CHAPTER 6

ECOLOGY RESERVE FIELD STUDY: FIRE AND PLANT OPAL

Introduction

In Chapter 5 it was shown that the plant opal assemblages in the litter and soil at the Pilliga sites differed. The morphological classes most affected were the hairs and prickles, which were reduced by some 50% in the soil, and a reduction in the proportion of two-dimensional sheets. Similarly it was found that at Oxford Falls the soil assemblage lacked certain elements of the vegetation assemblages - the most notable being an absence of plant opal sheets in the swamp sediment.

It is evident that processes causing this change must intervene between the time the plant material dies and it is incorporated into the soil. Such processes must either selectively remove platey plant opal in preference to the more robust and compact forms, or reduce it in size so that it becomes undetected (i.e. <2 um diameter). Dissolution is also a possibility, but the comparison is being made here between the litter layer and the mineral soil directly beneath it and it is considered that dissolution processes would be minimal. Thus we are looking for a process or set of processes which expose and disagregate the plant opal, and/or selectively remove those parts of the litter containing a high proportion of the platey material.

The removal of plant material by soil fauna and the reduction in size of the litter as it passes through the gut of microorganisims may indeed be the only process required to produce this effect. Microarthropods (e.g. springtails and mites) reduce the size of the material they eat to between a few microns to 100 microns in size (Dunger, 1956), but it is possible that other processes contribute, in particular fire.

In Australia, an important event which occurs with a frequency of 8 - 12 years is fire (Cheney, Raison and Khana, 1980). Many fires ash all of the litter on a forest floor and expose it to the winnowing of the wind and rain. Such action would remove material of a platey nature; mainly the ash of leaves and herbs containing a high proportion of platey opal. "Platey opal" includes two-dimensional sheets and rods, and multicelled sheets of plant opal. These have a very low thickness to surface area ratio and are composed of connected two-dimensional plates and platey rods often with other shapes such as lobates, attached (Plate 34). Left behind would be the charcoal of wood containing opal with a low surface area to volume ratio. The silica deposition in New South Wales woods was described by Bamber and Lanyon (1960) as irregular shaped rounded deposits with mature aggregates up to 25 um in diameter, and it could be expected that the larger pieces of charcoal derived from wood and the un-burnt wood would contain a large proportion of these. It is also possible that fires may reach a sufficient temperature for changes to occur to the opal itself - warping, fusion and mineralogical changes.

In order to observe what happens to plant opal during and immediately after a fire, an area was sampled before, immediately after and at two subsequent intervals during the first month after burning. While similar areas to the Pilliga sites would have been ideal for such sampling, unfavourable conditions meant that the Pilliga Forests were not able to be burnt for hazard reduction by the Forestry Commission, and no wild fires in suitable sites were recorded during the period of this study.

An area on Hawkesbury Sandstone having similar vegetation to that in the Oxford Falls catchment is used by Macquarie University as an Ecology Reserve. While conditions were not particularly favourable in

Sydney either for hazard reduction burning, the Metropolitian Fire Board kindly agreed to supervise such a burn on 1 December 1989, thus it was in this area that the observations were made.

A Brief Literature Review

Upon the ashing of plant material, the plant opal is released from its surrounding plant tissue. Such a method was used to provide plant opal for study before it was found that "dry-ashing" releases silica which may not be typical of that found in the plant (Chapter 7).

Jones and Milne (1963) in their study comparing wet and dry ashing of plant material, found that accessory elements were incorporated into the dry-way silica in greater amounts. They noted that a temperature of 1470°C was required to form cristobalite in the pure silica system while cristobalite had been reported to be formed at temperatures as low as 600°C in the presence of sodium and potassium. They heated a sample of wet-way silica at 600°C for 12 hours; however no cristobalite formed. Dry-way silica ashed at 450° and 550°C produced cristobalite and tridymite, probably due to the presence of accessory elements. The dry-way silica particles were very large, often warped and twisted and coated with carbon, and details of structure were blurred by fusion.

