is some confusion as to what materials comprise the so-called tropical 'red' soils and the terms used to describe them.

It is a misconception to consider tropical 'red' soils as forming a distinct, clearly defined soil type as they encompass a wide variety of soils whose material, engineering and, in all probability, 'electrical' properties vary considerably. This variation has been reflected in the ever increasing literature on tropical soils since the 1960's, which has seen red, brownish red, reddish brown and brown soils described as 'laterites', 'laterite soils', 'lateritic soils', 'non-lateritic tropically weathered soils', 'latosols', and 'tropical red clays'. Under certain conditions, hardened horizons, often referred to as "laterite", may be associated with the soil profiles. These form as a result of the accumulation of iron which, in some areas, may develop into a continuous sheet of indurated ferricrete (grains cemented by Fe-oxides/hydroxides) ("laterite") forming a surface or near-surface duricrust (hard layer). However, not all red clay soils harden irreversibly on exposure to form laterite (as defined by the original description of Buchanan, 1807) and it is unfortunate that the terms 'lateritic clays' and even 'laterite' are still used by some workers to describe any reddish/brownish red tropical soil. It should be appreciated that some of the soils broadly described as tropical 'red' clays, may be brown/dark brown rather than red. For example 'andosols' are tropical soils that range from dark brown or yellowish brown to brownish red in colour, but are part of the same weathering process that, given the appropriate conditions, leads to the development of more typical tropical red soils. Andosol soils are typically found in the tropics at higher and wetter elevations on recent volcanic rocks (particularly ashes, tuffs and pyroclastic material) where allophane clay minerals are formed from the rapid weathering and alteration of volcanic 'glass'.

The tropical weathering of parent rocks is complex but primarily involves the progressive chemical alteration of primary minerals, the release of iron and aluminium sesquioxides, increasing loss of silica and the increasing dominance of *new* clay minerals (such as smectites, allophane, halloysite and, as weathering progresses, kaolinite) formed from dissolved materials. Continued depletion of silica under prolonged weathering in hot humid climatic zones may eventually cause alteration of kaolinite to the aluminium oxide, gibbsite, as free alumina is formed in the soil profile. At any particular site, mineral composition and microstructure will depend on numerous factors such as the nature of the parent material, the age of the land surface (time for soil formation), climate, topography and drainage conditions.

Duchaufour (1982) developed a scheme that attempts to put the soils formed under the complex tropical weathering process into a logical context. The scheme places emphasis on the compositional soil characteristics (particularly mineralogical composition). Duchaufour recognised three main phases of residual 'red' soil development in tropical areas: fersiallitisation (fersiallitic soils), ferrugination (ferruginous soils) and ferrallitisation (ferrallitic soils). Each of these phases are typically related to a broad set of climatic conditions and are characterised by an increasing degree of weathering of primary minerals, an increasing loss of silica, and an increasing dominance of *new* clay minerals formed from dissolved materials. This overall process is generally referred to as 'ferrallitisation' (or commonly, and confusingly, as 'laterisation'). Ferrallitisation

represents the final phase, where complete weathering of all primary rock-forming minerals has taken place under hot humid climatic conditions over a long period of time. Thus the development of these soils should be seen as phases in the same weathering process, forming part of a weathering continuum from fersiallitic through to ferrallitic soils.

2.2 Moisture sensitivity of tropical clay soils

An important aspect of these soils is that as weathering proceeds new clay minerals are formed that are in equilibrium with the climatic, temperature and moisture conditions acting upon them. As conditions change so do the minerals that are formed. The final ferrallitic soil phase of development is dominated by fairly 'stable' clay minerals (such as kaolinite). However, the fersiallitic and ferruginous soils are characterised by clay minerals that are 'less stable' and often in a poorly crystalline state (amorphous or gel-like). These clays may undergo irreversible changes when subjected to drying and/or marked changes in humidity. These changes not only influence the nature of the clay minerals themselves but also the structural (or 'fabric') characteristics of the soil that, in turn, influence its density, porosity and moisture-holding properties. **This is a critical issue as tests undertaken on a tropical soil that has been allowed to dry out may bear little relation to the material properties (and almost certainly the electrical properties) of the same soil *in situ*.** As the mineralogical changes subject to drying are irreversible, re-wetting of the dried material in the laboratory does not return the soil to its *in situ* pre-dried state.

2.3 The Cambodian "Battambang soil" investigated in the current study.

This soil was received in a disturbed and dry condition, thus some irreversible alteration to the clay minerals and the soil structure is likely to have occurred, rendering it fundamentally different in terms of its material, and very likely its electrical, properties when compared to the same soil *in situ*. Thus the soil structure and mineralogy determined for the Battambang sample by scanning electron-microscopy and X-ray diffractometry, described in this report, may not accurately reflect the nature and composition of the soil in its pre-dried condition. The presence of amorphous (non-crystalline) clay minerals such as allophane and of the various forms of halloysite and even poorly-crystalline kaolinite are often difficult, if not impossible, to detect effectively following pre-drying of these soils. The presence of these minerals is significant as they possess a marked affinity for water and, therefore, affect the moisture holding characteristics and capabilities of the soil. This, in turn, will influence the electrical properties.

Without more detailed information as to the location of the Battambang soil (including elevation, topographical and geological setting) it is difficult to ascertain precisely what the Battambang soil is classified as pedologically. The FAO-Unesco soil map of the world (albeit at the very small scale of 1:5 million, showing pedological soil types in very broad terms only) indicates that much of the tropical clay soils in Cambodia are 'Nitosols' or 'Ferric Acrisols'. These soil types equate approximately to 'ferrisols' in the Duchaufour scheme. Ferrisols are part of the Ferruginous group of soils where weathering of the primary minerals are not complete. In very humid zones the ferrisols tend to occur at higher altitudes where lower temperatures slow down ferrallitisation, or

where soil erosion rejuvenates the soil profiles and prevents their complete (ferrallitic) development. It is interesting to note that the pedological description of Nitosols found in Kenya (based on the FAO-Unesco classification scheme) includes the following information:

Typical Nitosols are well-drained, dark red or dark reddish brown friable fine clayey soils. The dry fine earth usually shows a degree of magnetism.

Investigations by BGS of similar soils in Kenya and Indonesia have shown that the ferrisols in these countries contain variable proportions of amorphous or poorly crystalline (disordered) types of the clay minerals halloysite and kaolinite. Allophane may also have been a likely constituent but is difficult to detect. The particular soil mineralogy and structure (fabric) although widespread in tropical/sub-tropical environments is very rarely found naturally in temperate climates.

The Cambodian ‘Battambang’ soil investigated in this study is more brown than red in colour, and in its dried state (and when wetted up from its dried state) has the appearance and “sandy silt-like” texture of an ‘andosol’ or andosol-like (‘andic’) soil of the type found on volcanic rocks at higher and wetter elevations in Indonesia. However, as noted above, when received and tested from a pre-dried state, it is difficult to determine the *in situ* mineralogy of such soils accurately, either by mineralogical analytical techniques or by simple engineering index tests (e.g. modified plasticity and particle size analysis tests). These tests may give a crude indication of the likely clay minerals present in a moist soil sample, not previously dried prior to testing. Knowledge of the geological bedrock, underlying the location from where the sample was collected, along with elevation and climatic (rainfall) conditions, would also give an indication as to whether the soil is likely to be an andosol, or other soil type.

3. MINE DETECTOR RESPONSE TESTS ON SAMPLE 11.

The bulk soil sample from Five Elms Lane, Crediton, collected from 10 cm depth is a red-brown clayey soil with a few small pebbles of volcanic rock fragments and sandstone. It was wet sieved and all the size fractions were tested for mine detector response in the same manner as the previous samples. A few of the previous samples were re-tested at the same time to ‘calibrate’ the response of the detector. This measure of response is the observers’ qualitative estimate of the volume and tone produced by the speaker on the detector. The detector response is due to a combination of the magnetic susceptibility and the electrical conductivity of the soil.

The results of the test are shown in Table 2 along with the results from the previous tests for comparison. Samples 1 and 5 from the original batch of samples caused no response from the mine detector and were omitted from the tables. The sample generally gave a weak response, less than all the other samples examined and was therefore not used for further tests. Sample 10 was used for the detailed examination as a typical example of UK red clay soil.

Table 2. Response of mine detector to each soil size fraction

Particle-size (mm)	2	3	4	6	7	8	9	10	11
Bulk									
+25	*	*	*	*	*		*	*	*
-25 + 19		*	*	*	*		*		*
-19 + 12.5				*	*				*
12.5 + 9.5				*	*				*
9.5 + 4.75									
-4.75 + 2.36									
-2.36 + 2									
-2 + 1									
-1 + 0.5									
-0.5 + 0.25									
-0.25 + 0.125									
-0.125 + 0.063									
-0.063									

Very strong

Strong

Moderate

Weak

None

*

4. KAPPAMETER MAGNETIC SUSCEPTIBILITY TESTS

The bulk samples and all the size fractions of the samples were tested for their magnetic susceptibility with a kappameter and the results are given in Table 3. A kappameter is a small, hand held magnetic susceptibility meter, roughly the size of a large flashlight, that is used by geologists to measure drill cores and hand samples of rocks. It measures the susceptibility of a volume a few cm in diameter. For reference a typical non-magnetic rock gives values less than 1, a relatively magnetic rock with a few percent of iron ore might give values around 5, whilst the pure magnetite concentrate used to spike the analogue sample gives values around 300. Some of the size fractions of the Cambodian soils were too small to give accurate readings and will probably err on the low side, while some samples were so small no reading was taken. The units of the kappameter are 10^{-3} SI units.

