VOLUME II

A FIELD APPRAISAL OF THE ROLE OF
PLANT OPAL IN THE
AUSTRALIAN ENVIRONMENT

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Submitted for the degree of Doctor of Philosophy
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A model for plant opal cycling

Appendix F Sample Preparation Techniques

Introduction

A: Sample preparation

(i) Overview of techniques:

1. Extraction of plant opal from plants

2. Extraction of plant opal from sediments

(ii) Mounting

(iii) Methods used in this thesis

1. Extraction from plants

2. Extraction from sediments

3. Mounting of samples

B: Analysis techniques

Introduction

(i) Percentage content of plant opal by weight

Surface area

(ii) Plant opal morphology and assemblage analysis

Appendix G Data Sheets

Introduction

Data sheets

Grain size analysis

Scanning and statistics sheets

Surface area data

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1. SEM micrographs have a scale bar in the bottom right-hand corner, the length of which is noted in the caption.
2. On the captions of photographs of thin sections and plant opal taken under a petrological microscope the following descriptors are used:
   PPL Plain Polarised Light
   XPL Crossed Polarised Light
3. MU numbers refer to the photographed sample's number in the samples held in the Macquarie University Museum.
4. Soil Profile numbers are given where necessary. A full description of each profile is to be found in Appendix D.
PLATE 1
SEM micrographs

<table>
<thead>
<tr>
<th>A</th>
<th>bilobate</th>
<th>planar view, sediment from A2, solodized solonetz, site 8, Pilliga, scale is 1 um</th>
</tr>
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<tr>
<td>MU 50482</td>
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<table>
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<tr>
<th>B</th>
<th>bilobate</th>
<th>side view, sediment from A1, site 12, Pilliga, scale is 10 um</th>
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<tr>
<td>MU 50519</td>
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<table>
<thead>
<tr>
<th>C</th>
<th>polylobate</th>
<th>top view, sediment from A1, site 8, Pilliga, scale is 10 um</th>
</tr>
</thead>
<tbody>
<tr>
<td>MU 50517</td>
<td></td>
<td></td>
</tr>
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</table>

| D          | polylobate or rod? sediment from A2, site 8, Pilliga, scale is 1 um               |
|------------|-----------------------------------|----------------------------------------------------------------------------------|
| MU 50481   |                                    |                                                                                   |

<table>
<thead>
<tr>
<th>E</th>
<th>cross</th>
<th>planar view, sediment from B1, site 8, Pilliga, scale is 1 um</th>
</tr>
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<tr>
<td>MU 50483</td>
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<td></td>
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</table>

<table>
<thead>
<tr>
<th>F</th>
<th>cross</th>
<th>top view, sediment from A1, Botanic Gardens, Sydney, scale is 1 um</th>
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<table>
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<tr>
<th>G</th>
<th>saddle</th>
<th>top view, sediment from B2, site 8, Pilliga, scale is 1 um</th>
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<tr>
<th>H</th>
<th>double outline</th>
<th>top view, carton from termite's nest, site 8, Pilliga, scale is 1 um</th>
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<tr>
<td>MU 50503</td>
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PLATE 2
SEM micrographs

A cone MU 50499
from leaves and stem, Stipa sp.
scale is 10 um, side and top views.

B cone MU 50497
sediment from A1, Botanic Gardens, Sydney, scale is 1 um
Top view.

C cone MU 50500
from leaves, Stipa sp.
scale is 10 um, side and top view.

D rod, thin and cones MU 50492
spiked (top of micrograph) various views.
from Stipa sp., Pilliga,
scale is 10 um

E rod, thick MU 50487
smooth from Pteridium esculentum roots, Oxford Falls, Sydney,
scale is 10 um, side view.

