Clay Mineralogy & Geophysics for Environmental Management & Mineral Exploration - ClayGEMME 2004 -

September 27-October 1, 2004 Adelaide, South Australia, Australia

Book of Abstracts and Program for Conference Symposium

CSIRO Land and Water, Seminar Room, Waite Campus, Waite Road, Urrbrae, South Australia



Australian Clay Minerals Society (ACMS) (19th Biennial Conference)

In association with:

Soil Mineralogy Commission of the International Union of Soil Sciences (IUSS)





The Australian Society of Exploration Geophysics (ASEG)

Australian Society of Soil Science, Incorporated (ASSSInc).









Australian Clay Minerals Society (ACMS) website at: <u>HTTP://WWW.CLAYS.ORG.AU</u>

FOREWORD

Conference and Workshop on "Clay Mineralogy and Geophysics for Environmental Management & Mineral Exploration" (ClayGEMME 2004)

27th September to 1st October, 2004, CSIRO Land and Water, South Australia.

ClayGEMME 2004 brought together over 60 researchers, industry professionals and students with interests in clay mineralogy, geophysics, soil science, environmental science and mineral exploration. It was organised by the Australian Clay Minerals Society (19th Biennial Conference) and CRC for Landscape, Environments and Mineral Exploration (CRC LEME) - in association with the "Soil Mineralogy Commission" of the International Union of Soil Sciences (IUSS - as a mid-conference meeting); The Australian Society of Exploration Geophysics (ASEG) and the Australian Society of Soil Science, Incorporated (ASSSInc - South Australian Branch).

Days 1 and 2 involved the Workshop component, aptly sub-titled: "*Turning theory into practice and toys into tools*". Day 1 involved technical presentations by leaders in their fields, covering topics such as pedology and clay mineralogy, remote sensing and gamma ray radiometrics, and petrophysics (electromagnetics, magnetics and ground penetrating radar).

On Day 2, a field practical was conducted in the Herrmann's catchment, Mt Lofty Ranges, which included a Pb-Zn mineralised zone, degraded wetlands (acid sulfate soils), soils (saline, sodic and eroded) and streams (polluted). Participants were shown how to link pedological, geophysical and mineralogical data to deduce sub-surface geochemical processes and understand how specific properties of clay minerals (layer silicates, oxyhydroxides, sulfides, etc) impact on results and interpretations. Hands-on training was conducted in the use of field-portable geophysical and mineralogical equipment instruments.



Photograph of delegates taken in the Herrmann's catchment during the field practical component of the workshop

Demonstration of ground penetrating radar (see photograph below), illustrated the occurrence of paleo-channels and soil morphological features. The focus of this multi-disciplinary workshop was unique because it was - for the first time anywhere in the world – where the catalyst was provided for links to be forged between experts in geophysics, clay mineralogy and pedology.



Photograph in the Herrmann's catchment showing demonstrations of geophysical equipment during the field practical component of the workshop

Day 3 (conference symposium) included presentations of papers at the following sessions: (1) Poorly Ordered Nanoparticulate Minerals (PONM) in Soils or Sediments; (2) Clay mineralogy and geophysics incorporating remote sensing, mineral exploration and environmental management; (3). New techniques; (4). Applications of clays in forensics, industry and technology. Discussions were relaxed and stimulating, and the debate rigorous.

The 4th day involved fieldtrips, featuring a visit to the Gillman tidal area, which is the focus of coastal acid sulfate soil process research (Brett Thomas' PhD). This also involved a joint discussion meeting with the project's steering committee (Barker Inlet and Port Estuary Committee). In the afternoon, the field the tour visited sites in the Mt Lofty Ranges, where Dr John Keeling (PIRSA and CRC-LEME) demonstrated links between clay mineralogy and commercial clay mineral exploitation at a selection of well characterised sites.

ClayGEMME 2004 drew on the unique expertise that is being developed and spearheaded in CRC LEME and CSIRO Land and Water under the joint guidance of Drs Rob Fitzpatrick (pedology and clay mineralogy) and Graham Heinson (geophysics) in the emerging field of "soil and forensic/mineral exploration geophysics". This workshop and meeting has now given us some very "good leads and ideas" for the two IUSS symposia in 2006.

Rob Fitzpatrick

Source: International Union of Soil Sciences (IUSS) Bulletin No. 105 2004; p. 47. (modified)

Citation of whole volume and abstracts are properly referenced as follows:

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- Keeling J. L., A. J. Mauger and M. D. Raven (2004). Hyperspectral and other techniques in the search for diamondiferous kimberlite near Pine creek, Terowie. p 6-7. *In* R.W. Fitzpatrick and G.S. Heinson (eds) Book of Abstracts of Conference Symposium on "Clay Mineralogy & Geophysics for Environmental Management & Mineral Exploration", Adelaide, 27th September to 1st October, 2004. Australian Clay Minerals Society 19th Biennial Conference: <u>http://www.clays.org.au</u>.

Conference Technical Program and Index to Abstracts

ClayGEMME 2004

Time / (Page No.)	Technical program: Wednesday 29 th September		
8.30 - 9.00	Registration		
9.00 - 9.10	Introduction and Welcome		
(1-3)	Rob Fitzpatrick: President of Australian Clay Minerals Society (ACMS)		
Clay mineralogy and geophysics incorporating remote sensing, mineral exploration and environmental management: Chair: Andrew Baker			
9.10 - 9.30 (6 - 7)	Hyperspectral and other techniques in the search for diamondiferous kimberlite near Pine creek, Terowie		
	J L Keeling, A J Mauger and M D Raven		
9.30 – 9.50 (8)	Linking airborne radiometric K, soil mineralogy and terrain to map soil patterns in a salt-affected South Australian toposequence		
	Mark Thomas, Rob Fitzpatrick and Graham Heinson		
9.50 – 10.10 (9)	Geophysical aids for detailed soil maps		
	G.J Street, S. Abbott and K.A. Beckett		
$ \begin{array}{r} 10.10 - 10.30 \\ (10 - 12) \end{array} $	Spectral discrimination of soil and regolith attributes within Herrmann's catchment, Mount Lofty Ranges, SA		
	Timothy Raggatt, Megan Lewis and Rob Fitzpatrick		
10.30 - 11.00	MORNING TEA		
Poorly Ordered Nanoparticulate Minerals (PONM) in Soils or Sediments: Chair: Mark Thomas			
11.00 – 11.20 (13 – 14)	Nanoscale cementation of red-brown hardpan		
	Anna Mahizhnan, R. R. Anand and S. A. Wilde		
11.20 – 11.40 (15 – 16)	Interlayer porosities of HDTMA-vermiculites as determined by positronium annihilation lifetimes		
	W.P. Gates, A. Hill P.G. Slade, and P. Meakin		
11.40 - 12.00 (17 - 18)	Water is a necessary and sufficient condition for the formation of halloysite rather than kaolinite		
	G. Jock Churchman		
12.00 – 12.20 (19 – 20)	Lead isotopes and sulfur-containing minerals for constructing geochemical dispersion models in sulfidic wetlands		
	Andrew Baker and Rob Fitzpatrick		