Table 3. Kappameter readings on size fractions

	2	3	4	6	7	8	9	10	11
Bulk	0.44	0.64	1.08	21.4	22.8	0.26	0.19	0.28	0.36
+25	0	0	0	0	0	0.1	0	0	0
-25 + 19	0.02	0	0	0	0	0.03	0	0.05	0
-19 +12.5	0.02	0	0	0	0	1.76	0.03	5.3	0
12.5 + 9.5	0.04	0.01	0.07	0	0	0.19	0.07	0.69	0
9.5 + 4.75	0.04	0.05	0.1	3.66	4.11	0.78	0.15	0.89	0
-4.75 + 2.36	0.35	0.12	0.2	5.62	9.29	1.11	0.16	1.5	0
-2.36 +2	0.03	0	0.61	1.96	3.99	0.97	0.24	0.46	0.12
-2 + 1	0	1.04	0.3	5.82	8.24	1.35	0.25	1.09	0.26
-1 + 0.5	0	0.83	0.35	5.07	4.12	1.3	0.27	0.59	0.25
-0.5 + 0.25	0	0.92	0.4	9.21	8.64	0.7	0.29	0.75	0.28
-0.25 + 0.125	0	0.81	0.5	7.6	9.39	0.4	0.28	0.4	0.23
-0.125 + 0.063	0	0.71	0.43	3.57	3.83	0.31	0.28	0.23	0.17
-0.063	0.48	1	1.37	32.2	20.4	0.48	0.82	1.24	0.58

The kappameter readings have been grouped as weak less than 1, moderate 1-5, strong 5-10 and very strong greater than 10 and then shown in a table with boxes coloured similarly for the mine detector response Table 4.

Table 4 Comparison of mine detector response and kappameter readings.

	M K		M K		M K		M K		M K		M K		M K		M K					
Partidesize(mm)	2	3	4	6	7	8	9	10	11	2	3	4	6	7	8	9	10	11		
Bulk	Yellow	Blue	Orange	Blue	Orange	Red	Red	Orange	Blue	Orange	Blue	Orange	Blue	Orange	Blue	Orange	Blue	Yellow	Blue	
+25	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
-25+19	Blue	Blue	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
-19+12.5	Blue	Blue	Blue	Blue	*	*	*	*	*	Yellow	Yellow	Blue	Blue	Blue	Blue	Blue	Blue	Blue	Blue	*
12.5+9.5	Blue	Blue	Blue	Blue	*	*	*	*	*	Yellow	Yellow	Blue	Blue	Blue	Blue	Blue	Blue	Blue	Blue	*
9.5+4.75	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Blue	Blue	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-4.75+2.36	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-2.36+2	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-2+1	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-1+0.5	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-0.5+0.25	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-0.25+0.125	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-0.125+0.063	Blue	Blue	Blue	Blue	Orange	Orange	Orange	Orange	Orange	Orange	Yellow	Yellow	Blue	Blue	Orange	Blue	Blue	Blue	Blue	Blue
-0.063	Orange	Blue	Orange	Blue	Orange	Red	Red	Orange	Blue	Orange	Blue	Orange	Blue	Orange	Blue	Orange	Blue	Orange	Blue	Orange

Colouring as Table 2, left hand column mine detector response, right hand column kappameter response.

Some interesting observations can be made from these sets of readings. The highly magnetic Cambodian samples, 6 & 7 give broadly similar response on both pieces of

equipment but the mine detector response from the bulk sample of 7 is quite noticeably greater despite the fact that they have almost identical kappameter readings. Many of the other samples cause strong mine detector responses but have only weak magnetic susceptibility, with values around 1. This suggests there is a factor other than the presence of magnetite that causes the mine detector response.

5. PREPARATION OF SIMULATED MAGNETIC TROPICAL SOIL

The aim of this part of the work was to try to produce a sample of soil with similar magnetic susceptibility to the most magnetic Cambodian soil, sample 7. Approximately 1 kg of sample 10 was sieved at 4mm to remove the pebbles and used as the host soil. A sample of approximately 2 kg of 'pure' magnetite was obtained from Kilton Industries. These are not suppliers of chemical grade products, they are a supplier of bulk mineral concentrates for industrial purposes, usually in batches of 20 tons. The magnetite supplied was crushed material that is size graded to be less than 300 mesh (53 μm) and was used directly without further treatment.

Small plastic bags containing roughly 30 grams of magnetite were added to the soil until a kappameter response of around 20 was achieved. This required the addition of around 230 grams of magnetite. The magnetite was then added to the soil and the sample thoroughly homogenised by stirring in a beaker. The kappameter reading on the homogenised sample was around 40. This sample was also tested for mine detector response which showed that it caused a strong response but not as strong as sample 7, despite the fact that it had a higher kappameter reading (40 compared to 20).

This sample was used for the moisture and resistivity cell measurements.

6. DETAILED MINERALOGICAL STUDIES

Samples 7 and 10 were split to take a subsample for thorough disaggregation by ultrasonic probe and resieving on 63 μm to obtain a truer fine fraction. This was used for XRD analysis.

6.1 Characterisation of soil samples by Scanning Electron Microscopy (SEM)

6.1.1 Sample description and preparation

Samples of soil from Battambang, Cambodia, and Posbury Clump, Devon, were received. These had previously been size separated and oven-dried. The following samples were examined:

Sample 7 (G175) 63 to 2 μm fraction Soil from Battambang, Cambodia

Sample 10 (G178) <63 μm fraction Soil from Posbury Clump (10 cm depth), Devon, UK

Simulated tropical soil sample 10 mixed with magnetite

'Pure' magnetite Obtained from mineral dealer

Samples 7 and 10 were found to be caked, presumably due to the presence of clays. The mixture of sample 10 with magnetite was more disaggregated resulting from the homogenisation process.

Stub mounts were prepared as follows: a small amount of the finest, most well-disaggregated material from each sample was shaken over sticky carbon tape on an aluminium stub and any excess material was then shaken off. The stubs were then given a thin (~ 20 nm) carbon coating.

6.1.2 Scanning Electron Microscopy (SEM) observations

Scanning Electron Microscopy produces greatly magnified images of solid materials using a beam of electrons rather than light (as in conventional microscopy), so that features smaller than the wavelength of visible light can be viewed. Objects as small as 1 micrometer (10^{-6} m) or less can be viewed in this way.

The stubs were examined using a Cambridge S250 Scanning Electron Microscope in both Secondary Electron Imaging (SEI) and Back-Scattered Electron Imaging (BSEI) modes, at between 10 and 20 kV accelerating voltage. Qualitative chemical analysis was performed using a 'Link' Energy-Dispersive Spectroscopy system.

In Secondary Electron mode, images are produced of the surface topography of the sample. This technique has been used to show the grain size distribution, morphology of grains, and overall texture of the soils. Back-Scattered Electron Imaging produces images in which the densest materials (those which contain the heaviest elements) appear brightest. For example, an iron oxide mineral grain would appear very bright compared to a less dense grain of quartz (silicon dioxide). The Back-Scattered Electron image is produced from deeper in the sample so topography is less pronounced and very thin surficial material may not be visible at all. Qualitative chemical analysis was performed to determine the chemistry of individual mineral grains.

6.1.3 SEM study results

Cambodian 'Battambang' soil – sample 7 (G175) (63 to 2 μm fraction)

Typical Secondary Electron images (see Plates 1 and 2) show the grain size distribution of the material. Most of the material consists of grains greater than 10 μm in diameter. These coarser grains are mostly of quartz (usually well-rounded), or

more angular Fe oxide, with a variable Ti component; these are likely to be magnetite. One well-rounded grain contained Fe and Mn in equal proportion, with minor Ti and Ni. Analyses of these coarser grains included variable amounts of Al and Si in similar proportion in some places. This may be due to surficial coatings of clay, which are visible on some grains in Plate 2.

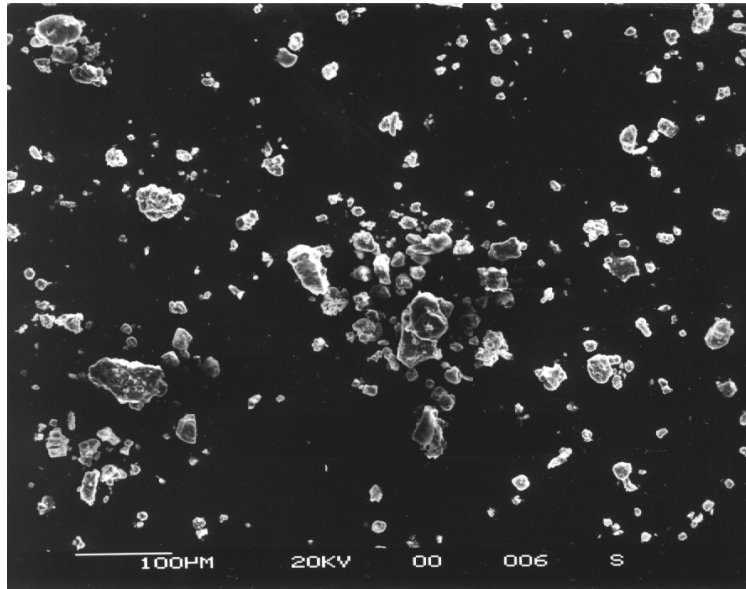


Plate 1. Sample 7 (Battambang soil). Secondary Electron Image showing typical grain size distribution. A 100 μm scale bar is shown in the lower left corner.

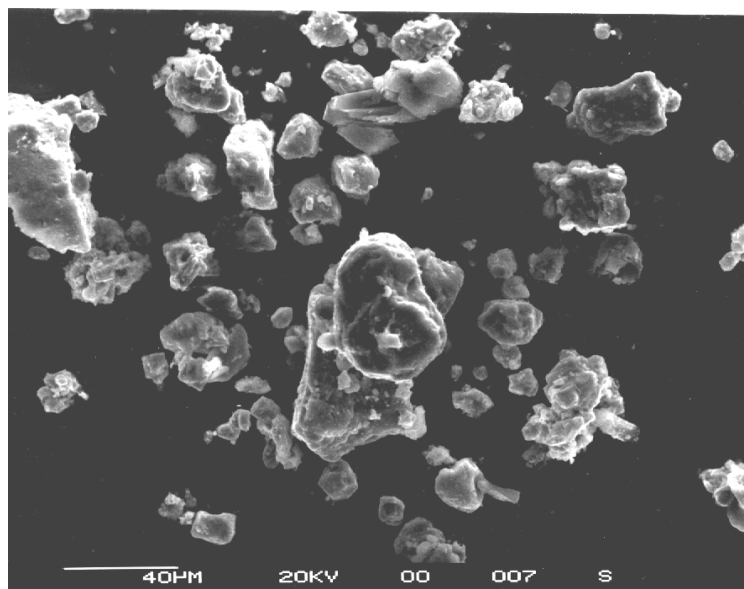


Plate 2. Sample 7 (Battambang soil). Secondary Electron Image showing typical grain size distribution. Quartz occurs as well-rounded grains (e.g. large central grain). Fe-Ti oxides are more angular (e.g. large grain beneath this). The small, flaky or platy coatings on larger grains are probably clay. Note the loose aggregates of small grains (e.g. to lower right).