F rod, thick MU 50492
spiked from Stipa sp.
Pilliga, scale is 10 um

G rod, thin MU 50501
spiked from root of Stipa sp.
scale is 10 um

H rod, thick MU 50483
jigsaw, sediment from B1, site 8,
Pilliga, scale is 10 um, top view.
PLATE 3
SEM micrographs

A  rod, thick  jigsaw sediment from B1, site 3, Pilliga, scale is 1 um, top view.
   MU 50483

B  rod, platey  jigsaw, sediment from A1, site 3, Ecology Reserve, scale is 10 um, top view.
   MU 50521

C  rod, platey  jigsaw, leaves and stem from Stipa sp., scale is 10 um top view.
   MU 50502

D  rod, platey  jigsaw, from Stipa sp. Pilliga, scale is 10 um top view.
   MU 50490

E  rod, thick  ridged, sediment from organic pan podzol, Narrabeen, scale is 1 um
   MU 50486

F  rod, thick  ridged, sediment from domes, site 8, Pilliga, scale is 10 um
   MU 50481

G  rod, thick  rough, sediment from the organic pan, podzol, Narrabeen, scale is 10 um
   MU 50486

H  sheet 2D irregular honeycomb, from Banksia asplenifolia leaves, Oxford Falls, scale is 10 um top view.
   MU 50477

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<th>Description</th>
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<td>plain, from <em>Dodonaea vicosa</em> ssp. <em>cuneata</em> leaves, site 8, Pilliga, scale is 10 μm</td>
</tr>
<tr>
<td>B</td>
<td>2D irregular</td>
<td>perforated, from <em>Calytrix tetragona</em> site 2, Pilliga, scale is 1 μm</td>
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<td>C</td>
<td>2D regular</td>
<td>bulbous, multicelled, from Ecology Reserve ash, scale is 10 μm, top view</td>
</tr>
<tr>
<td>D</td>
<td>2D regular</td>
<td>multicelled, from Ecology Reserve ash, scale is 10 μm, top view, contains several morphologies in growth position</td>
</tr>
<tr>
<td>E</td>
<td>2D irregular</td>
<td>honeycomb, sediment from B1, site 8, Pilliga, scale is 10 μm</td>
</tr>
<tr>
<td>F</td>
<td>3D regular</td>
<td>smooth, sediment from A1, Pilliga site 8, scale is 10 μm, top view</td>
</tr>
<tr>
<td>G</td>
<td>3D regular</td>
<td>rough, carton from termite's nest, site 8, Pilliga, scale is 10 μm, top view</td>
</tr>
<tr>
<td>H</td>
<td>3D regular</td>
<td>rough, sediment from A1, Gardens, Sydney, scale is 10 μm, top view</td>
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Plate 5
SEM micrographs

A  sheet 3D regular verrucose, sediment from A1, site 8, Pilliga.
MU 50517 scale is 10 um

B  sheet 3D irregular multicelled, from Ecology Reserve ash, scale is 10 um
MU 50523 top view

C  sheet 3D irregular multicelled, from leaves of Dodonaea viscosa spp.
MU 50495 cuneata, site 8, Pilliga
scale is 10 um top and side views

D  prickle, long multicelled, from Ecology Reserve ash, scale is 10 um
MU 50521

E  prickle, long from Ecology Reserve ash scale is 10 um
MU 50521

F  prickle, long sediment from A1, site 7, Pilliga, scale is 10 um
MU 50516

G  prickle, long sediment from A1, site 8, Pilliga, scale is 10 um
MU 50517

H  prickle, short sediment from the organic pan, podzol, Narrabeen
MU 50485 scale is 1 um
Plate 6
SEM micrographs

A  prickle, gourd-shaped sediment from A1, site 9, MU 50519 Pilliga, scale is 10 um

B  sphere, single smooth, sediment from domes, MU 50482 Pilliga site 8 scale is 1 um

C  sphere, single spiked, sediment from A1, site 8, MU 50518 Pilliga, scale is 1 um

D  sphere, single rough, sediment from A1, site 7, MU 50515 Pilliga, scale is 1 um