Arimura and Kanno (1965) dry-ashed plant material by ignition for 10-12 hours in an electric furnace at 600°C, 800°C and 900°C. One specimen was fused with sodium carbonate at 870°C for 12 hours. Their results showed that crystaline silica minerals were formed by the dry-way ashing at lower temperatures than ordinary inversion temperatures due to the presence of sodium and potassium in the leaves. They found a very small amount of quartz in plant opal separated from soils which they attributed to the ashing action of wild grass fires (temperature

700°C). However Jones and Beavers (1963c) heated opal separated from Cisne silt loam to 900°C for 4 hours and found no cristobalite reflections on the XRD's.

Thus, it would appear that plant opal requires heating to at least 450°C in the presence of accessory elements which might be expected to be found in the leaves of the plant, and for at least 12 hours in order for crystaline silica to form. While the first two conditions are definitely met during a bush fire, the third is rarely met. Such conditions may occur when a large log continues to burn for several hours. Under dry leaves and branches 10-20 cm in diameter, Floyd (1966, quoted in Clinnick, 1984) found temperatures reached 510°C during a fire, but remained above 100°C for only 15 minutes. Under larger logs (>1 m in diameter) temperatures were similar and were maintained for more than 12 hours; thus there could be significant mineralogical as well as morphological changes to plant opal under these conditions.

Carbon coatings have been reported on plant opal extracted from soil and on plant opal prepared by ashing (Jones and Milne, 1963). When present on soil plant opal, the coatings are indicative of fire. These are to be differentiated from carbon inclusions which probably represent carbon from the original plant sap and impart a darker colour to the plant opal. This is, however, a point about which there is some confusion in the literature (Bowdery, 1989).

The effects of a fire which might influence the amount and rate of ash and charcoal and subsequent litter incorporation into the underlying soil need also to be considered. Clinnick (1984) reports changes to soil porosity amd water repellency, and the possible effects on soil fauna which might be reduced in number for several years subsequent to fire.

Raison, Woods, Jakobsen and Bary (1986), examining the soil temperatures during low-intensity prescribed burning in a *Eucalyptus pauciflora* forest, found that the ash and charred litter initially formed a 2-5 mm layer which was washed into the soil during the first winter after the fire, although they provide no evidence of this. In contrast, Blong, Riley and Crozier (1982) who examined the sediment yield for 12 months after a moderate bush fire, found a large organic component (leaves and charcoal), previously unrecorded, for the first few months after the fire. Burgess, Olive and Reiger (1980) examined erosion after fire in a eucalyptus forest in south-eastern New South Wales and reported suspended sediment loads at least doubled after a fire. Similar figures are found in Teller (1967). This would appear to indicate that the large amount of plant material and the store of plant opal contained within it is lost from the site after a fire.

Erosion after fire in the Sydney Basin has been observed by Mitchell and Humphreys (1987) on hillsopes similar to those of the study area. These are low angle hillslopes with sparse vegetation which are subject to rainwash (a term used by the authors to encompass sheet flow agitated by raindrop impact), particularly after fire. Mitchell and Humphreys discussed the formation of litter dams which form during first runoff events after fire. These are a form of ephemeral microrelief which comprise a contour parallel dam wall of light organic debris behind which sediment accumulates to form a microterrace. Mitchell and Humphreys present a model of litter dam morphology, discussing its role in slope erosion and in the general model of soil formation, particularly of texture contrast soils.

The formation of litter dams in early stages after a wild fire at a site close to the Ecology Reserve (Humphreys and Mitchell's North Ryde site) was described. Immediately after the fire Mitchell and Humphreys observed a fine ash and charcoal coating 2 - 3 cm thick covering the soil, 30% of which consisted of linear shaped, carbonised plant remains. After rain (6 mm estimated at 30 mm/h) litter dams 1-3 cm high of ash and litter formed. Thirty-five days after the fire, strong winds removed the ash, leaving larger charcoal pieces, and destroying most of the first generation dams. The subsequent rainfall events erected second generation litter dams. The micro terraces behind the dams were composed of coarser mineral sediments and fine organic material.