Many of the finer (<10 μm) grains are loosely aggregated into larger particles (e.g. Plate 3). These are the features called peds and are described in several of the reports from the BGS 'Tropical red clay soil' project'. In these aggregates, generally well-crystallised grains of Fe-Ti oxides and quartz are coated in a poorly crystalline material which may act to bind the grains together. The Fe-Ti oxides are generally equant (often octahedral or cubic) and contain Ti either in equal proportion to Fe possibly ilmenite (FeTiO_3) or Ti as a minor component, probably magnetite (Fe_3O_4). These grains are likely to be of magnetite with a variable Ti component, although hematite may also be present. Occasionally minor amounts of Mn and Cr are also detected; these are common constituents in magnetite.

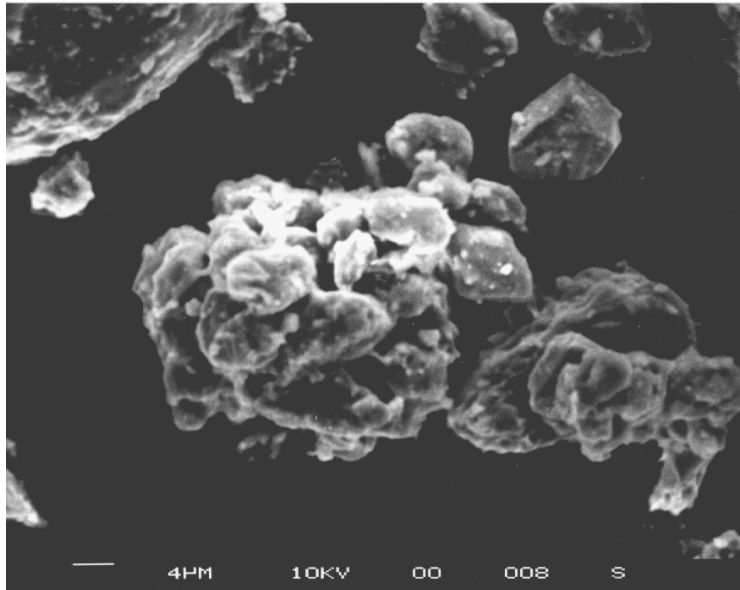


Plate 3. Sample G175 (Battambang soil). Secondary Electron Image showing loose aggregate of fine grains. The grains themselves are about 5 μm in diameter and include quartz (e.g. bottom centre), Ti-bearing magnetite (e.g. cubic grain to upper right of aggregate), and possible kaolinite plates (e.g. bright grain to upper right). The coating which appears to bind them is a poorly crystalline aluminosilicate.

It was impossible to obtain a pure analysis of the surface material, but where grains were well covered, Al and Si were detected, suggesting that this could be a poorly crystalline clay such as kaolinite. Back-Scattered Imaging was used to show the Fe-rich grains beneath this aluminosilicate surface. In Plates 4 to 7, two aggregates of fine particles are imaged by both Secondary Electron and Back-Scattered imaging. The former shows the surface topography which is often poorly crystalline or platy, while the latter clearly shows crystalline, octahedral or cubic, Fe-rich grains beneath the surface (with bright intensity). The Fe-rich octahedral grains can be as small as 2 μm .

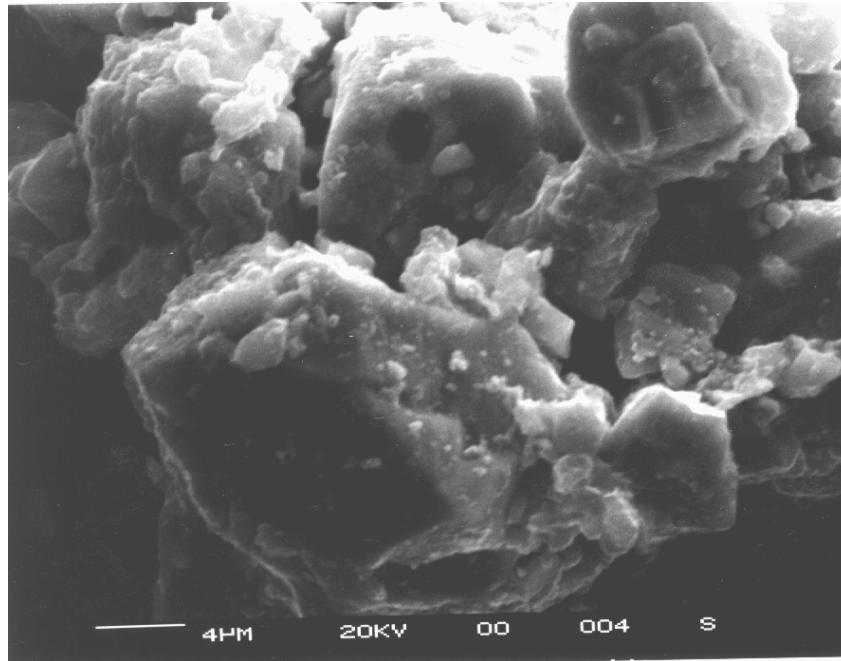


Plate 4. Sample 7 (Battambang soil). Secondary Electron Image showing loose aggregate of fine grains. This image shows the surface of grains to be coated in platy to poorly crystalline aluminosilicate material.

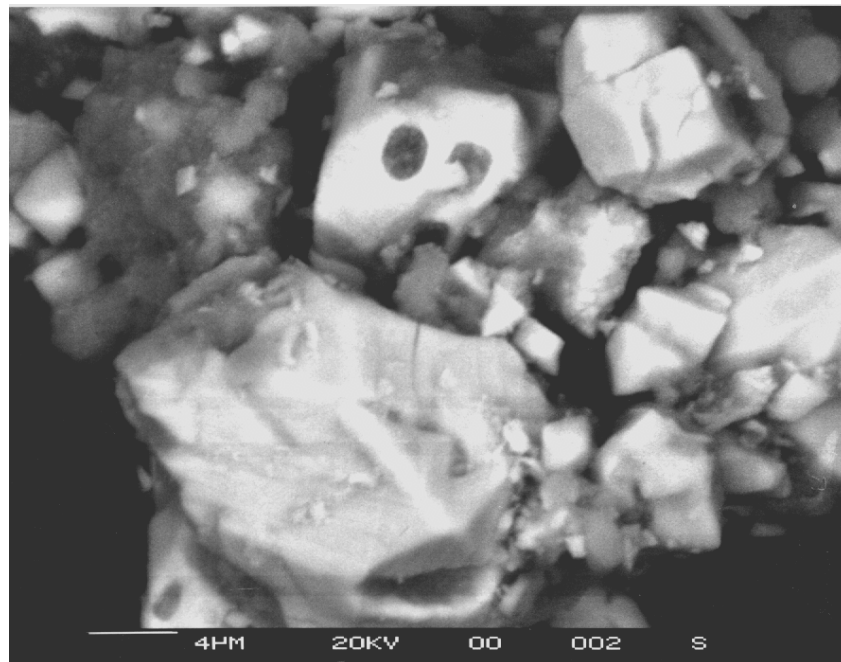


Plate 5 Sample 7 (Battambang soil). Back-Scattered Electron Image of the same region shown in Plate 4. This shows coarse grains beneath the surface coating. Many grains are octahedral with bright intensities (due to Fe content) and are probably magnetite.

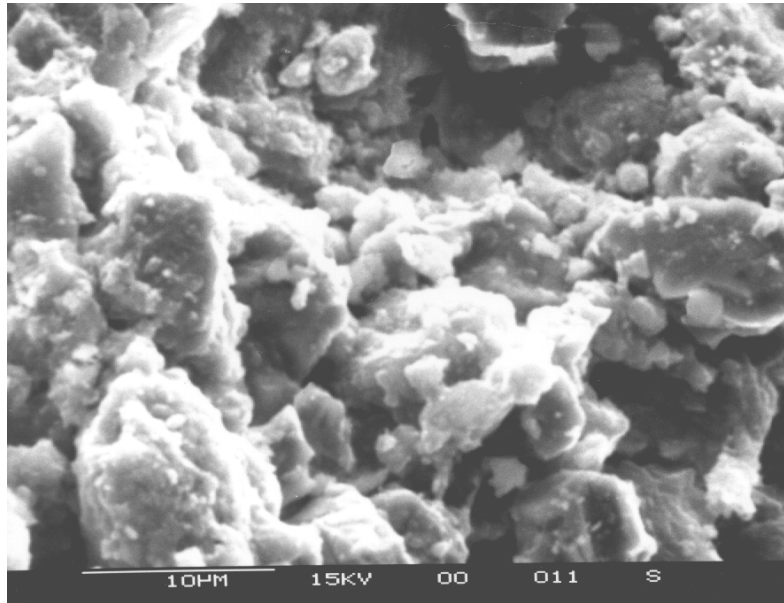


Plate 6. Sample 7 (Battambang soil). Secondary Electron Image showing loose aggregate of fine grains. This image shows the surface of grains to be thickly coated in platy to poorly crystalline ?aluminosilicate material.

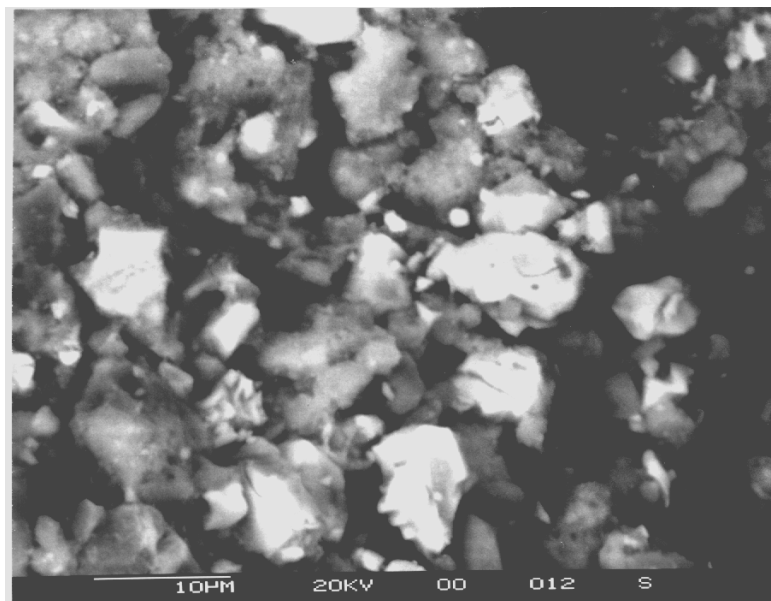


Plate 7 Sample 7 (Battambang soil). Back-Scattered Electron Image of the same region shown in Plate 6, showing coarse grains beneath the surface coating. The bright, angular grains are Fe-rich with variable Ti and are probably magnetite

The fine particles which had not aggregated together were also examined. These consist of aluminosilicate (kaolinite?) and Fe oxides with minor Ti, occurring either as octahedral grains (presumably magnetite) and sub-hexagonal plates with rounded edges (possibly haematite, Fe_2O_3). The different morphologies of these two iron oxides are due to their different crystallographic structures: magnetite has a cubic structure, whereas hematite has trigonal symmetry and thus can form hexagonal plates.