C and D are possibly chrysophyte tests

E  sphere, single perforated, from Unk sp.1, MU 50488 Oxford Falls scale is 10 um

F  sphere, compound, smooth, from leaves Melaleuca uncinata, site 1, Pilliga scale is 1 um

G  sphere, compound, smooth, from leaves of Eucalyptus gummifera, MU 50489 Oxford Falls scale is 1 um

H  sphere, verrucose, sediment from A1, site 8, MU 50517 Pilliga, scale is 10 um
| Plate 7 |
|-----------------|---------------------------------|
| **SEM micrographs** |
| **A** | sphere, compound, rough, sediment from Ecology Reserve ash |
| MU 50520 | scale is 10 um |
| **B** | diatom (centrale) sediment from B1, site 8, Pilliga, scale is 1 um |
| MU 50483 | |
| **C** | diatom (centrale) sediment from A1, site 7, Pilliga, scale is 10 um |
| MU 50516 | |
| **D** | diatom (centrale) sediment from organic pan, podzol, Narrabeen |
| MU 50486 | scale is 1 um |
| **E** | diatom (pinnale) sediment from domes, Pilliga site 8 |
| MU 50481 | scale is 10 um |
| **F** | chrysophyte? carton, site 8, Pilliga |
| MU 50503 | scale is 10 um |
| **G** | chrysophyte sediment from A1, site 12, Pilliga |
| MU 50515 | scale is 10 um |
| **H** | sponge spicule sediment from A1, site 7, Pilliga, scale is 10 um |
| MU 50516 | |
PLATE 8
SEM micrographs

Scale is 10 um unless otherwise stated.

Plant opal separated from Oxford Falls swamp
Core OFC2. MU# 50478

A Sand-sized plant opal. Mainly thick sheets and multicelled material. Scale is 100 um.

B Sand-sized plant opal. Close up of the right-hand group in A. May exhibit some welding together of grains by re-crystalization.

C Sphere, single, rough.

D Group of multicelled platey material including silicified stomatal cells.

E Thick, jigsawed rod.

F Thick rod, spiked.

Cyperaceae-type phytolith from Acacia species in the Piliiga State Forests.

G Acacia calamifolia. Contains a few of the phytolith, mainly in single cones.

H A. spectabilis. The bulbous variant is common and appears single only.
PLATE 9
SEM micrographs

Scale is 10 um unless otherwise stated.

Cyperaceae-type phytolith from Acacia species in the Piliiga State Forests.

A  
**A. spectabilis.** A group of bulbous phytoliths.

MU# 50511

B-D  
**A. lineata.** Rows of cones with some satellites.

MU# 50513

E  
**Acacia deanei ssp. deanei.** Single conical body on plate.

MU# 50512

F-G  
**A. deanei ssp. deanei.** Rows of conical bodies with satellites.

MU# 50512

H  
**A. triptera.** A row of conical bodies with satellites.

MU# 50514
PLATE 10
SEM micrographs

Plant opal from the Oxford Falls podzol, Soil Profile #OF2.

A rough sphere from the A1 (0-50 mm). Scale is 10 um.
MU# 50504

A thick, ridged rod from the A2 (150-200 mm). Scale is 10 um.
MU# 50506

A pitted thick rod from the organic pan (400-450 mm). Scale is 1 um.
MU# 50507

Part of a thick, ridged rod from the organic pan (400-450 mm). Scale is 10 um.
MU# 50507

Site 8, mallee. Soil Profile 8iv.