12.30 - 14.00	LUNCH Demonstration of Mid Infrared to help characterise soils and sediments		
New techniques and environmental applications. Chair: Will Gates			
14.00 – 14.20 (21 – 22)	Flow-through hydrothermal cell: a new Australian tool for in-situ phase transformation studies using neutron diffraction		
	C. Tenailleau, A. Pring and B.K. O'Neill		
$ \begin{array}{r} 14.20 - 14.40 \\ (23 - 24) \end{array} $	Modification of soils to restrain movement of anionic pollutants: laboratory, pilo and field studies		
	Jock Churchman, Britta Pitann, Suzanne Pope and David Chittlebourough		
14.40 - 15.00	Toward the characterisation of sulfide minerals in acid sulfate soils using XRS		
(25)	Mark Fritz, Rob Fitzpatrick and Keith Norrish		
15.00 – 15.20 (26)	Reducing colloid movement through soils: a study in catchments of the Mount Lofty Ranges, South Australia		
	Jon Varcoe, David Chittleborough, Jim Cox and John van Leeuwen		
1530 - 1600	AFTERNOON TEA		
15.50 10.00			
Appl	ications of clays in forenics, industry and technology: Chair: Brett Thomas		
Appl 16.00 – 16.20	In Third Coord Thir ications of clays in forenics, industry and technology: Chair: Brett Thomas How pedology and mineralogy can assist in solving crime cases: a case study		
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Hyperspectral and other techniques in the search for diamondiferous kimberlite near Pine creek, Terowie

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The discovery in 1969 of diamond indicator minerals near Terowie by DeBeers subsidiary Stockdale Prospecting Ltd led to the location, in 1971, of a substantial kimberlite diatreme, over 6.3 hectares, near Pine Creek homestead, 33 km east of Terowie. Smaller diatremes and kimberlite dykes were subsequently located in the district including a swarm of diamond-bearing kimberlite dykes near Eurelia from which bulk sampling recovered 163 diamonds totalling 1.46 carats (Muggeridge, 2001). The Pine Creek kimberlite cluster is thought to be associated with a north-west-trending swarm of kimberlite dykes of probable Jurassic age, ~175 Ma, interpreted to have been emplaced over an area of some 100km by 20km centred on the township of Peterborough (Flinders Diamonds Ltd, 2004). Similar dyke swarms in South Africa host commercial grades of diamonds, but the grades vary widely between dykes and include numerous dykes completely barren of diamonds. The exploration objective is therefore to locate as many dykes as possible and to test each for diamond grade using an initial 20kg sample for micro-diamond content with follow up bulk sampling where results are positive (Flinders Diamonds Ltd, 2004).

The use of ground and airborne spectral techniques to locate near surface kimberlite and other diamond host rocks was pioneered by DeBeers. The company included Pine Creek as a test site for both ground (1992) and airborne techniques (1997) (Hussey, 1998). In March 2002, PIRSA/CRC LEME acquired 4x26km data strips over Pine Creek using the HyMap airborne spectrometer (HyVista Corporation). Each run covered a 2km swath with a pixel size ~4.5m. Interpretation of the data was supported by surface and subsurface samples of Pine Creek kimberlite from trenches excavated for indicator mineral dispersion investigations (Howard, 2003). Mineralogy down the profile in weathered kimberlite is summarised in Table 1. The results, together with scanning electron microscopy/EDX analyses confirm that Mg-Fe smectite is the dominant mineral phase responsible for a strong 2300nm wavelength absorption feature over the kimberlite. Dolomite, with a broad 2326nm feature, is also present.

Processing techniques included spectral unmixing/end-member selection, and colour RGB composites for bands 115:118:109 with density slicing on band 115 (2313nm). These effectively outlined near-surface kimberlite in the Pine Creek cluster. They also highlighted two smaller known diatremes in the Calcutteroo area. In all cases, surface disturbance by previous trench sampling significantly enhanced the spectral anomaly. Field checking of a number of identified spectral "targets" showed these to be recrystallised Mg-carbonate veins or soil carbonate; others remain ambiguous.

Vegetation cover was an impediment in some areas and aeolian inputs were sufficient to mask shallow bedrock in others.

Depth	Bulk mineralogy	<0.2µm
0.0-0.5m	Quartz, calcite, dolomite, vermiculite, smectite,	Nontronite, saponite,
	hematite, muscovite, kaolin, talc, feldspar	calcite, kaolin, muscovite
0.5-0.8m	Calcite, dolomite, quartz, vermiculite, smectite,	Nontronite, saponite,
	hematite, phlogopite, kaolin, talc, feldspar	calcite
0.8-1.3m	Calcite, dolomite, quartz, vermiculite, smectite,	Nontronite, saponite,
	hematite, phlogopite, talc	calcite, sepiolite?
1.3-2.2m	Calcite, dolomite, ankerite, vermiculite, smectite,	Nontronite, saponite
	anatase	

Table 1: Pine Creek Kimberlite: mineralogy (XRD) of profile in weathered kimberlite.

Results were not sufficiently encouraging to recommend expenditure for a more regional hyperspectral coverage, especially given that narrow dykes, commonly <1m wide, are the focus of current exploration in the area. Company exploration techniques presently include detailed airborne magnetic surveys, stream and loam sampling for indicator minerals picroilmenite, pyrope, chromite and chrome diopside, and targeted soil geochemistry for elevated Nb, Cr and Ni. The role of strike-slip macro-faults as a structural control on kimberlite dyke emplacement in this region was raised by Curtis (2003) who argues that detailed structural mapping also would aid in locating further kimberlite vent sites.