Devon 'Posbury Clump' subsurface soil – sample 10 (G178)

Plate 8 shows the grain size distribution of this soil sample. In comparison with the Cambodian sample, it appears to contain a much greater proportion of grains greater than 50 μm in size. These are of quartz and K-feldspar. The 10 to 50 μm fraction consists of quartz, K-feldspar and some biotite mica flakes, which can be up to 30 μm in size. The finer (<10 μm) fraction (Plate 9) consists mainly of biotite mica flakes, with lesser amounts of quartz and K feldspar and occasional sodic plagioclase and Ti oxide.

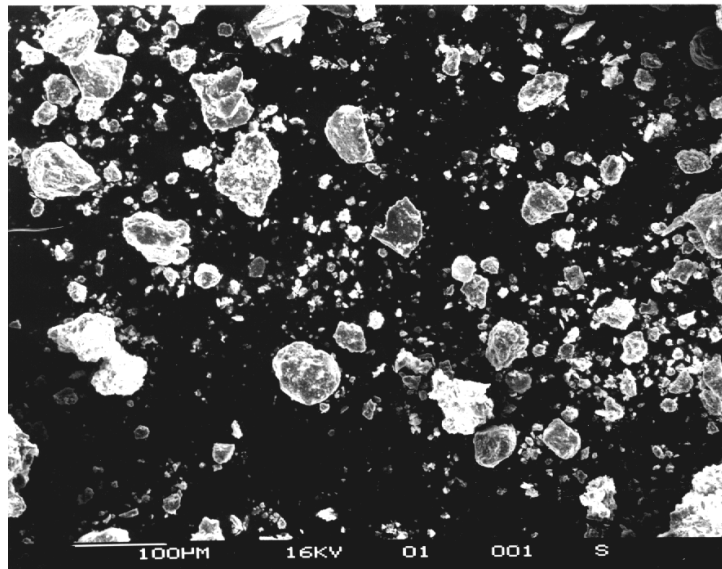


Plate 8. Sample 10 (Devon soil). Secondary Electron Image showing typical grain size distribution. The large, well-rounded grains (e.g. lower left of centre) are of quartz while large, more angular grains are K feldspar. Biotite mica forms plates or flakes up to 30 μm in diameter.

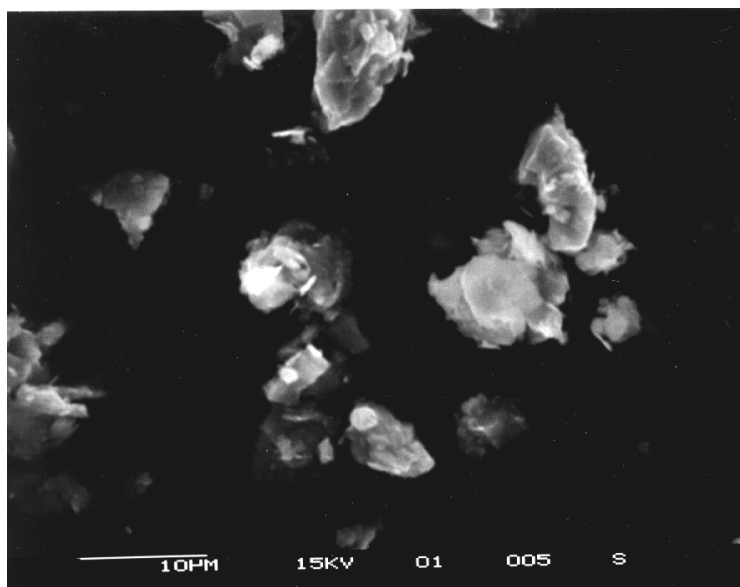


Plate 9. Sample 10 (Devon soil). Secondary Electron Image of some smaller (<10 μm) grains. These are mostly platy biotite mica.

Devon soil sample 10 with magnetite artificially added

This artificial mixture was briefly examined to see how the size distribution of the magnetite in it compared with the natural magnetite grain sizes in the Cambodian soil. Plate 10 shows that the former is dominated by relatively coarse-grained angular fragments of magnetite, up to about 50 μm in diameter. The magnetite in this sample is generally in the form of much larger grains than in the Cambodian soil, and is not coated in poorly crystalline clay. It is not, however, visibly more or less crystalline than in the Cambodian soil.

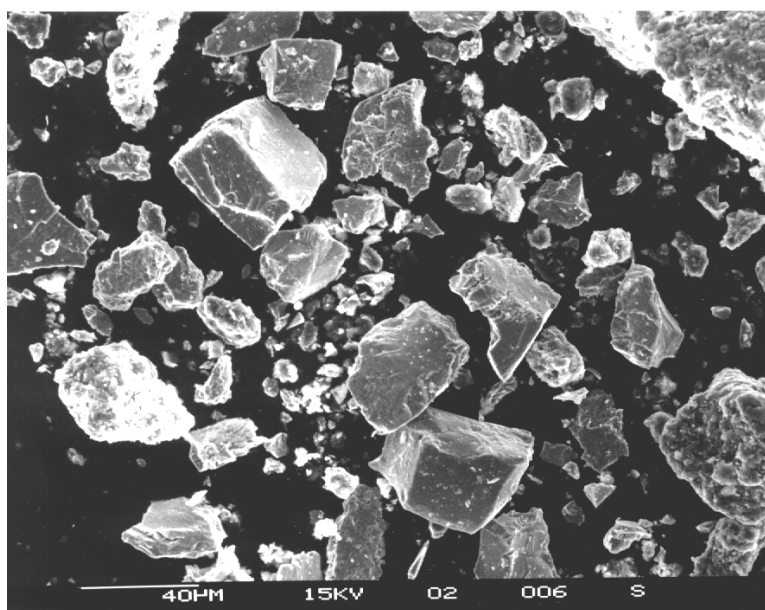


Plate 10. Sample 10 (Devon soil) mixed artificially with 'pure' magnetite. Secondary Electron Image showing grain size distribution. The darker, angular to cubic grains are the artificially added magnetite. The very bright grains with rough surfaces (upper left and mid-left) are from the Devon soil and are probably coated with fine grained hydrous material (e.g. phyllosilicate), causing them to 'charge up' under the electron beam.

6.1.4 Discussion and conclusions from sem study

The Cambodian soil sample 7 (G175) consists mostly of quartz and Fe oxide, mostly as magnetite (which contains minor to substantial Ti) with a little hematite. A poorly crystalline clay (possibly kaolinite) coats grain surfaces and may have acted to bind smaller grains together into aggregate particles or peds. The magnetite appears well-crystallised and is present throughout the grain size range but can be very fine grained (as small as 2 μm in diameter). BSE imaging shows iron to be located within discrete crystalline grains rather than in the poorly crystalline clay.

In comparison, the Devon soil sample 10 (G178) consists mostly of coarse-grained quartz and K feldspar, with lesser amounts of biotite mica. The artificial mixture of sample 10 with a 'pure' magnetite source differs from the Cambodian soil in that the magnetite is present as larger grains than in the Cambodian soil, and is not coated in, or aggregated together by, poorly crystalline clay.

7. X-RAY DIFFRACTION STUDIES

7.1 Laboratory methods

7.1.1 General

A representative portion of each sample was removed, dried at 55°C and hand-crushed to pass a 5 mm sieve. A subsample of this crushed material was then hammer-milled to <0.12 mm in the BGS Sample Preparation Facility. For whole-rock X-ray diffraction (XRD) analysis, an approximately 3 g subsample of the powdered material was micronised under acetone for 10 minutes to ensure a fine and uniform particle-size.

7.1.2 Clay fraction separation, oriented and random mount preparation

For each sample, approximately 10 g of crushed material was placed in a 500 ml bottle with approximately 200 ml deionised water, shaken on a laboratory shaker overnight, and treated with ultrasound for 2 minutes before wet sieving on 63 µm. The >63 µm ('sand' fraction) material was then dried at 55°C and bagged. The <63 µm suspended material was placed in a 250 ml measuring cylinder with 1 ml 0.1M sodium hexametaphosphate ('Calgon') solution to disperse the individual clay particles and prevent flocculation.

After standing for a period determined from Stokes' Law, a nominal <2 µm ('clay') fraction was removed, dried at 55°C and bagged. The remaining 2-63 µm ('silt' fraction) material was also dried at 55°C and bagged.

100 mg of the dried <2 µm material was re-suspended in a minimum of deionised water and pipetted onto a ceramic tile in a vacuum apparatus to produce an oriented mount. The mounts were then Ca saturated using 2 ml 0.1M CaCl₂.6H₂O solution and washed twice to remove excess reagent and allowed to dry at room temperature.

Dry, micronised powders were back-loaded into standard aluminium sample holders to produce whole-rock random powder mounts.

7.2 X-ray diffraction analysis

XRD analysis was carried out using a Philips PW1700 series diffractometer equipped with a cobalt-target tube and operating at 45kV and 40mA. Whole-rock powders were scanned from 3-50°2θ at 0.7°2θ/minute. The <2 µm samples were scanned from 2-32°2θ at 0.55°2θ/minute as air-dry mounts, after glycol-solvation and after heating

to 550°C for 2 hours. Diffraction data were analysed using Philips X'Pert software coupled to an International Centre for Diffraction Data (ICDD) database running on a Gateway PC system.

Following identification of the mineral species present in the samples, whole-rock mineral quantification was achieved using the Reitveld refinement technique using Siroquant v.2.5 software. This method avoids the need to produce synthetic mixtures and involves the least squares fitting of measured to calculated XRD profiles using a crystal structure databank. Errors for the quoted mineral concentrations are probably $\pm 5\%$ accuracy for quartz, magnetite, hematite, rutile and dolomite. Larger errors are possible for the quoted kaolinite, 'mica', chlorite, and feldspar concentrations.