The very dark lining of the faunal channels. This is comprised of faecal pellets pressed together. Scale is 100 um.
MU# 50509

A faecal pellet from within the faunal channels, B horizon. Scale is 1 mm.
MU# 50509

Plant opal from carton, Pilliga Forests, Site 8. MU# 50503

Sheet, 3D, regular, from termite carton. Scale is 10 um.
G

Plant opal from termite carton. Scale is 10 um.
H
The boundary between Site 7 (upper broom plain) and Site 8 (mallee) is very sharp. This photograph, taken from the middle of the road looking west through both sites from the broom plain, shows the abrupt change in both species and vegetation height. There are numerous ant nests in the road, which has been slightly built up and is better drained than the surrounding broom plain.
PLATE 12

Detail of the mallee (Site 8) vegetation. The many stemmed Eucalyptus is *E. viridis*. The low, bright green shrub is *Dodonaea viscosa ssp cuneata* or hop bush.
PLATE 12
PLATE 13

Soil pit, Site 8 (mallee), soil profile #PF8iv. Tape is in cm. Each 10th cm is darkened.

A  View of the pit (to over 1 m). The soil is a Dy4.41 (Northcote, 1974).

B  750 - 1000 mm+ The Pilliga Sandstone forms the substrate for this soil. The sandstone is white/cream with many lenses of darker clay in it.
Soil pit, Site 8 (mallee), soil profile #PF8iv. Tape is in cm. Each 10th cm is darkened.

A The A1 (0-180 mm) of loamy sand, which overlies the domes and penetrates down the sides of each. The cement-like tops of the domes are exposed.

B A closer view of the dome tops. These are penetrated by a few, large faunal channels.
PLATE 15

The lower broom plain (Site 9) looking towards the forest (Site 12). This boundary is also very abrupt. Photograph is taken looking west.
A The gilgai area (Site 11). The trees are *Casuarina cristata*. Note the hollow and hummock appearance of the ground. Relief is around 2 m.

B The "sand monkey", Site 10. Vegetation comprises *Callitris glaucophylla* and *E. chlorocladia*, with *Xanthorrhoea glauca* ssp. *angushfolia* in the shrub layer.
A litter tray located in the mallee (Site 8). The trays measured 50 cm x 50 cm and were constructed of plastic electrical conduit and terylene curtain material.
In these photographs the chisel handle is 10 cm long.

A Site 8, mallee. This photograph shows the surface of the site, which can range from bare soil where animals or ants have their runs, to a thick cover of litter.

B Site 8, mallee. Around the bases of the *E. viridis* are halos of bark and litter in which termites build their nests. The ground around the trees is very spongy due to the network of channels under the surface.
PLATE 19

In these photographs the chisel handle is 10 cm long.

A Site 8, mallee. The termites also build nests around dead material on the mallee floor. This mound is about 40 cm in diameter and very common in this area.

B Site 8, mallee. The underneath of a fallen log has been packed with a mixture of faecal pellets and soil (sheeting) by the termites. They are then able to work their way into the wood.
In these photographs the chisel handle is 10 cm long.

A Site 8, mallee. A log broken open to show the packing of sheeting within it.

B Site 8, mallee. Around 40-50 mm of material has been packed under and around this log. A large amount of soil is brought to the surface and deposited in such areas. Scale is in cm.
In these photographs the chisel handle is 10 cm long.

A  Site 8, mallee. A closer view of the sheeting, showing the packing fabric and the channels along which the termites move.

B  Site 8, mallee. A fragment of a mound broken off to show the incorporation of litter within it. The termites bury the litter before eating it, and thus are responsible for the incorporation of much of the litter layer into the soil before it is broken up or decomposed. Scale is in cm.
Plate 22

A  Mesh bag used to contain litter in litter decomposition experiment. A large amount of soil is adhering to the outside; this is the side of the mesh bag which was exposed on the surface and the soil has been deposited by termites.

B  Rocky Creek, to the south of the field site after rain. The creeks in the area are sandy.
Plate 23

Photographs taken of petrological slides.

A MU 50613: Site 7, upper broom plain pinch sample 7b2. Sheet, 3D, rough-surfaced and pitted. PPL.

B MU 50615: Site 9, lower broom plain pinch sample 9a1. Note the lobates (arrow). PPL.
Plate 24

Photographs taken of petrological slides.