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Linking airborne radiometric K, soil mineralogy and terrain to map soil patterns in a salt-affected South Australian toposequence

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In this study we combine soil data (soil mineralogy, morphology and chemistry), terrain and airborne radiometric K from a representative toposequence in an upland farming area near Spalding in the mid-north of South Australia to characterise and map complex soil-landscape patterns (Fitzpatrick *et al.* 2003). These patterns, which were aggregated into distinct landscape soil units (LSUs) based on soil and landscape properties, govern the distribution of saline-sodic patterns in the landscape.

X-ray diffraction (XRD) analyses of the clay fraction (< 2 micron fraction) from representative soils from the LSU's indicate that layer silicates are dominated by either (i) co-dominant mica and smectite, or (ii) chlorite. Both mica and smectite are radiometric K-rich minerals, whereas chlorite is not a radiometric K-rich mineral. Airborne-based, high radiometric K response/co-dominant mica-smectite zones were found in toeslope/footslope areas, corresponding to deep alluvial sodic clay soils (LSU 4). Airborne-based, low radiometric K response/co-dominant mica-smectite zones were found in backslope areas, corresponding to leached sodic-saline duplex soils (i.e. loams over clays) (LSU 3). Chlorite dominated the soil-clay fraction higher in the toposequence. In these zones low radiometric K responses on shoulder slopes corresponded to sodic clays (LSU 2), while in crest zones, these radiometric patterns corresponded to shallow clay loams (LSU 1).

Soil data indicated that low radiometric K response soils (LSUs 1-3) were generally saline-sodic (EC_{se} 2.5 - 5.5 dS/m; ESP >5 at <1 m), whereas high radiometric K soils (LSU 4) were non-saline. By linking spatial patterns from soil data and landform, we propose that the reduced subsoil hydraulic conductivity of LSU 4 soils (due to sodic soil-clay fraction dispersion) impedes downslope subsoil freshwater flows, creating a landscape-wide barrier to subsoil freshwater flushing. This in turn has lead to salts being backed up and concentrated in the upslope soils - particularly in the LSU 3 soils that are immediately upslope. We conclude that these representative toposequence patterns may be used to predict saline-sodic soils in these types of landscapes.

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Geophysical aids for detailed soil maps

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A 2000 hectare section in an intensive irrigation area was mapped with ground gamma radiometric and electromagnetic conductivity instruments. The collected data has been analysed for detailed soil mapping. Unsupervised classification of the data showed the radiometric and electromagnetic data reflect patterns that are most likely related to soil and/or landscape properties. At least 10 separate classes can be separated from the geophysical data. The classes were field checked and shown to have observable differences in texture and texture contrast between horizons. Existing geological maps and topographic data show the area is dominated by a colluvial/alluvial fan emanating from a creek draining from laterite weathered Archaean basement. The analysis of the geophysical data shows fine detailed patterns in the final maps that reflect the variability of such an environment. Such detail could not be achieved using conventional soil mapping techniques. Final products from this study include maps of texture differences, drainability of soils and soil textures. The data can now be used to guide future land use and for land management.



Figure 1. Map of soil texture differences in B horizon draped on a vertically exaggerated topography.

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Spectral discrimination of soil and regolith attributes within Herrmann's catchment, Mount Lofty Ranges, SA

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Introduction

The power, and ultimately the success, of remote sensing systems lies within the way radiation over the visible, near- and short wave- infrared portions of the electromagnetic spectrum (400-2500nm) interacts with earth surface materials and the ability of sensors to record this interaction (Campbell, 1996). These interactions exist as spectral absorption and emission features (spectral signature), and are the diagnostic features allowing the spatial determination of the distribution of many hydroxyl and carbonate minerals (Hunt, 1979).

The diagnostic spectral features, which are accessible to remote sensing through atmospheric windows, are caused by vibrational overtones, electronic transfers, charge transfers and conduction processes characteristic of each type of mineral (Hunt, 1979). Thereby allowing the potential determination of the mineral components of soil. Clay minerals, for example, have distinguishing spectral absorption features in the short wave- infrared (~1950nm-2300nm) and successful mapping, to some degree, of smectite, kaolinite, montmorillonite and illite has been documented in many reports, even in vegetated terrains (Chabrilat *et al.* 2002; Ryan and Lewis 2002). Iron oxides and hydroxides also have diagnostic spectral features in the near-infrared (Hunt 1979). The texture, moisture content and organic matter also contributes to the spectral response of soil (Stoner and Baumgardner 1981).

Although laboratory spectrometers have been able to gain the spectral signature of minerals and soil with a high spatial and spectral resolution since the 1970s, only recently have there been remote sensing systems with the same resolution properties. These systems are known as hyperspectral sensors and allow the principles of "laboratory grade" spectra to be applied to spatial mapping (Papp, 2002).

The airborne HyMap[™] (128 spectral band) system is one such hyperspectral system, whose spectral data can be incorporated into image processing software. Such software enables the visual display and processing of data to highlight the occurrence and abundance of specific minerals by using such techniques as Band Ratios, Spectral Angle Mapping, Spectral Feature Fits and Matched Tuned Matched Filtering. All of which have gained popular usage in processing spectral data, and exists as functions in the ENVI software package.

Aims

The underlying aim was to determine and map the spatial distribution and abundance of minerals contained in the catchment using laboratory, field (collected using ASD

FieldSpec) and image reflected spectral data from the solar domain (400-2500nm). Confirmation of such mineralogy was made using XRD.

This included using United States Geological Survey laboratory spectra, field collected spectra and image derived spectra in the previously named processing steps applied to a spatial subset of atmospherically corrected HyMap[™] hyperspectral imagery.

This subset was derived from nine high spatial resolution (~2.5m pixel resolution) flight lines obtained during a late summer (February 2001). Only a small portion of the HyMapTM data was selected for processing, which allowed a time efficient (through reducing the amount of data to be processed) study to identify the most suitable spectral processing techniques for highlighting and identifying landscape features and attributes. The selected research area covers a 2km^2 catchment (Herrmanns Catchment) 10km east of the township of Mount Torrens in the Mount Lofty Ranges, approximately 45km east of Adelaide in South Australia. The selection of Herrmanns Catchment was made based on the reasonably small size of the catchment, and for the variety of soil-regolith attributes and processes that occur in the catchment. Herrmanns Catchment has also been extensively studied throughout the last three decades, and has attracted an interest for mineral prospecting for the close proximity to the Mount Torrens Pb-Zn-Ag mineralisation (CRAE, 1975; Skwarnecki *et al.*, 2003; Fitzpatrick *et al.*, 2003).