Mitchell and Humphreys commented that in the Sydney Basin water repellence induced by fire is always observed after fires and is of major importance in preventing infiltration. Their observations show that such water repellence lasts for a long period of time - at least for the lifetime of the litter dams. Such a hydrophobic layer between 3 and 120 mm was observed at their North Ryde site, and such water repellent layers have also been observed at other sites. This enables runoff to occur quickly and litter rafting also occurs. Initial runoff was often observed by the authors to be turbid, and under microscope examination proved to contain 64 - 82% silt/clay, including high proportions of platey minerals such as graphite and sericite, minerals which occur in Hawkesbury Sandstone. The absence of these minerals in soil materials on the Hawkesbury Sandstone has been used as an indicator of transported material (Mitchell and Humphreys, 1987). If they are preferentially removed due to their platey nature in suspended loads, it is probable that the exposed platey plant opal in ash is similarly removed.

The consequences of this are far reaching. The standing crop of leaves and undergrowth as well as the litter on the forest floor, is often completely consumed in a fire, and the plant opal it contains is to be found in the ash and charcoal layer. If this layer is then largely washed off in the following rains, the plant opal is lost to the site, probably to be deposited in swamps, flood plains and, ultimately, the ocean. How much this entails is of interest, since it is possible that the amount of plant opal being incorporated into the soil between fires in any area is only a minor proportion of that which is lost due to fire. Thus, for example, calculations of decomposition rates of plant opal in soils which are based on the amount of opal to be found in the plants growing on the soil, may be grossly over-stated.

There are then, several major effects that fire may have on plant opal. Its mineralogy may change under special circumstances where temperatures are high enough for long enough. It may suffer morphological changes; gain carbon coatings, become warped or twisted and, most importantly, it may be exposed to the selective removal of some morphological shapes.

The Study Site

The study site (the Ecology Reserve) is situated between 40 and 50 m above sea level on a 5-6° slope in Hawkesbury Sandstone on the Hornsby Plateau, Marsfield, 12 km north west of Sydney, Australian Universal Grid Reference 256617 on the U0960-7 Pymble Orthophotomap (Figure 6.1). For a full discussion of the Hawkesbury Sandstone landscape, see Chapter 2.

The soil at the site includes earthy sands [terminology of Stace *et al.*, 1968] (Uc4 and 5) [terminology of Northcote, 1974], lithosols (Uc1) and yellow podsolics (Dy) [see Appendix E, soil profile descriptions ER1 to ER3]. Vegetation consists of a woodland of *Eucalyptus* species including *E. haemastoma* and *E. gummifera* with an understory of *Banksia, Acacia, Hakea* and *Grevillea,* and a ground covering including *Pteridium esculentum.*