A method proposed by Churchman *et al.* (1984) based on the different rate and extent of formamide intercalation was used to distinguish between the kaolin group minerals halloysite and kaolinite. Concentrated $< 2 \mu\text{m}$ oriented mounts were scanned three times from $7.5\text{--}16.5^\circ 2\theta$ at a speed of $0.55^\circ 2\theta/\text{minute}$: (i) wet – having allowed excess water to drain away, (ii) as air-dry mounts, after glycol-solvation and after heating to 550°C for 2 hours.

7.3 Clay mineral XRD-profile modelling

In order to assess the relative proportions of any clay minerals present in the samples, modelling of their XRD profiles was carried out using Newmod-for-Windows™ (Reynolds & Reynolds, 1996) software. The modelling process requires the input of diffractometer, scan parameters and a quartz intensity factor (instrumental conditions) and the selection of different clay mineral sheet compositions and chemistries. In addition, an estimate of the crystallite size distribution of the species may be determined by comparing peak profiles of calculated diffraction profiles with experimental data. By modelling the individual clay mineral species in this way, *mineral reference intensities* were established and used for quantitative standardization following the method outlined in Moore and Reynolds (1997).

7.4 XRD study results

The results of whole-rock XRD analysis for the samples are summarised in Table 5. Chi-squared values of < 3 were obtained for both the samples using the Siroquant software, values of < 5 representing good quality data. The results of $< 2 \mu\text{m}$ XRD analyses are summarised in Table 2.

Table 5. Summary of quantitative, whole-rock XRD analysis

Sample	%mineral				
	kaolinite	quartz	magnetite	hematite	boehmite
Cambodian soil, sample 7	47	31	15	6	<1

Sample	%mineral								
	quartz	kaolinite	'mica'	K-feldspar	albite	hematite	rutile	chlorite	dolomite
Devonian mudstone, sample 10	51	18	14	7	5	3	1	<1	<1

Table 6. Summary of quantitative, <2 µm XRD analysis

Sample	%clay mineral		
	kaolinite	illite	chlorite
Cambodian soil, sample 7	100	nd	nd
Devonian mudstone, sample 10	21	75	4

The Cambodian soil (7) is predominantly composed of kaolinite ($\text{Al}_4\text{Si}_4\text{O}_{10}(\text{OH})_8$) and quartz (SiO_2) with minor magnetite (Fe_3O_4) and hematite (Fe_2O_3) and a trace of boehmite ($\gamma\text{-AlOOH}$). In Figure 1, the Cambodian soil whole-rock diffraction trace is shown labelled with its mineral component diagnostic peak positions. Routine <2 µm XRD analyses (Figure 2) confirm that kaolinite is the only clay mineral present in the Cambodian soil (7). The broad nature of the kaolinite peaks suggests that it is composed of small crystallites and could be termed a 'b-axis disordered' species. The invariant position of the c.7Å XRD peak after formamide intercalation indicates a lack of any halloysite in the soil (Figure 3).

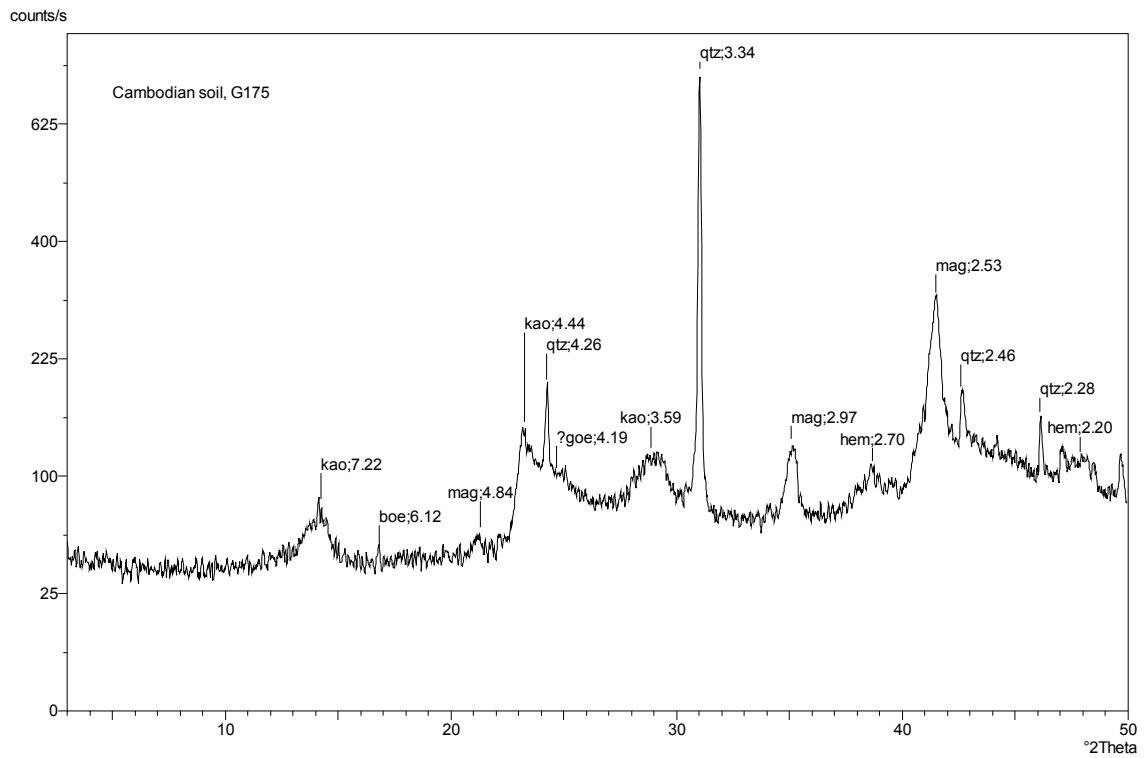


Figure 1. Whole-rock X-ray diffraction trace, Cambodian soil sample 7.

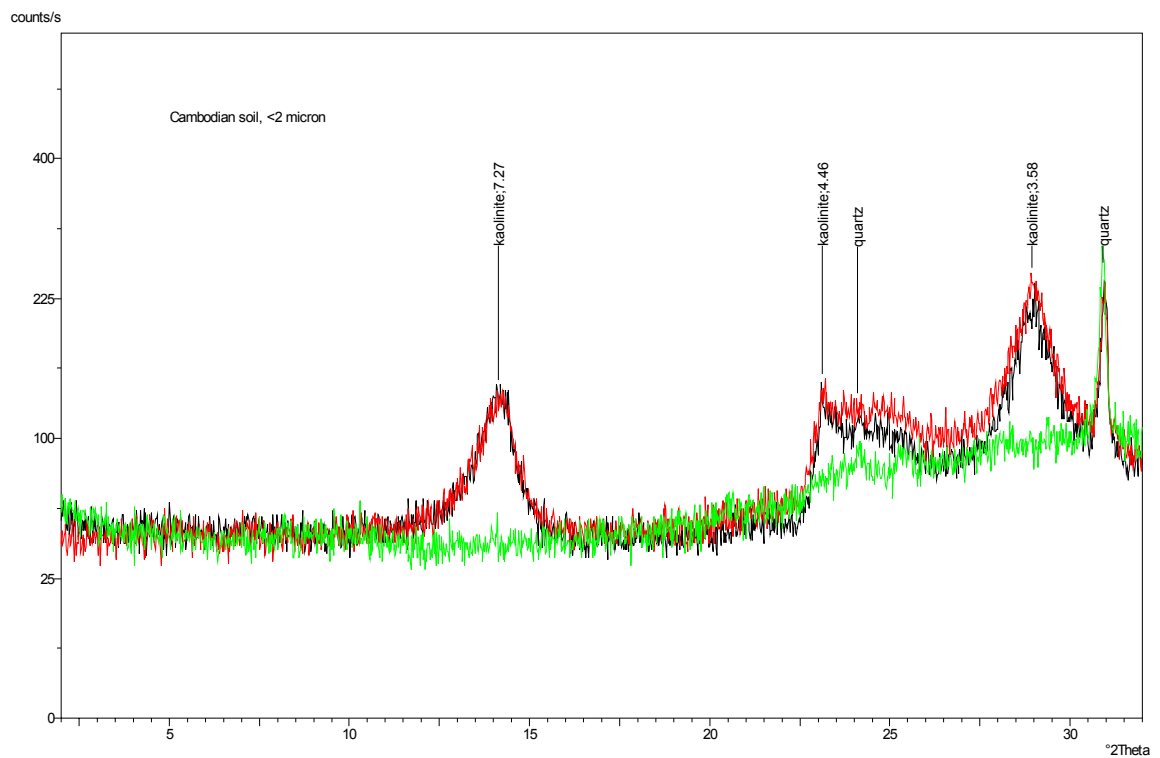


Figure 2. <2 μm X-ray diffraction traces illustrating the effects of routine diagnostic testing, Cambodian soil sample 7. (Black trace = air-dry, red trace = ethylene glycol-solvated, green trace = heated at 550°C/2 hours)