However, the main focus of previous research has been on inland saline land and saline sulfidic soils, which have involved pedology, mineralogy, hydrology and physiochemistry investigations (Fitzpatrick *et al.*, 1996; Skwarnecki *et al.*, 2003). The work undertaken on the catchment has lead to an understanding of the processes shaping the catchment, and provides a fantastic example of how science has helped landholders and local landcare groups to manage landscapes. Whilst also developing improved approaches and practices to mineral exploration, and highlighting the link between the CRC LEME programs of mineral exploration, natural resource management, and education and training.

Results

Although the minerals associated with the saline sulfidic environments have spectrally observable features and were of prime interest in the study, the size of the wetlands containing the sulfide minerals and vegetative growth of the catchment always presented a limitation, even with the ~2.5m pixel resolution of the HyMap[™] imagery. However, successful processing with USGS, field collected and image derived spectra was achieved with spectral subsets of the HyMap[™] imagery. These spectral subsets were set in the important discriminative absorptions bands of between 600-800nm regions (Iron Oxides) and 1950-2300nm regions (Clay minerals) as recognised by Ryan and Lewis (2002). Although determining the mineral distribution was limited to exposed soils at dams, scalds, erosion areas, bare rock or areas that bare attributed to livestock and farming practices.

An analysis of the ASD FieldSpec field collected spectra was also performed as part of the Herrmanns Catchment study. The 1.4µm and 1.9µm moisture absorption feature

previously identified by Hunt (1977) was seen in the spectra through observations between *in situ* and oven dried spectral soil sample.

Interesting correlations were also seen in the spectra for the distribution of quartz through a toposequence studied by Fitzpatrick *et al.* (1996). Although quartz has no significant spectral features, it does reduce the overall intensity of reflectance. Iron oxides also stood out prominently in field collected spectra, and existed with minor noise in the spectra of the imagery. The distribution correlated with mapping of ferruginous saprolite by Skwarnecki *et al.* (2003). The field-collected spectra (supported by XRD) also showed goethite in the ferruginous saprolite and hematite in the nearby soils, reflecting the weathering of goethite to hematite.

Conclusions

The use of hyperspectral remote sensing in the solar domain (400-2500nm) to the spatial determination of clay and iron oxide minerals has significance to recognisance studies for both mineral exploration and environmental management. Particularly mapping the spatial occurrence of minerals and landform features associated with inland acid sulfate soils. For example, identifying waterlogged saline scalds, which are an early indicator of the formation of saline swamp-like and structureless soils that are very prone to water erosion (Fitzpatrick *et al.* 1996).

Furthermore the potential for low costs in the collection of spectral data and simplicity in analysing and interpreting this spectral data is a benefit, particularly given adequate information can be derived from the spectral bandwidth and band positions from cheaper and more readily available multispectral systems.

Limitations, as recognised by Hunt (1977), to the reliability of remotely sensed spectral data exist as inherent problems of the sensing system caused by radiance angles, sensor field of view, instrument noise, response time, atmospheric effects and less than adequate spectral resolution.

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Nanoscale cementation of red-brown hardpan

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Red-brown hardpan occurs extensively in featureless, wide, past or present depressions of the arid and semi-arid regions of the Murchison, Pilbara and Eastern Goldfields drainage divisions of Western Australia (Anand and Smith, 1993). It is a near-surface indurated horizon, mainly developed in colluvium and alluvium. Red-brown hardpan commonly varies in thickness from a few centimetres to many metres (Bettenay and Churchward, 1974) and is reddish brown, laminated, blocky, strongly and irreversibly cemented, with ped surfaces coated by manganese oxides. The cements and cementation processes involved in the formation of red-brown hardpan were investigated using X-ray Diffractometry (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Near Infrared Spectroscopy (NIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and dissolution analyses.

Red-brown hardpan is composed of three components: (1) clasts as discrete particles, (2) matrix that consists of clays, silt-sized clastic particles, iron oxides and interstitial spaces and (3) dense, massive, wax-like cements. The cement occurs in different forms, such as small veins and cutanic layers and often occurs as a network and ranges in thickness from $3-30\mu$. SEM investigation of the cement reveals a sub-micron layered structure (Figure 1 A). Individual particles of the layers are composed of spherical to sub-spherical poorly-ordered kaolinite plates ranging in size from 15-46 nm (Figure 1 B), and cemented by opaline silica in a membrane-like form.

TEM investigations confirm the cement is composed of (1) nanometre-sized poorly ordered kaolinites (Figure 2A and C) and (2) amorphous silica (Figure 2B, numbered 1).

Red-brown hardpan is formed in different pedogenic environments. Its parent material is colluvium and alluvium derived from lateritic profiles and contains detrital clays translocated by illuvial processes. The clasts are embedded in a matrix of detrital clays. The interstitial spaces, pores and pore walls are lined by illuviated clays and thus sealing the lower horizons. Plugging of the lower horizons creates a stagnation of aqueous solutions that react with the colluvium and dissolve Si and Al. Intermittent and repetitive infiltration of illuvial clays alternates with times of evaporation, apparently turning the Si and Al rich aqueous solution into a gel, and initiating precipitation of kaolinite and secondary silica, that progressively cements the colluvium.



Figure 1. A. SEM image showing the cross section of the cement composed of an opaline silica layer (1) and cement (2). B. Structure of the cement composed of sub-rounded, poorly ordered kaolinite plates.



Figure 2. TEM images of red-brown hardpan cement. A. Few nanometre thick plates of poorly ordered kaolinite (arrowed). B. Opaline silica (1) and mixtures of opaline silica and poorly ordered kaolinite (2). C. Lattice fringe spacing of 2.81 Å suggesting the (022) plane of kaolinite.

This study suggests that the processes involved in the cementation of the colluviumalluvium are: (1) deposition, illuviation and authigenic precipitation of clays (predominantly kaolinite) and (2) induration and consequent cementation by kaolinite and opal-A on nanometre scale.