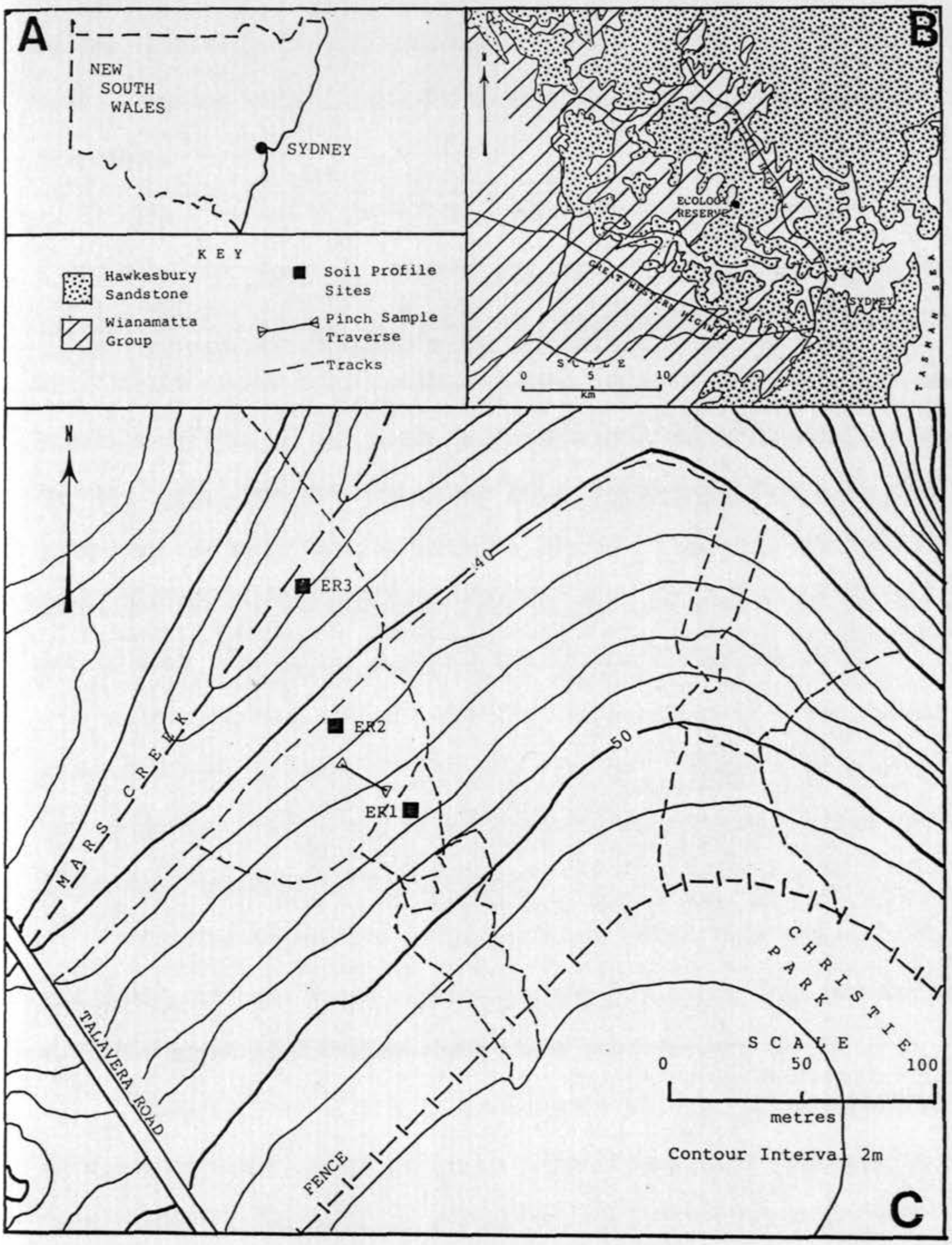


FIGURE 6.1: ECOLOGY RESERVE FIELD SITE
A: N.S.W. SHOWING LOCATION OF SYDNEY
B: GEOLOGY AND LOCATION OF SITE
C: DETAILS OF TOPOGRAPHY AND SAMPLING LOCATIONS

The nearest meteorological station is situated at Macquarie University, 300 m south of the field site. Table 6.1 presents a summary of the rainfall and temperature records for the 10 year period 1981 to 1990. Average annual rainfall is 1211 mm with a summer maximum.

Sampling

The fire was a controlled fuel reduction burn which took place on 1 December 1989 (day 1). Before the fire, six samples of cleaned vegetation were placed in sealed metal cannisters and set on top of the soil in the litter layer under selected large logs in order to examine possible effects on the plant opal contained in the plant tissue upon burning. At these same sites two sets of ceramic tiles with heat sensitive crayon-lines on them were placed to measure the heat of the fire; one set buried standing upright, with an edge flush to the ground and covered with litter, and one set in the litter layer.

The complete litter layer from three sites (2-3 subsamples from each site) was collected before the fire by J. Howell, who, after drying and weighing, kindly made it available to me for examination of the plant opal assemblage in the litter.

Samples of the 0-5 cm topsoil were taken from sites located at the upper, mid and lower slope positions (Site ER1, ER2 and ER3, Figure 6.1) for grain size analysis and plant opal content of the silt.

Observations of the site were made at regular intervals after the fire, particularly after rainfall. Pinch samples of the surface 0-1 cm (see Chapter 7 for details) were taken at 1 m intervals on a 20 m midslope traverse downslope on day 2, day 14 and day 30 after the fire (see figure 6.1). These were analysed for sand:silt:clay:charcoal ratios and the plant opal content of the silt fraction (after combustion) was used to determine the proportion of plant opal in this fraction and to examine the morphology of the plant opal.