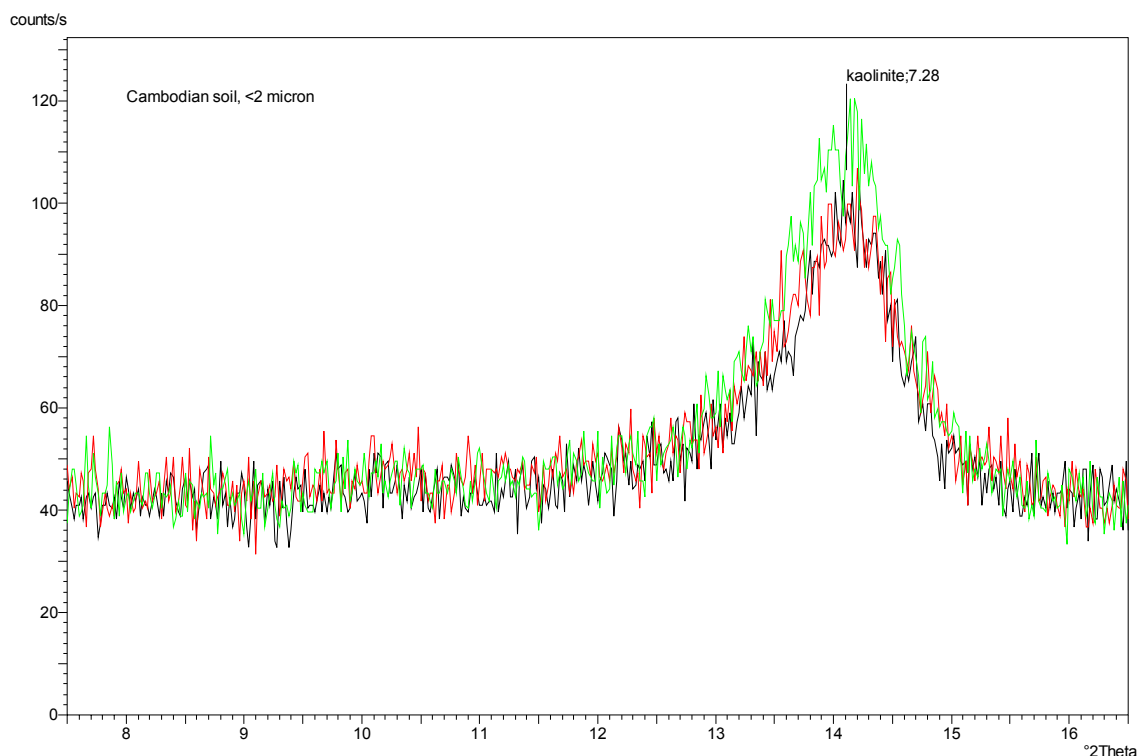


Figure 3. <math><2\ \mu\text{m}</math> X-ray diffraction traces illustrating the effects of formamide intercalation, Cambodian soil sample 7. (Black trace = wet, red trace = formamide intercalation, green trace = heated at 110°C/90 minutes)

However, the Devonian mudstone (10, Figure 4) is predominantly composed of quartz with minor kaolinite, undifferentiated mica species including illite ('mica', mostly K-aluminosilicate), feldspar (K-feldspar and albite, K- and Na-aluminosilicate), hematite and rutile (TiO_2) with traces of chlorite (Fe,Mg-aluminosilicate) and dolomite ($\text{CaMg}(\text{CO}_3)_2$). $<2\ \mu\text{m}$ XRD analyses (Figure 5) detected the presence of illite, kaolinite and minor chlorite. In contrast to the Cambodian soil, the kaolinite in the Devonian mudstone presents relatively sharp XRD peaks suggesting that it is composed of relatively large crystallites. The high degree of crystallinity exhibited by the kaolinite in the Devonian mudstone suggests it is unlikely to contain any halloysite rendering the formamide-intercalation test superfluous. Since halloysite may irreversibly collapse at even room temperatures, the previous air-drying of both samples means that halloysite was unlikely to be detected.

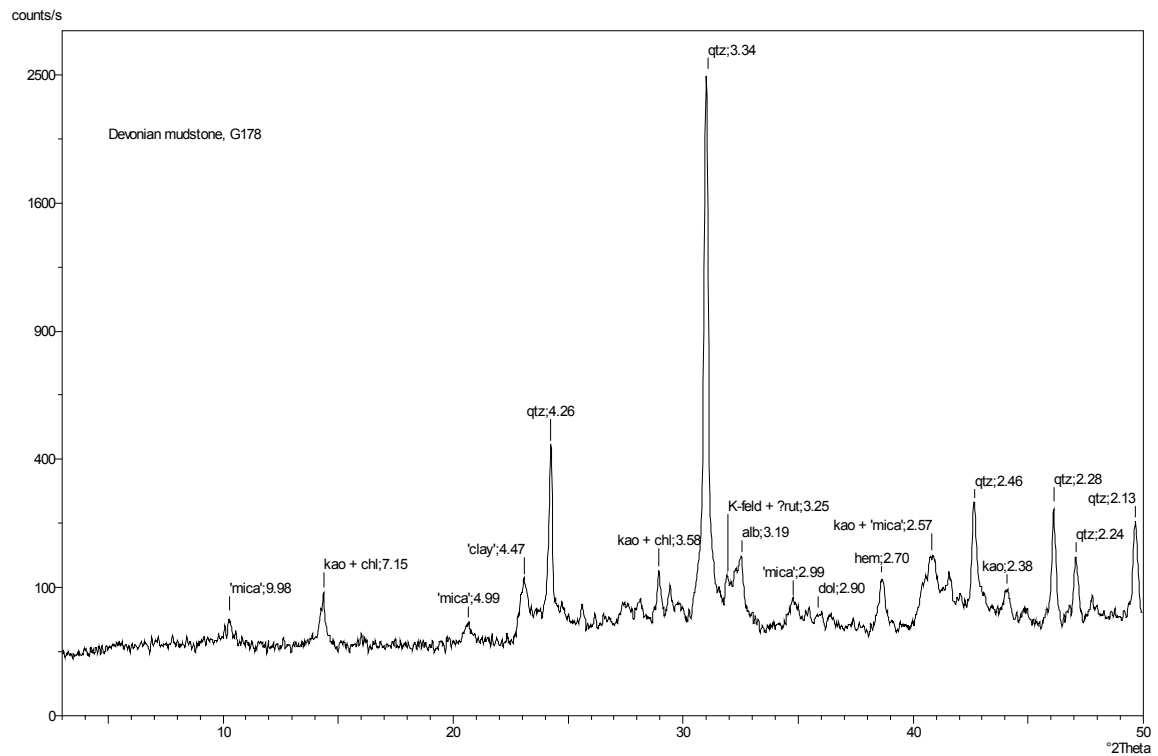


Figure 4. Whole-rock X-ray diffraction trace, Devonian mudstone sample 10.

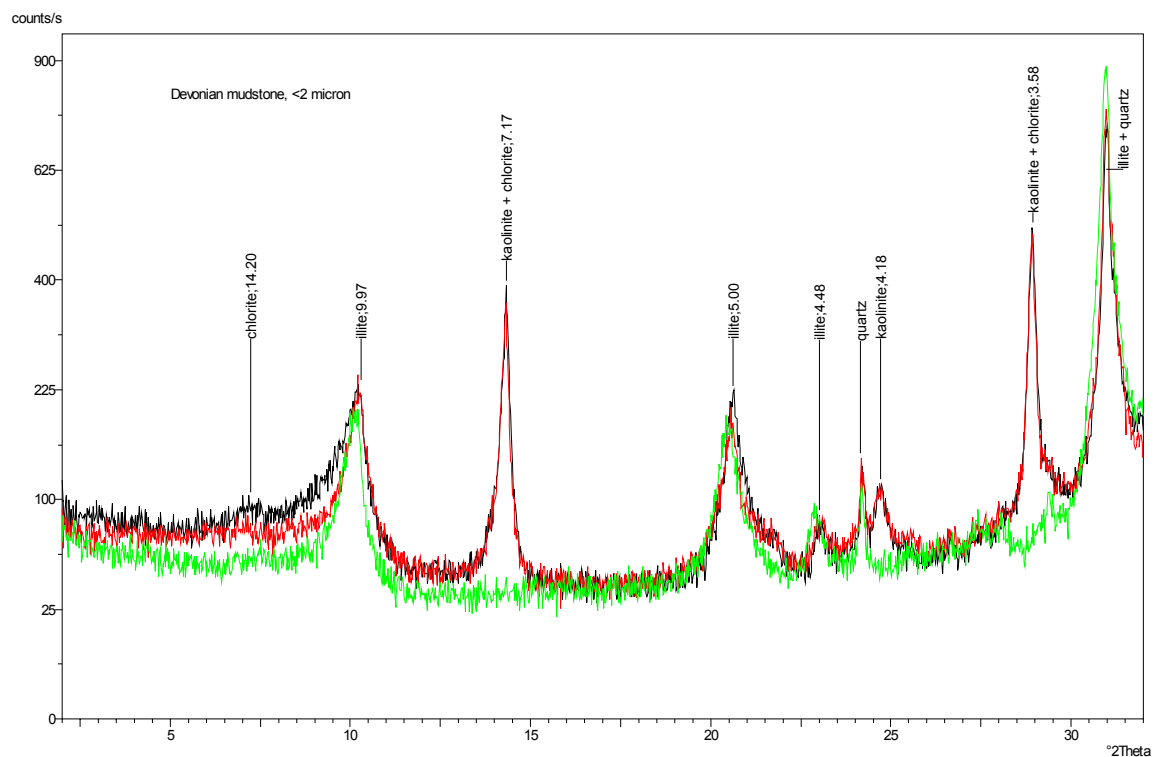


Figure 5. <2 μm X-ray diffraction traces illustrating the effects of routine diagnostic testing, Devonian mudstone sample 10. (Black trace = air-dry, red trace = ethylene glycol-solvated, green trace = heated at 550°C/2 hours).

XRD analysis of the coarse crystalline magnetite concentrate indicate that it has similar peak shapes to magnetite found in the Cambodian soil. The previous report suggested that the magnetite in the Cambodian soil might be poorly crystalline causing the broadness of the peaks. The magnetite peaks are relatively broad compared to many other minerals but this test shows that it is an inherent feature of magnetite not a result of poor crystallinity

8. RESISTIVITY – MOISTURE CONTENT MEASUREMENTS

Resistivity – moisture content measurements were made on a Cambodian soil sample 7 (G175) and a simulated magnetic tropical soil made from sample 10 (G178) mixed with fine grained, powdered magnetite.

Resistivities were determined for a range of moisture contents for the two ‘soils’ using a galvanic cell, as described below. As shown in Figure 6 and Plate 11, this cell passes a current between two current electrodes C1 and C2 and measures a potential difference between electrodes P1 and P2 connected to geophysical four-terminal resistance measuring instrumentation (ABEM Terrameter). These measurements are presented as resistances as the result of the passage of a bi-polar constant current through the cell alternating at a frequency of 1 Hz. The two materials were calibrated against a cell of de-aired tap water used as the pore fluid (Plate 12).

Water was removed from the ‘soil’ samples using a paper-wick method being developed at BGS, using capillary forces (i.e. suction) to draw water out of the sediment sample (Plates 13 & 14). This method maintains the ionic balance of the pore fluid, essential to these studies, not possible using evaporative methods. There is, however, a lower limit to the amount of water that can be removed using this method, which may be above the natural moisture content of the materials.