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Interlayer porosities of HDTMA-vermiculites as determined by positronium annihilation lifetimes

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Positronium annihilation lifetime spectrometry (PALS) was applied to HDTMA vermiculites with the view to understanding changes in their porosity as affected by method of preparation. HDTMA cations are ordered on vermiculite basal surfaces into interdigitated close-packed arrays with repeat distances coinciding with the *b* cell dimension (Slade and Gates, 2004a). This face centred orthohexagonal super cell has axes equivalent to 3axb with respect to the standard vermiculite cell (Figure 1). Distinct parallel rows of cations form, with aliphatic tails alternately raising up from, and down to, a particular surface along the *b*-axis of the crystal. Each row of HDTMA cations is separated from adjacent rows by empty channels of ~3.60 Å width along *a*, but the rows extend infinitely along *b*. HDTMA-Br organic salt molecules can occupy the channel sites and even after extensive washing with water, some channels remain partially occupied (Slade and Gates, 2004b). The van der Waals attractions operating between the aliphatic tails of the HDTMA-Br molecules and the HDTMA charge-balancing cations are, *en masse*, quite strong and a van der Waals breaking solvent (eg, ethanol) is required to remove the entrained organic salt.



Face centred 3axb Figure 1. (outlined box) orthohexagonal array of HDTMA cations on interlayer surfaces of Nyasaland vermiculite showing positions where HDTMA-Br molecules might reside within channels aligned along b, or in rows where cation vacancies might exist. For vermiculites with layer charges $\leq 1.5 e^{-}$ per unit cell the super cell may not be fully occupied, thus the width along a can vary at Values specific vacancies. indicate channel lengths between adjacent HDTMA-Br molecules.

Certain radioactive decay processes of some elements result in the production and emission of positrons and γ -rays. For ²²Na, a 1.28 MeV γ -ray is emitted within 3 ps of a positron emission event, and is used to define positron birth. Once emitted, positrons interact with molecular electrons, forming a bound state, with a finite lifetime which is reduced in proportion to the density, but increased in proportion to the void space of the material. Further interaction with electrons in molecular bound states at pore surfaces causes annihilation of the positronium ions and the emission of 511 keV γ -rays. The time between the two γ -ray events, ie, between the creation of a positron and its annihilation, defines positronium lifetime. Thus positronium ions, being preferentially localised within the void spaces of materials, can be used to determine their porosity. These values may be otherwise unobtainable by conventional means (Hill, 2003).



Figure 2. Positronium annihilation lifetimes (left) determined on ethanol-washed and waterwashed HDTMA Nyasaland vermiculite. The positronium ion intensities (right) determined for ethanol-washed Nyasaland vermiculite following $N_2 - air - N_2$ purge cycles.

Positronium lifetimes measured (under dry N_2 gas purge) for ethanol-washed HDTMA vermiculites were ~15% longer than for the water-washed HDTMA vermiculites containing HDTMA-Br salt molecules (Figure 2). Positronium intensities, a measure of pore volume, were also greater for the ethanol-washed than for the water-washed HDTMA vermiculites. Positronium lifetimes and intensities measured in the presence of dry N_2 increased, but decreased when the purge was changed to ambient air (Figure 2); the magnitude of such changes was greater for the ethanol-washed HDTMA vermiculites. These results are consistent with greater interlayer pore space, and greater connectivity of the interlayer pores to the atmosphere, for the ethanol-washed compared to the water-washed HDTMA vermiculites.

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Water is a necessary and sufficient condition for the formation of halloysite rather than kaolinite

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The main compositional difference between halloysite and kaolinite is the occurrence of interlayer water in halloysite prior to its dehydration. In 1989, S.W. Bailey proposed that halloysite also contained exchangeable interlayer cations, consequent upon a larger permanent negative charge for its aluminosilicate layer than for that of kaolinite. This paper is a critical examination of that hypothesis based on our studies.

Halloysite and kaolinite formed alongside one another in apparently identical lithological, chemical and physical environments within saprolites from both granites and volcanic tuff in Hong Kong. Each of these minerals appeared in morphological forms that indicated a high degree of crystallinity, implying uninhibited environments for their formation (Fig. 1). Kaolinite was formed whenever the occurrence of oxidation products such as manganese and ferric oxides indicated that drying had taken place; otherwise only halloysite formed. Kaolinite formed after halloysite chronologically in weathering profiles on igneous rocks in Western Australia and also on a variety of rocks in New Zealand. The upper levels of these profiles have likely experienced drying over time, favouring the formation of kaolinite over halloysite.



Figure 1. Examples of highly crystalline forms of halloysite (left) and kaolinite (right) occurring in saprolites from Hong Kong.

Reliable determinations of CECs for halloysites have shown no consistent discrepancies from those of kaolinites. Bailey proposed that more tetrahedral Al in halloysites than kaolinites could give them a higher layer charge. However, ²⁷Al-NMR showed that none of a range of halloysites had sufficient concentrations of tetrahedral Al to affect their layer charge. Water is lost very easily from halloysites, indicating only weak H-bonding to the layers. It was lost especially rapidly when halloysite was re-hydrated

after expansion to form a 14 Å complex with potassium acetate. Weak H-bonding of interlayer water suggests that it occurs free of exchangeable cations.

Structurally, hkl reflections in XRD patterns of complexes of halloysites with polar organic compounds could not be indexed for an expanded kaolinite, confirming that halloysite and kaolinite are distinct mineral species. Therefore they cannot undergo transformation from one to the other in the solid phase. The central role played by water in the genesis of halloysite confirms that halloysites form as hydrated minerals.

From a practical viewpoint, identification of kaolin mineral types may indicate past drying history. Occurrences of halloysite without kaolinite indicate a continuously moist environment, while kaolinite occurring alongside halloysite, or in its place, could show that drying has occurred.

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Lead isotopes and sulfur-containing minerals for constructing geochemical dispersion models in sulfidic wetlands

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In this study we use soil morphology, scanning electron microscopy (SEM), energy dispersive x-ray (EDX), powder x-ray diffraction (XRD) and isotopic techniques to construct a geochemical dispersion model for the formation of sulphur minerals and their oxidative products in sulfidic wetlands.

Study sites consist of three typical, perched wetlands in saline sulfidic discharge areas. Two are located at the Mount Torrens Prospect (45 km east of Adelaide - 139° 01' E; 34° 53' S; area about 2 km²) where minor Pb-Zn-Ag mineralisation occurs in calc-silicate rocks at the base of the Talisker Calc-siltstone (Skwarnecki *et al.* 2002). The twinned site is located at Rodwell Creek 40 km south of Mt Torrens. Strong evidence for sulfide mineralisation has been found but its exact location is yet to be determined. Weathering of the sulfides has produced sulfate-rich groundwaters, which have led to the development of sulfidic seepages that contain a range of acid sulfate soil materials such as sulfidic materials (secondary sulfides containing mainly pyrite) and sulfuric horizons (oxidised sulfidic materials containing a variety of oxyhydroxysulfate and oxide minerals).