Table 6.1: SUMMARY OF RAINFALL AND TEMPERATURE RECORDS FOR THE ECOLOGY RESERVE* OVER THE TEN YEAR PERIOD 1981 TO 1990

Year	Annual Rainfall (mm)	Annual Mean Temperature Maximum deg C	Minimum deg C
1981	983.0	22.5	11.7
1982	718.8	22.8	11.1
1983	1060.4	22.2	11.8
1984	1296.0	21.9	10.9
1985	992.8	21.0	11.2
1986	1129.8	22.2	10.4
1987	1222.6	22.4	11.2
1988	1952.2	22.8	12.2
1989	1262.0	22.0	11.6
1990	2321.0	20.1	11.7
Long Term Average (20 year)	1211.0	22.4	11.3

* Data extracted from Marsfield (Macquarie University) Meteorological Station Records.

The Meteorological Station is situated 300m south of the field site.

Observations and fire characteristics

The fire burned for about 2 hours, with spot fires still found burning in log piles at 07:00 the following day (day 2) when the first set of pinch samples was removed. The tiles and cannisters were retrieved and examined. Combustion was incomplete in most of the cannisters and insufficient to ash any material. On the ground, all litter excepting larger diameter wood was consumed and there was considerable leaf scorch on some trees.

The temperature tiles placed in this and in a similar adjacent experiment were unsuccessful in recording the fire's temperature. However, independent measurements of the temperature of the fire were recorded (T. Auld, pers. comm., 1991). Three sets of AD590 temperature probes were placed on each of three sites (upper, middle and lower slope). The probes recorded to a temperature of 200-250°C. The only probes which went above this temperature were in the lower slope at 1.5 and 2 cm depth. The maximum period of time that this occurred for was 10 - 20 minutes. Thus the requirement that high teperatures be reached and sustained for a long period of time was not reached in these areas.

Meteorological details for the period covering the fire and subsequent sampling are given in Table 6.2. On the day of the fire, a light wind moved particulate material in the smoke to a height of several hundred metres and on the morning after the fire, small wind eddies at the surface were observed to be lifting ash around 2 m into the air and several metres down-wind.

Light rain fell on days 3 and 4, and on the subsequent 9 days, 8 experienced rain, which on day 5 was quite heavy. Details of this storm are given in Table 6.3. On 5 December (day 5), 66.4 mm of rain fell, the period of most intensity being between 02:00 and 03:30, (estimated at 40-50 mm/h). Observations immediately after this rainfall showed

Table 6.2: METEOROLOGICAL OBSERVATIONS FOR DECEMBER 1989
MARSFIELD (MACQUARIE UNIVERSITY) STATION,
NEW SOUTH WALES

Day	Temperature Max	Temperature Min	Rainfall	Evapor ation	Anemo- meter
	deg C	deg C	(mm)	(mm)	at 2m
1	34.*	18.5	0.0	7.0	115
2	34.2	23.*	0.0	)	
3	25.*	17.*	0.8	)23.6	
4	21.0	15.3	0.8	)	546
5	20.7	17.0	66.4	O/F	174
6	22.3	15.7	31.2	6.2	75
7	22.0	16.7	14.8	2.8	182
8	23.*	15.0	3.4	3.0	149
9	24.*	13.0	0.tr	)	
10	27.2	15.*	2.4	)11.4	
11	32.5	15.*	3.4	)	296
12	28.5	20.0	3.6	7.8	121
13	27.0	17.5	0.0	2.4	86
14	26.0	19.5	8.6	5.6	172
15	21.*	13.8	0.0	7.8	174
16	22.2	9.5	0.0	)	
17	22.*	14.*	0.0	) 18.0	
18	27.*	10.*	0.0	)	377
19	26.*	11.5	0.0	)	
20	34.3	15.*	0.0	)	
21	19.*	16.*	0.6	)	
22	23.*	14.*	0.0	)33.6	
23	25.*	13.*	0.0	)	
24	21.*	18.*	0.0	)	
25	29.0	14.*	0.0	)	856
26	36.*	24.*	0.0	)	
27	28.*	18.*	0.0	)	
28	22.*	17.*	0.0	)	
29	24.*	16.*	0.0	) 61.8	
30	27.*	12.*	0.0	)	
31	27.*	13.*	0.0	)	
MEANS	25.8	15.8			
SUM			136.0 on 11 days		

* Temperature from Thermograph

The Meteorological Station is situated 300m south of the field site.