A  MU 50608: Site 2 broom plain pinch sample 2/1. Contains many rough surfaced 3D sheets. PPL.

B  MU 50610: Site 6 broom plain pinch sample 6/1. Contains many rough surfaced 3D sheets. PPL.
Plate 25

Photographs taken of petrological slides.

A MU 50542: *Acacia lineata*. Pilliga State Forests. Plant opal comprises mainly smooth surfaced bodies. PPL.

B MU 50536: *A. tindaleae*. Pilliga State Forests. A large proportion of the plant opal from this species is ridged (arrow). PPL.
Plate 26

Photographs taken of petrological slides.

A-B  MU 50543: *A. triptera*. Pilliga State Forests. Most of the plant opal is highly ridged, including rods (arrow in A) and 3D sheets. PPL.
Plate 27

Photographs taken of petrological slides.

A  MU 50538: *A. calamifolia*. Pilliga State Forests. Many very long ridged rods in this species. PPL.

B  MU 50538: *A. calamifolia*. Pilliga State Forests. Sheets are also heavily ornamented. PPL.
Plate 28

Photographs taken of petrological slides.

A   MU 50537: *A. burrowii*. Pilliga State Forests. Plant opal comprises many ridged 3D sheets. PPL.

B   MU 50541: *A. deanei* ssp *deanei*. Pilliga State Forests. Ridged rods are common in this species. PPL.
Photographs taken of petrological slides.

A  MU 50539: *A. spectabilis*. Pilliga State Forests. A few ridged sheets and rods. Note the bulbous "cyperaceae-type" phytoliths (arrow), beside ridged material. PPL.

B  MU 50539: *A. spectabilis*. Pilliga State Forests. Both smooth and ridged 3D sheets. PPL.
Plate 30

Photographs show the large number of ridged sheet and rods in this species. PPL.
Plate 31

A  MU 50591: Plant opal from the topsoil, 0 - 50mm, Ecology Reserve site 1. Sample E1. A large amount of opal coated with charcoal is present (arrow, LHS of photograph), as well as opal containing occluded material and appearing darker. PPL.

B  MU 50593: Plant opal from the topsoil, 0 - 50mm, Ecology Reserve, site 2. Sample E3. Some charcoal coated plant opal, but the majority of darker pieces contain occluded elements (arrow). PPL.
Plate 32

A  MU 50626: Plant opal separated at a Specific Gravity of 1.7. This comprises large pieces of charcoal within which can be seen plant opal (arrow). PPL.

B  MU 50626: Plant opal separated at a Specific Gravity of 1.7. A rod coated in charcoal (arrow). PPL.
Plate 33

A  MU 50627: Ash from the Ecology Reserve fire. This material has not been treated in any way. It comprises much exposed plant opal, and a great amount of plant opal still encased in charcoal. PPL.

B  MU 50628: Material from the Ecology Reserve fire. In this case an attempt has been made to separate the ash from the plant opal, with very little success. Note the large multicelled plate containing silicified stomata, some of which have been exposed (arrow). PPL.
A  MU 50617: Ash from the Ecology Reserve fire which has been heated in a muffle oven to remove most of the charcoal. Despite this, a little charcoal remains, and the platey material in particular is very dark. It appears that the carbon is either fused to the silica surface, or the opal is more able to incorporate carbon during a fire. It is noticable that this darker material is removed rapidly after the fire. Note the large amount of big, multicedeled sheets present (arrow). Ash from the day 2 sample: sample 2a. PPL.

B  MU 50618: This material is from the day 14 sample (sample 5a). It has been heated and separated from sediment. There are fewer dark pieces of plant opal, and very little charcoal encased opal remaining. The platey, multicedeled material is also less and smaller. PPL.
A  MU 50618: Ecology Reserve fire. This material is from the day 14 sample (sample 5a). It has been heated and separated from sediment. Again it shows fewer dark pieces of plant opal and very little charcoal encased opal or multicelled material. PPL.