The generic model (Fitzpatrick *et al.* 1996) for the formation of the sulfidic materials in these wetlands involves saline groundwaters, enriched in sulfate (with Pb, Zn, etc. sourced from the mineralised zone), seeping up through soils and concentrating by evaporation forming precipitates. The combination of rising sulfate-rich groundwater, anaerobic conditions associated with saturated carbon-rich soils yield pyrite-enriched material through anaerobic bacterial reduction of sulfate. Weak and/or incipient oxidation of the sulfidic materials has produced minerals such as jarosite and plumbojarosite in sulfuric horizons overlying mineralised zones.

Lead isotopic analysis was used to refine this generic model (Baker and Fitzpatrick 2003). The resultant geochemical dispersion model:

- Helped explain groundwater and surface water interaction with Pb reservoirs in the study area.
- Showed that heavy metals and other contaminants tend to build up in organic rich soil horizons with high hydraulic conductivity.
- Provided insight into the seasonal fluctuations observed in the wetlands.

Further detailed studies of heavy metal containing sulfur minerals in reducing and oxidised environments were undertaken for this study. Spatial variability – within Herrmanns and Mt Lofty.

Investigation of organic rich soil horizons using SEM indicated that there was a lowtemperature, biologically mediated PbS precipitation occurring within organic matter. EDX showed that rootlets contained high levels of Pb and S. Studies of sulfidic wetlands proximal to the study sites (within 5 km radius of Mt Torrens) also found evidence of biomineralisation (Skwarnecki *et al.* 2002). Precipitation of PbS and ZnS was located in rootlets within sulfidic horizons. Significant quantities of pyrite (FeS₂) were found in close proximity to biomineralisation at the study sites using SEM and XRD.

X-ray diffraction was used to identify the oxidative weathering products of sulfidic materials including jarosite, plumbojarosite, Ferryhydrite and Schwertmanite. Lead isotopic analysis established the origin of the Pb incorporated in jarosite.

Abundant pyrite proximal to biomineralised heavy metals presents a potential environmental hazard on oxidation. Sandy soils with limited buffering capacity present the most significant risk with toxic heavy metals and acidic water (pH < 4) being released into the environment.

Future work will include transmission electron microscopy (TEM), hydro geochemical modelling, S-isotopes and synchrotron-based x-ray photoelectron emission microscopy (PEEM). This will provide the basis for further improvement of the geochemical model that can be applied to sulfidic wetlands throughout the Mt Lofty Ranges.

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Flow-through hydrothermal cell: a new Australian tool for in-situ phase transformation studies using neutron diffraction

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A flow-through cell, unique in Australia, has been developed at the University of Adelaide and the South Australian Museum for the in-situ study of hydrothermal crystallisation and phase transition of minerals using neutron diffraction. This novel apparatus allows the kinetics and mechanisms of transformation in complex inorganic solids, such as iron-nickel sulphides, zeolites and clay materials, to be investigated under natural conditions.

Diffusion processes at low temperatures are important in controlling the chemical, structural and physical properties and the microstructure of minerals in the geological environment. In cases such as iron-nickel sulphides, principal objects of our research project at the South Australian Museum, Adelaide, these diffusion processes are also of critical importance in mineral processing. Neutron and X-ray diffractions are essential for structural determination and to measure the temperature dependence of Fe/Ni ordering in pentlandite, (Fe,Ni)₉S₈, the primary nickel mineral in nearly all Ni-sulphide deposits, invariably associated with pyrrhotite, Fe_{1-x}S. In the family of iron-nickel minerals violarite, FeNi₂S₄, is the most economically important member of the thiospinel group of minerals. It occurs abundantly in the supergene alteration zones of many massive and dissiminated Ni sulphide deposits where it replaces primary pentlandite or millerite, NiS.

Thus, the transformation study of pentlandite-pyrrhotite to violarite under simulated supergene conditions is of great importance in understanding the mechanisms involved. Neutron diffraction is a very powerful tool for determining the structure and magnetic contribution of a material at a specific temperature and pressure. We have recently developed a flow-through hydrothermal cell that can operate under critical aqueous environments, which is specifically designed for in-situ neutron measurements at ANSTO, Sydney (on MRPD and HRPD instruments) over a wide range of temperatures (25-300°C) and pressures (1-85 bars). The design of a similar cell for high flux in-situ neutron diffraction at ISIS, Oxford, UK (suitable for HRPD, GEM and Polaris instruments) is also part of this project. The apparatus (see schematic below) has two principal components: (1) a hydrothermal cell and (2) a pressure circuit. It will be permanently available at ANSTO for any Australian researcher wishing to undertake insitu neutron diffraction studies under genuine laboratory synthesis conditions.



Modification of soils to restrain movement of anionic pollutants: laboratory, pilot and field studies

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Many elements are lost by runoff from grazed pastures, e.g. in dairying. Some of the elements in runoff e.g. P and dissolved organic carbon (DOC) are of particular concern because they play important roles in the contamination of water bodies Both P and DOC occur in an anionic form in water.

Laboratory studies showed that amendment of bentonites with a synthetic liquid polymer (poly-DADMAC) that is commonly used for drinking water treatment, leads to their adoption of a positive charge and strong uptake of phosphate and other anions. Our subsequent studies have shown that: (1) all types of clays and soils can be amended the same way; (2) sorbed anions are only very slightly desorbed in water; (3) leaching columns of soils with topsoils amended with poly-DADMAC at least delays losses of P from the column, and (5) poly-DADMAC shows no perceptible microbial degradation in soils.

We are carrying out a study of the effectiveness of application of poly-DADMAC to soils on a sloping catchment used for dairying for the restraint of P and DOC that is transported from the pastures into water. A rainfall simulator was used to tests the effectiveness of applications of poly-DADMAC to restrain P and DOC.

The concentrations of both P and DOC in runoff could be decreased substantially (by up to 5-10 times their values in runoff from untreated sites) by applications of poly-DADMAC, provided sufficient time (some days) was allowed for the reaction of polymer with soil (e.g. Fig. 1).