The resistances were converted into resistivity measurements and formation factors for both samples and plotted against the corresponding moisture contents. Formation Factors, being the sample resistance divided by the corresponding values measured on the control water sample, are insensitive to temperature variations. The graph shown in Figure 7 clearly shows that there are major differences between the two materials. These are not only the obvious numerical differences in moisture content for similar formation factors and resistivities, but also that there appears to be a difference in the trend line formed during the experiment.

In the case of the synthetic sample there was a significant degree of shrinkage during the test. This amounted to a vertical change of 6.3 mm over the time of the experiment. There was also a lateral movement of about 1mm away from the sides of the cell. The change was less significant between stage 5 and stage 7 as can be seen from the graph. This indicates that the majority of the change took place in the earlier

stages, which might account for the greater scatter of the points on the graph at these times.

The received sample from Cambodia, sample 7, was hard and granular (granules composed of clay) and had an initial moisture content of 22% (i.e. each granule). On addition of water, the grains absorbed water and softened slightly. At the start of the test the water and sample filled the cell. At the end of the test this sample did not change volume and the particles remained granular. The decrease in moisture content appeared regular throughout the test indicating no large change in suction. Most of the water removed could be considered as free water (in between the clay granules). As the volume of the sample remained the same the reduction of water produced a decrease in saturation and a corresponding increase in air in the inter-granular space.

The artificially mixed sample, G178, comprised hard, very fine gravel sized and finer granules composed of silt and clay, having a moisture content of 1%. On addition of water the granules absorbed water, swelled and softened. At the start of the test the sample filled the cell but as water was removed the volume reduced. At the end of the test this material had changed to a soft to firm, uniform clay with no obvious granules. Most of the water was removed during the first few drying stages. Water removal resulted in shrinkage. The efficiency of water removal reduced as the test progressed, the corresponding reduction in volume indicating suction pressures increased within the sample as the moisture content reduced. It is likely that the sample remained saturated throughout the test.

The behaviour and resistivity response of these two samples was different. Although the resistivities of the two samples are generally similar their water content is extremely different.

The granules (made of clay) of Cambodian sample 7, soaked up little water and behaved as a granular material, whereas the artificial sample, absorbed water readily to form a uniform clay. These differences are due to the structure and mineralogy of the samples. The Cambodian sample is a residual soil formed in situ from the weathering of the parent rock whereas the artificial sample was made from a sedimentary mudstone.

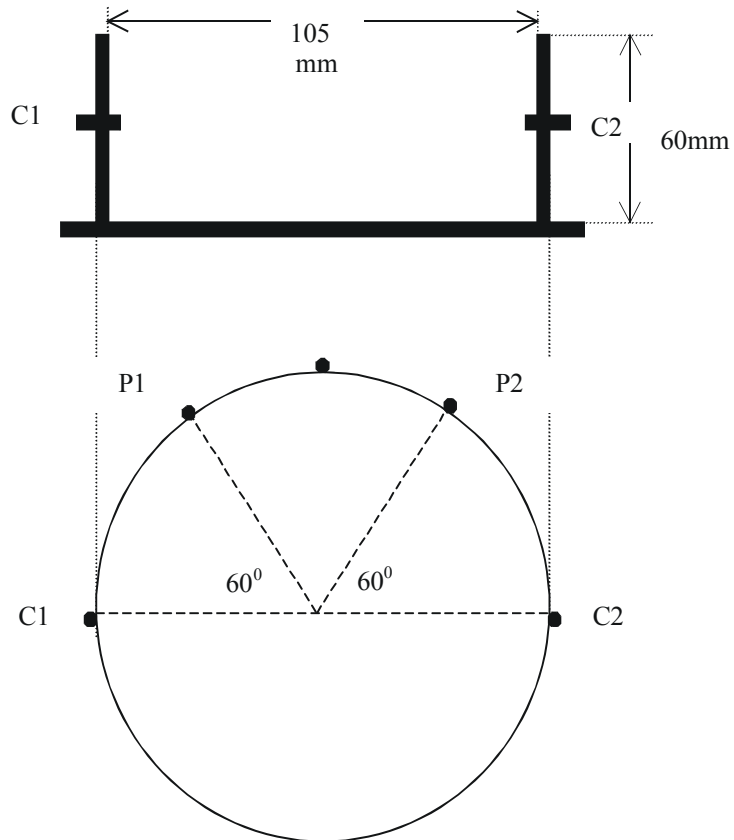


Figure 6. Schematic of resistivity cell.

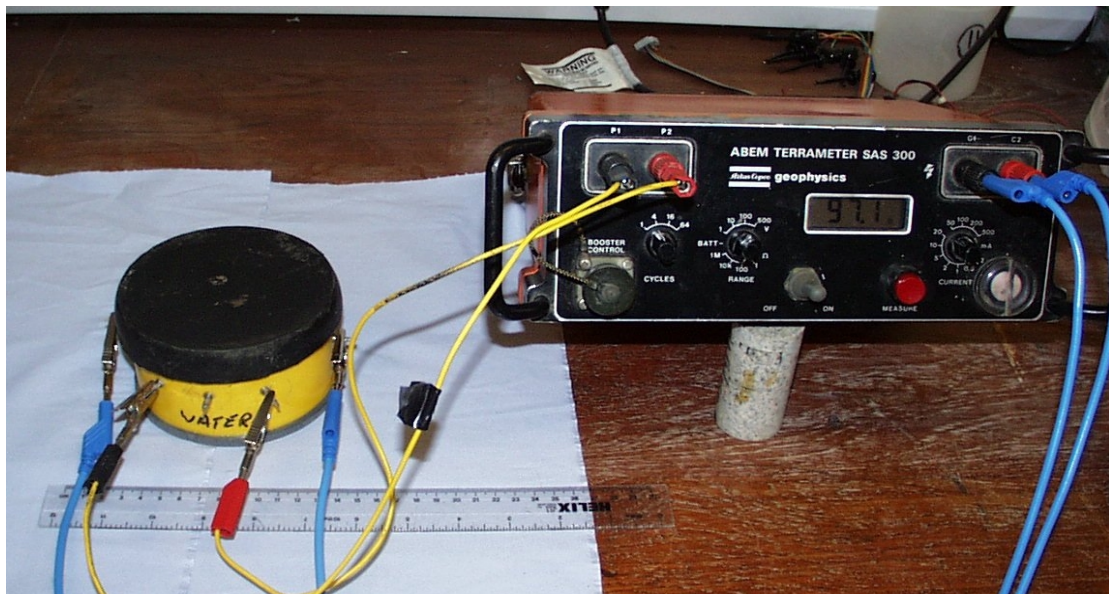


Plate 11. Resistivity cell containing water (control), connected for four-terminal a.c. resistance measurement (1Hz)



Plate 12. The beaker containing the sediment is filled with de-aired water by allowing it to overflow the top of the container. This should be done slowly to prevent material being lost. When the opaque patches disappear, the container can be removed from the vacuum apparatus.



Plate 13. Square apertures cut into lids allow moisture to be removed using paper wick



Plate 14. Resistivity cell with paper wick in place, sealed with tape to prevent evaporation, preventing excessive loss of salts from pore fluid.

DERA

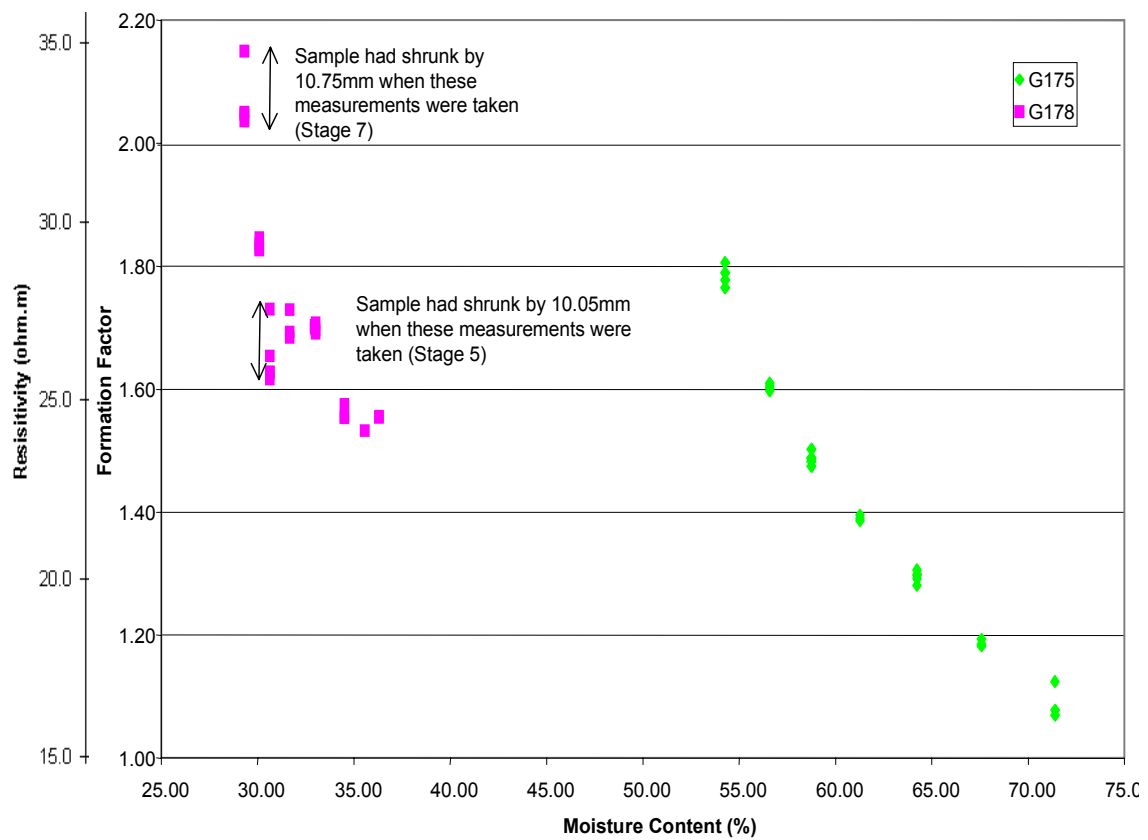


Figure 5. Resistivity (& Formation factor) – Moisture content relationship for reconstituted Mercia Mudstone with magnetite (G178) and Cambodian soil (G175).