Table 6.3: SUMMARY OF RAINFALL AT THE MARSFIELD (MACQUARIE UNIVERSITY) METEOROLOGICAL STATION ON 5 DECEMBER 1989

Date	Time From	To	Pluviograph (mm)	Gauge (mm)	Remarks
4 December					
	09:00 -	11:00	0.0		
	11:00 -	13:00	0.0		
	13:00 -	15:00	0.0		
	15:00 -	17:00	0.0		
	17:00 -	19:00	0.0		
	19:00 -	21:00	0.0		
	21:00 -	23:00	0.0		
	23:00 -	01:00	0.6		
5 December					
	01:00 -	03:00	39.4		Heaviest from
	03:00 -	05:00	24.2		02:00 - 03:30
	05:00 -	07:00	0.2		
	07:00 -	09:00	2.0	66.4	
	09:00 -	11:00	8.0		
	11:00 -	13:00	10.0		
	13:00 -	15:00	7.4		
	15:00 -	17:00	0.0		
	17:00 -	19:00	0.tr		
	19:00 -	21:00	0.0		
	21:00 -	23:00	0.4		
	23:00 -	01:00	0.tr		
6 December					
	01:00 -	03:00	1.2		
	03:00 -	05:00	0.4		
	05:00 -	07:00	3.6		
	07:00 -	09:00	0.4	31.2	

The Meteorological Station is situated 300m south of the field site.

that litter dams (after Mitchell and Humphreys, 1987) comprising mainly charcoal were forming. Subsequently, these litter dams moved and reformed after rainfall, moving material to a depth of 1 cm (J. Howell, pers. comm.).

Sample processing

(i) Litter samples

The sub-samples from each site were oven dried, mixed, cut up and the plant opal extracted from each of two 1 g samples (see Chapter 7 for extraction details). One sample was used for calculations of percentage plant opal in the litter while the remaining was used for the examination of morphology.

(ii) Ash samples

The 20 subsamples in each pinch sampling were combined, oven dried for 24 h, passed through a -1 phi sieve to remove large materials (mainly charcoal), and each was then divided into three subsamples of 5-10 g. All samples were then ashed in a muffle furnace for 24 hours at 560°C to ash the charcoal (these samples were found on inspection, to have no noticeable mineralogical changes or warping and fusion). The samples were divided into sand, silt and clay, and the silt fraction retained for plant opal extraction (see Chapter 7).

The plant opal retained for examination of morphology was mounted on SEM stubs and glass slides for optical microscopy.

Results

(i) Grain size and amount of plant opal

Initially, several attempts were made to remove the charcoal from the ash in the pinch samples, all of which failed. It was found that, while a large proportion of the larger fragments of charcoal were able to be floated off in water, the smaller pieces would not separate from the ash and other sediment (Plate 33A and B). The results of such an attempt using the day 2 samples are shown in Table 6.4.

Attempts to separate the charcoal in sodium polytungstate also failed. Specific gravity was lowered from 2.3 and at a specific gravity of 1.7, charcoal was still being floated off along with many plant opal pieces (Plate 32). A full discussion of this problem is to be found in Chapter 7.

Ashing the charcoal would have the effect of adding the plant opal it contained to the other material in the sample. Since the prime objective of this study was to examine the fate of all plant opal whether in the ash or in the charcoal, this method was employed. However, the question of whether the addition of the charcoal-bound plant opal to the morphological samples changed their characteristics then had to be addressed.

Table 6.5 shows the weight loss after ashing, and Table 6.6 summarises the grain size analysis and plant opal content of the three sets of samples. The day 2 samples averaged a loss of 28.74% weight, and comprised 52.77% sand, 31.79% silt and 15.44% clay. These figures are not inconsistent with those in Table 6.4. The fine charcoal in these samples appear to have contributed plant opal to the silt and clay fractions on ashing as would be expected, and would seem to indicate a large proportion of the plant opal is bound in the charcoal.