Figure 1. Mean values for concentration of P in runoff following treatments of soil with different amounts of polymer

It appeared to be necessary only to treat strips of plots (at their base) to enable the same extents of removal of P and DOC from runoff that were achieved by treating whole plots (Fig. 2). This means that the cost of treatment can be greatly reduced.



Figure 2. All values for concentration of DOC in runoff following treatments of different proportions of plots with 0.01 g polymer/g of soil that was treated

Preliminary results with a cationic derivative of the natural biopolymer, chitin viz. chitosan have shown that this is similarly effective for modifying clays to generate a net positive charge and enable the uptake of anions (P was used) from water.

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Toward the characterisation of sulfide minerals in acid sulfate soils using XRS

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The management of acid sulfate soils requires analytical methods that can provide accurate and reliable data on the content of reduced inorganic sulfur species such as pyrite. Chromium reducible sulfur method is the preferred technique for the determination of pyrite in soil samples. However, it is sometimes impractical (and costly) to perform chromium reducible analyses, as samples have to be dissolved by time consuming and hazardous acid decomposition procedures.

In 1989 Pinkerton and Norrish successfully speciated S(VI) sulfate and covalent sulfur (methionine) in plant samples using X-ray spectrometry (XRS). The method employed was unique as it only relied on measuring the peak intensity and applying an overlap correction function. Due to is high accuracy (within 0.5% relative) and robustness this method has used for the routine analysis of analytical samples. X-Ray Fluorescence Spectroscopy has been used in geological surveys of sulfate minerals, soils, sediments and acidic drainages. However the speciation of sulphur minerals in these matrices has never been attempted.

The main advantage of an XRS method over chromium reducible speciation techniques is that XRS works by direct surface analysis. Consequently as no solvents or acids are required and no additional hazardous wastes are generated. Furthermore as prepared samples can be used for major and trace elemental analysis, sample speciation can be performed simultaneously with conventional quantitative procedures.

In this paper we will discuss the progress of an XRS speciation technique and its potential application to the characterisation of sulfide minerals in coastal acid sulfate soils.

Reducing colloid movement through soils: a study in catchments of the Mount Lofty Ranges, South Australia

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Fresh water streams in Southern Australia generally have high levels of dissolved organic carbon (DOC) and many streams and water storages have environmentally significant amounts of phosphorus. These two agents are significant contributors to low water quality in southern Australian reservoirs, waterways and water storages and are sufficiently high in DOC to warrant removal by water treatment. Some streams draining the Mount Lofty Ranges watershed, for example, have four times the recommended US Environmental Protection Agency levels of phosphorus and levels of dissolved organic carbon above 20 mg/L. In some streams nitrate is also unacceptably high. We hypothesized that the movement of these components was in the form of organomineral complexes of colloid size and that soil management, in particular the application of calcium amendments, offered the prospect of significantly reducing the problem.

The amounts and pathways of organic carbon, clay colloids and P movement were studied in paired catchments, on small hillslope plots and in lysimeters. We measured loss in both surface runoff and throughflow. Surface application of gypsum markedly reduced losses of natural organic matter and clay. Reduction in P movement was also measured.

We show that calcium amendments, in the form of gypsum can significantly attenuate a number of environmentally important contaminants and thereby improve the quality of water draining from catchments. The issue now is how to make this a practical proposition for landholders and catchment managers.

How pedology and mineralogy can assist in solving crime cases: a case study

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Background

It has now become well known that appropriate soil science techniques can effectively be employed to assist detectives or forensic scientists in the search, location and recovery of soil and mineral samples from crime or disaster scenes. The first use of soil evidences appeared in the fictional literature of the Sherlock Holmes series written by Sir Arthur Conan Doyle. Several actual case studies where soils were used in physical evidence have appeared in the forensic literature for over a century (e.g. Murray 1991 and Saferstien 1998). According to Murray (1991), George Popp was apparently the first scientist who examined soil evidence, which led the solution of the murder case in 1904. The principle used in forensic investigations is to determine if samples "compare' or "do not compare". Then if they do compare a professional judgement should often be made to establish the degree of probability that the samples originate from a single place. Consequently, it is important to first define the word "compare" because no two physical objects can ever, in a theoretical sense, be the same (Murray and Tedrow, 1992). Similarly, a sample of soil or any other earth material cannot be said in the absolute sense, to have come from the same single place. However, according to Murray and Tedrow (1992) it is possible to establish "with a high degree of probability that a sample was or was not derived from a given place".

Soil types vary between different areas and may also have some unique characteristics because of the natural impacts and transfers made by human and other living beings over time. Consequently, forensic examination of soils is not only concerned with analyses of naturally occurring soils, regolith, minerals, rocks, organic matter and animal matter but also includes detection of manufactured materials (e.g. synthetic fertilizers, glass, paint chips, asphalt, brick fragments, and cinders). The presence of such artifacts may impart soil with unique characteristics of a particular location. Many screening and analytical methods have been applied for determining soil characteristics that differentiate and discriminate soil samples. Two of the best known methods used in forensic laboratories for discriminating soils for forensic purposes are soil colour comparison and soil mineralogical analyses.

Case Study

This forensic case discusses how detailed morphological (colour, texture, structure, roots, particle shapes), chemical (pH, EC, elemental composition) and mineralogical (X-ray diffraction) investigations on soil-regolith material from a shovel, boots and jewellery combined with field observations and geological/soil map examinations were used to discriminate and match soils to help locate two buried bodies.

The suspect was arrested on Yorke Peninsula, 150 kilometres from the murder scene in the Adelaide Hills, a day after two women disappeared. Although the victims' bodies had not been located, there was an overwhelming case against the suspect because a bloodstained shovel with soil-like material on the blade was recovered from his vehicle.

Presence of angular quartz and 3mm white flakes together with low pH and EC values ruled out the origin of the soil from Yorke Peninsula but rather from the Adelaide Hills. Identification of crystalline kaolinite, mica and talc combined with geological/soil map information suggested its origin from a gravel quarry occurring in a specific province of strongly weathered regolith. This information, together with other physical evidence, led police and pedologists to undertake investigations in a quarry. Samples were taken of waterlogged soil-regolith in the quarry because their morphological features closely matched soil-like material on the shovel. XRD patterns of the waterlogged soil-regolith were identical to soil-like material on the shovel, suggesting the victims were buried at this site. This led detectives to conduct repeated observations at the waterlogged-site. Decomposition of bodies led to fox activity, which revealed their locations in the quarry 3 weeks after the victims' disappearance.