9. ANCILLARY INFORMATION

1. Information on the purchase of kappameters and conductivity meters has already been sent to DERA
BGS purchase their equipment from :
Earth Science Systems Ltd, Unit 1 Kimpton Enterprise Park, Claggy Road, Kimpton, Herts, UK SG4 8HP
Tel 01438 833611
Fax 01438 833541
E-mail enquiries@earthsciencesystems.co.uk
WWW <http://www.earthsciencesystems.co.uk>
2. Information on soil import has already been sent to DERA. BGS cannot help with the import of large quantities. DERA will have to contact the Soil Licensing Office at MAFF to discuss your particular needs with them and they should tell you what the possibilities are
3. The previous BGS reports on tropical red clay soils run to over 1000 pages. The titles of the reports are listed below. A quick scan through suggests they do not contain anything that has a very direct bearing on the problem currently being investigated. The reports concentrate mostly on the engineering properties rather than electrical/magnetic properties that are of most relevance to this problem. The introductory sections of some of the reports give good reviews of the formation of tropical soils and their classification that might be useful background. Sections on moisture content measurements and field sample collection might be useful if more soil testing is required. A mineralogical report describes features that are relevant to the formation of peds that possibly affect the moisture content properties. Copies of the relevant sections of these reports can be supplied if requested. The relevant BGS reports are listed under 'References', at the end of this report.

10. DISCUSSION

The kappameter tests show that apart from the dark brown soils from Cambodia, most soils have low magnetic susceptibilities. High magnetic susceptibility always causes a high mine detector response, but some samples with low magnetic susceptibility also cause a strong mine detector response. There is clearly a factor other than fine-grained magnetite causing a response in many samples. Previous studies of samples from Devon showed that this can be caused by small pebbles of weathered basaltic volcanic rock.

The SEM studies confirmed the presence of numerous very small crystal of magnetite, along with abundant clay and fine quartz in the Cambodian soil. The magnetite is well crystallised but only a few microns in size. A poorly-crystalline kaolinitic clay is abundant and tends to bind the small grains together to form larger aggregates, often referred to as peds. The Devon soil contains a much wider range of mineral types but very little natural magnetite and the ped structures are absent. The magnetite in the synthetic soil is coarser than that in the Cambodian soil. The magnetite concentrate could be crushed to a finer grain size to more closely simulate the natural situation.

XRD analysis confirms the presence of abundant magnetite in the Cambodian soil. A particular difference between the Cambodian and Devon soil is the type of clay mineral; in the Cambodian soil it is a poorly ordered kaolinite, while in Devon soil it is illite. Soil rich in poorly ordered kaolinite is typical for a mature tropical soil formed by intense weathering and leaching but is rare in temperate climates such as the UK. In the UK illite-smectite rich clays are normal. Small amounts of kaolinite-rich soil may be present in a few places in the UK, e.g. around the china clay (kaolin) areas in SW England but these are well ordered kaolin and will not have the same properties as the tropical soil. The nature of the clay minerals controls the formation of peds which in turn controls the moisture content properties of the soil.

It is interesting to note that the Cambodian 'Battambang' soil contains significantly more quartz in comparison to the soils previously studied by BGS developed over volcanic rocks from Kenya and Indonesia. The presence of c. 30% quartz in the Cambodian sample would almost certainly mean that it is not an andosol soil containing amorphous allophane clay minerals, despite being similar in texture and appearance to this soil type. Knowledge of the location and geological provenance of this Cambodian soil would help to give an indication of its pedological soil type, and hence a further general indicator as to its likely *in situ* mineral composition.

The moisture and resistivity properties of the Cambodian and UK soils are very different. This is largely due to the structure of the Cambodian soil that disaggregates to form small particles or peds. When mixed with water the Devon soil forms a 'homogenous', soft, wet lump of mud. In contrast the peds in the Cambodian soil do not absorb much water, they act like small sand grains and the sample is more like wet sand rather than a lump of wet mud. This is illustrated in Plate 15. This means the Cambodian soil can absorb a much greater amount of intergranular water which gives a much lower resistivity.

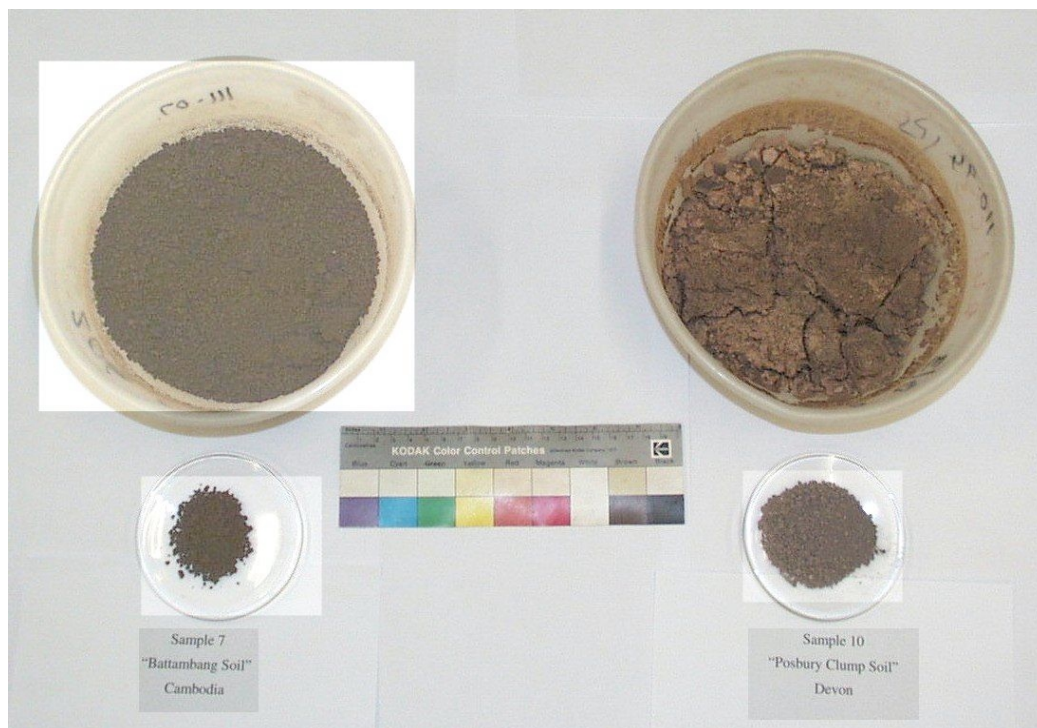


Plate 15. Photographs of soil samples after testing showing the granular nature of the Cambodian soil.

It is important to bear in mind that many tropical clays undergo irreversible changes when subjected to drying after sampling. These changes involve alteration of the clay mineral structures and soil fabric that, in turn, influence the soil density, porosity, moisture-holding and electrical resistivity properties. This is a critical concern as tests undertaken on a tropical soil that has been allowed to dry out may bear little or no relation to the material properties (including electrical properties) of the same soil *in situ*. As the mineralogical changes subject to drying are irreversible, re-wetting of the dried material in the laboratory does not return the soil to its *in situ* pre-dried state. **To get good representative results from most of the tests, soil sampling techniques have to be used that retain the soil as near as possible to its natural moisture condition.**

The mine detector response caused by the analogue soil, prepared by mixing powdered magnetite with Devon soil, was similar to that caused by the Cambodian soil.

11. THE WAY FORWARD

It is highly unlikely that a natural soil will be found in the UK (or, indeed, in any country in a temperate climatic zone) that will have the same compositional, moisture, magnetic and resistivity properties of the Cambodian soil *in situ*. It seems likely that the magnetic properties can be simulated by the addition of magnetite but moisture content-resistivity-conductivity relationships/properties are more complex and difficult to simulate. However, it may be feasible to prepare an artificial soil that goes some way to replicating these properties following further investigations. The critical first step would be to collect representative samples of the soils, from area(s) of interest in Cambodia. It is critical that these samples are collected and sealed immediately on sampling to retain their *in situ* moisture condition, and that this condition is maintained during transport to a laboratory. Locational details of the sampling location(s) should also be recorded, including a brief description of the sampling site (e.g. natural slope, jungle, cultivated field, road, track, plantation, etc.) and elevation details. Geological and pedological (soil map) information would also be extremely useful. Much of this latter information, if available, could almost certainly be obtained from archives in the UK. Subsequent laboratory analysis of the Cambodian soil samples from their *in situ* moisture condition would enable more meaningful compositional and mineralogical details to be obtained. Based on this data, and in conjunction with the background details of the sample sites, more informed consideration could then be given to the requirements of locating a similar soil or, more likely, preparing an artificial analogue soil in the UK to replicate the magnetic and electrical characteristics of the soils encountered in Cambodia.

12. REFERENCES

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DUCHAUFOR, P. 1982. *Pedology, Pedogenesis and Classification*. (English Edition) Trans. T R Paton. London: George Allen & Unwin.

FOOD AND AGRICULTURAL ORGANISATION OF THE UNITED NATIONS. 1969. *Definitions of soil units for the soil map of the world. 1: 5 000 000. Soil map of the world*. FAO-UNESCO Project, F.A.O., Rome.

_____ 1988. *Soil map of the world, revised legend, 1:5 000 000*. F.A.O., Rome

_____ *Map Sheet IX: Southeast Asia*; 1976

Listing of relevant BGS project reports:

Main reports:

BGS Technical Report No. WN/93/11 - *Engineering Geology of tropical red clay soils: Project background, study areas and sampling sites.*

BGS Technical Report No. WN/93/12 - *Engineering Geology of tropical red clay soils: Geotechnical characterisation: index properties and testing procedures.*

BGS Technical Report No. WN/93/13 - *Engineering Geology of tropical red clay soils: Geotechnical characterisation: mechanical properties and testing procedures.*

BGS Technical Report No. WN/93/14 - *Engineering Geology of tropical red clay soils: Sampling methods.*

BGS Technical Report No. WN/93/15 - *Engineering Geology of tropical red clay soils: Investigation and classification for engineering purposes.*

Relevant supplementary reports:

BGS Technical Report WG/88/2R - *Mineralogy of kaolinitic soils from Kenya and Indonesia*

BGS Technical Report WG/89/14R - *Mineralogy of further kaolinitic soils from Kenya and Indonesia*

BGS Technical Report WG/91/7R - *Mineralogical analysis of tropical red clay soils.*

BGS Technical Report WG/92/31R - *Petrographical examination of some tropical red soils from Kenya and Indonesia*

BGS Technical Report WG/95/2R - *Mineralogical and petrographical analysis of some tropical red soils from Kenya and Fiji.*

13. ACKNOWLEDGEMENTS

We would like to thank Kilton Industries for donating the sample of magnetite concentrate used in these trials.