B  MU 50619: Material from the day 30 sample (sample 8a). Mainly composed of more robust morphologies. Very little charcoal remains in the sample, and very few multicelled sheets. PPL.
A MU 50595: Ecology Reserve, Site ER3, 0 - 100 mm. This is a site on the creek bank. There is a large amount of charcoal and charcoal encased plant opal in this sample, as well as dark opal with occluded material in it. PPL.

B MU 50598: Ecology Reserve, Site ER3, 1000 mm+. The charcoal in this soil profile is still very evident at this depth and beyond. PPL.
Plate 37

A MU 50624: Diatoms from the Chalk Mountain deposit. These are quite distinctive, and do not appear in the soil in the field sites. PPL.
A MU 50625: Photograph of thin section of soil 0 - 70 mm, Site 8. This sample was removed undisturbed from the field, diatoms placed on the surface, and subjected to repeated wetting, ponding of water, and drying. The sample was solidified and sliced. Diatoms were present to the base of the sample. This photograph shows a diatom within a major void in the soil (arrow). PPL.

B MU 50625: As above. Here a group of the diatoms is caught in a void wall. It can be seen from this how such material would become incorporated into the soil. PPL.
Plate 39

A  MU 50625: Same sample as in Plate 38. This photograph shows a piece of plant opal within the soil (arrow). PPL.

B  MU 50625: As above, crossed polarised light [arrow] (XPL). It is envisaged that plant opal could migrate through the soil in a similar manner to the diatoms.
Plate 40

Photographs of plant opal separated from sediment
Site 8, mallee, Pilliga State Forests.

A MU 50599: Plant opal from sheeting constructed
above ground by termites. PPL.

B MU 50599: As in A. Photograph shows 3D sheets, one
of which has dark inclusions (arrow). PPL.
Plate 41

Photographs of plant opal separated from sediment Site 8, mallee, Pilliga State Forests.

A Mu 50603: Plant opal from A horizon. Includes a ridged rod (arrow) and rod containing inclusions. PPL.

B Mu 50603: Plant opal from A horizon. A pitted 3D rough sheet (arrow). PPL.
Plate 42

Photographs of plant opal separated from sediment
Site 8, mallee, Pilliga State Forests.

A  MU 50605: Plant opal from the domes (sample D1). Includes ridged rods and rough spheres (arrow). PPL.

B  MU 50607: Plant opal from faunal channels, B horizon sample (F1). A multicelled sheet (arrow). PPL.
Plate 43

Photographs of plant opal separated from sediment Site 8, mallee, Pilliga State Forests.

A-B MU 50606: Plant opal from the B horizon (sample B1). PPL.
Plate 44

Photographs illustrating the problems encountered during separation of plant opal from sediment by heavy liquids. Sediment used was from Site 6, broom plain pinch samples.

A During wet sieving of the whole sample to remove the sand it was found that large multicelled sheets as well as a few smaller diameter pieces of plant opal were caught in the sieve. This plate shows a large multicelled sheet (arrow). Sample MU 50620. PPL.

B Above in cross-polarized light, demonstrating the isotrophiic nature of the opal. XPL.
Photographs illustrating the problems encountered during separation of plant opal from sediment by heavy liquids. Sediment used was from Site 6, broom plain pinch samples.

A  MU 50623: Clay which is siphoned off during sedimentation. It contains many small, often sheety pieces of opal (arrow). PPL.

B  MU 50622: Silt material with a Specific Gravity greater than 2.3. This was found to contain no opal. PPL.
Plate 46

Photographs illustrating the problems encountered during separation of plant opal from sediment by heavy liquids. Sediment used was from Site 6, broom plain pinch samples.

A-B MU 50621: Supernatant liquid from cleaning the separated opal. This should be clear liquid but is found to contain a large amount of opal. PPL.
# APPENDICES

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