The proportion of the samples lost after 24 hours in the muffle furnace (Table 6.5) was 28.7% in the day 1 sample, rising to 60.7% in the day 14 sample and falling to 11.0% after 30 days. This is reflected in observations of the ground cover after the fire which changed from grey-white ash to largely charcoal which in turn diminished in quantity after 30 days. This loss can be taken to represent the proportion of charcoal present in the samples, while the higher proportion of silt and clay in the day 1 sample would appear to be due to the higher proportion

Table 6.4: INITIAL GRAIN-SIZE ANALYSIS OF DAY 2 SAMPLES

Sample	% sand	% silt	% clay	% charcoal
1	53.1	10.7	14.1	22.1
2	60.3	10.5	10.4	18.8
3	62.7	11.6	5.9	19.8
4	43.3	11.6	29.7	15.9
5	63.9	12.1	6.6	17.4
Mean %	56.7	11.2	13.3	18.8

Table 6.5: WEIGHT LOSS OF PINCH SAMPLES AT 560 DEGREES C

Day after Fire	Sample #	Initial Weight g	Final Weight g	Weight Loss g	% Weight Loss	Mean Weight Loss %
	1	6.40	4.55	1.85	28.93	
Day 2	2	5.56	3.84	1.72	30.92	28.74
	3	5.20	3.83	1.37	26.37	
	1	6.25	2.44	3.81	60.94	
Day 14	2	6.34	2.57	3.77	59.47	60.67
	3	5.54	2.13	3.41	61.60	
	1	13.17	11.74	1.43	10.86	
Day 30	2	10.50	9.25	1.25	11.87	11.04
	3	10.81	9.68	1.12	10.38	

Table 6.6: GRAIN-SIZE OF SAMPLES AFTER 24H AT 560 DEGREES C

Sample	sand %	silt %	clay %	Plant opal % in silt	% silt x Plant opal
Day 2	52.77	31.79	15.44	25	7.95
Day 14	52.25	33.43	14.32	13	4.35
Day 30	73.42	22.53	4.22	6	1.35

Table 6.7: GRAIN-SIZE ANALYSIS OF A1 HORIZON ECOLOGY RESERVE SITES

Soil Profile #	sand %	silt %	clay %	% plant opal in silt	% silt x plant opal
ER1	66.2	14.1	19.7	3.2	0.45
ER2	73.1	11.1	15.8	3.0	0.33
ER3	90.3	4.7	5.0	0.6	0.03

of ash present.

This is consistent with the results obtained by Blong *et al.* (1982) after fire in a similar area. They described a "puffy" surface of charcoal fragments and ash after the fire, and recorded changes in the sediment yield of plots, beginning with a high amount of leaf sediment from leaf fall, and including a high proportion of charcoal which tapered off during the recording period. Similarly, Mitchell and Humphreys (1987) reported a layer of ash and charcoal on their North Ryde site which, after rain, became organised into litter dams and microterraces. While in some instances the ash might become incorporated in such structures, they observed redistribution of the ash and charcoal by winds which left only the larger pieces of charcoal on the surface, and their observations at the sites would tend to confirm that the ash and smaller charcoal fragments are preferentially removed in runoff or wind very soon after fire.