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Arumpo bentonite in dam lining and geotechnical barriers

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Irrigation and water storage dams are an essential feature of Australian agriculture, and it is important that they do not leak or fail. Likewise, in landfill and containment barrier applications it is important that there is no contamination of the surrounding soils or groundwater. The linings or barriers must have a very low permeability and be physically stable. For highest performance barriers the NSW EPA has recommended a barrier equivalent to 900 mm of material with a permeability of 10-9 m/s.

Arumpo bentonite is ideally suited to dam and repository sealing and, applied as a blanket or in admixture with the local clay, provides an economical means of creating high performance barriers.

The unique sealing properties of Arumpo bentonite are associated with its extremely fine natural grain size, relatively modest swell and moderate liquid limit compared with most bentonites. Bentonite from the very large, high purity Arumpo deposit, wetted within the plastic limit, shows exceptionally low permeability, with values between 10-10 and 10-11 metres/sec depending on the overpressure, and would easily meet EPA specifications (Figure 1 – from Churchman *et al.*, 2002). In addition, country clays mixed with 10–15% of Arumpo bentonite show large decreases in permeability to values of 10-9 or less (Figure 2). Fine or granular material is easily transported and dispensed. These properties, and its high capacity for the absorption of heavy metals, make Arumpo bentonite ideal as a barrier and capping medium for waste dumps, cut-off walls, bore hole sealing and dam lining.

To date earth dams as large as 300 megalitres with poor natural sealing performance have been successfully treated with Arumpo bentonite, a landfill site near Lancefield, Victoria has been sealed to EPA standards, and a variety of geotechnical barriers have been installed.



Figure 1. Permeability of Arumpo* and Wyoming bentonites.





Figure 2. Permeability vs bentonite addition for a clayey sand

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Clays, Capillarity and Metal Ion Dispersion into Pleistocene Sediments at Moonta, South Australia

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The Moonta Mines on northern Yorke Peninsula, South Australia were an important source of copper providing a major contribution to the 330,000 tonnes of Cu produced from the Moonta-Wallaroo district during 1860-1923. Ore was mined from narrow shear-hosted copper sulphide veins, in host Moonta Porphyry of Palaeoproterozoic age. In this semi-arid landscape, <400mm annual rainfall, of flat to gently undulating topography and poorly developed surface drainage, shallow soil geochemistry was used with considerable success to delineate copper-mineralised shears, even where overlain by sediment cover (Jack, 1917; Sokorloff, 1948; Mazzucchelli, *et.al.*, 1980). Investigations by Hartley (2000) on metal ion distribution in sediments sampled from pit sections at abandoned mines at Poona and Wheal Hughes showed significant differences in Cu content in calcrete and Pleistocene clay at each mine (Fig 1). Explanation of the difference requires consideration of landscape position, subsurface geology, sediment texture, water table movement, and probable transport mechanisms for metal ions through the unsaturated or vadose zone.

The Wheal Hughes and Poona Mines are 1.7 km apart in an interdrainage setting, 4.5 km from the coast. Water table is about 20m below ground surface and is very saline (~42,000 mg/L). The porphyry is variably weathered for some 15m; the top 4 to 6m is highly weathered and mottled. Palaeomagnetic dating of iron-rich mottles at Poona gives Late Miocene to Pliocene ages of 8 ± 4 Ma (Pillans pers com., 2004). The weathered profile and supergene Cu processes were therefore well developed before deposition of the 2-4m-thick sandy clay equivalent of Hindmarsh Clay of Early Pleistocene age (~0.78 Ma). The clay is overlain by 1-2m of calcareous aeolian sand and clayey silt with nodular and platy calcrete, and 0.6m calcareous clay loam topsoil. At Wheal Hughes, the Pleistocene deposits overlie a 1-4m-thick remnant of coarse-grained, arkosic sandstone, Winulta Formation of Cambrian age, which caps the weathered porphyry (Keeling *et al.*, 2003).

Inferred low infiltration rates, generally lower water tables, and high evaporation during Pleistocene-Holocene times draw attention to the role of metal ion diffusion and upward capillarity. Both mechanisms of ion transport have deficiencies in explaining the significant accumulation of atacamite and the presence of alunite in Pleistocene clay at one site and not the other. The role of the Cambrian sandstone in limiting capillary rise is argued as a significant factor. However, further work is required to fully assess the factors driving metal mobilisation into transported cover and the twin sites of Poona and Wheal Hughes provide an ideal opportunity for such research.



Figure 1. Box and whisker plots of Cu content in near surface calcrete and alunite samples from pit sections at Poona and Wheal Hughes mines highlighting the difference in Cu dispersion for the two deposits relative to the regional anomaly threshold of 30 ppm Cu (after Hartley, 2000).

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Interpreting pedogenesis without x-ray eyes or a time machine

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Understanding the origins of soil features is confounded by the influence of soil forming factors (Jenny 1941) on suites of processes (Simonson 1959). However, there is a large body of knowledge to support various hypotheses and suppositions concerning the relationships between soil features and the pedogenic processes that have caused them.

In the Australian context the time and climate factors have particular importance since much of the land surface is old and has been subject to quite different climatic environments to those evident today. This has resulted in soils that have sometimes been referred to as 'polygenetic', i.e. have had diverse genetic influences, and may display paleo-features that reflect a different climatic influence to those that correspond to the contemporary environment. The obvious soil features that we describe are colour and texture and these are clearly an expression of weathering of a soil parent material. They are the result of the primary mineral material, the formation of secondary minerals and the redistribution of soil material in suspension or by solution and precipitation. Soil hydrology is the primary driver for these transformations. Soil structure, which may in part be the result of hydrological processes such as clay illuviation, provides the spatial context for interaction of water and soil, in particular the important process surrounding reducing and oxidising environments. 'Reading' the redoximorphic features in soil profiles requires a recognition of the structural controls on soil hydrology, and also an understanding or speculation of the climatic and landform influences. An explanation will be presented for the development of texture-contrast soils that are rich in ironstone gravels in South-West Victoria. The supporting evidence is purely visual and textural but could be enhanced with appropriate analytical techniques to identify clay and iron mineralogy and the age of materials in the landscape and soil profiles.

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