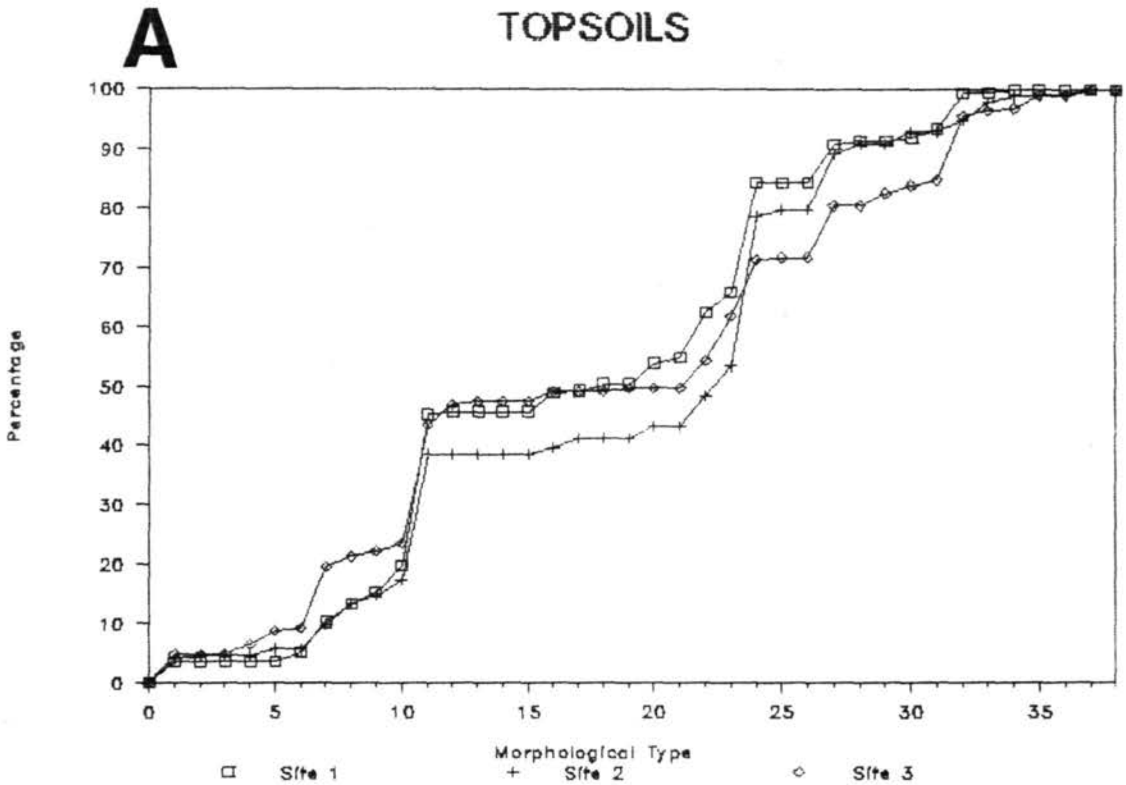
Table 6.6 shows the results of a grain size analysis of the samples after they had been ashed. The day 1 and day 14 samples were very similar in their proportions of sand, silt and clay; the day 30 sample contained a lower proportion of clay, and approached the expected grain size distribution of the A1 of the soil in the mid-slope position (Site ER2) in so far as the sand content is concerned (Table 6.7). The average percentage plant opal extracted from 2 subsamples of silt from each of these samples is also shown in Table 6.6. This clearly shows that the silt content of the day 2 sample contained twice the plant opal of the day 14 sample, and that by day 30 the plant opal content again approached that for the A1 of the soil (Table 6.7). In addition, the clay sized material in both the day 2 and 14 samples may have contained a very high proportion of plant opal which was removed by day 30.

(ii) Morphology

The morphology of the plant opal in the litter, pinch samples both before and after ashing, and topsoil was examined using the SEM and light microscopy. A plant opal assemblage for each sample was erected (Figure 6.2, derived from Table G16). The litter contained 67.9% platey morphological types (this term includes platey rods plus all two-dimensional sheets; single and multi-celled). The day 2 ash (Fig. 6.2C) contained a similar proportion (66%) of platey plant opal (Plate 34A), while by day 14 this had declined to 20.1% (Plate 34B) and by day 30 to 13.5% (Plate 35). In the topsoil (Fig. 6.2A), the proportion ranged from 13.7% at Site ER1 to 5.2% at Site ER3 (Plate 31 and 36).

Figure 6.2B compares the litter assemblage with the topsoils, while Figure 6.2D compares it with the ash. A Kolmogorov-Smirnov two-tail test (Table 6.8, derived from Table G17) compares all assemblages. The plant opal assemblages from the topsoils downslope were very similar; however, as was found in the Pilliga studies, the litter was different from all three topsoils, particularly in the platey material and rods. The day 2 ash was very different from both the day 14 and the day 30. Day 14 ash was, however, similar to day 30. Thus the major changes to the plant opal assemblage in the ash occurred between days 2 and 14, probably at the time of the major rainstorm. The litter was different from all 3 ash samples, although nearest in characteristics to that of day 2 as would be expected.

The largest differences between the ash assemblages were in the amount of rods and platey material. Between the litter and day 2 ash the differences were in the amounts of hairs and prickles, while between the litter and days 14 and 30 ash the amounts of rods and platey material were most different. The litter differed from the topsoil mainly in thin rods, platey material and hairs and prickles. These results were



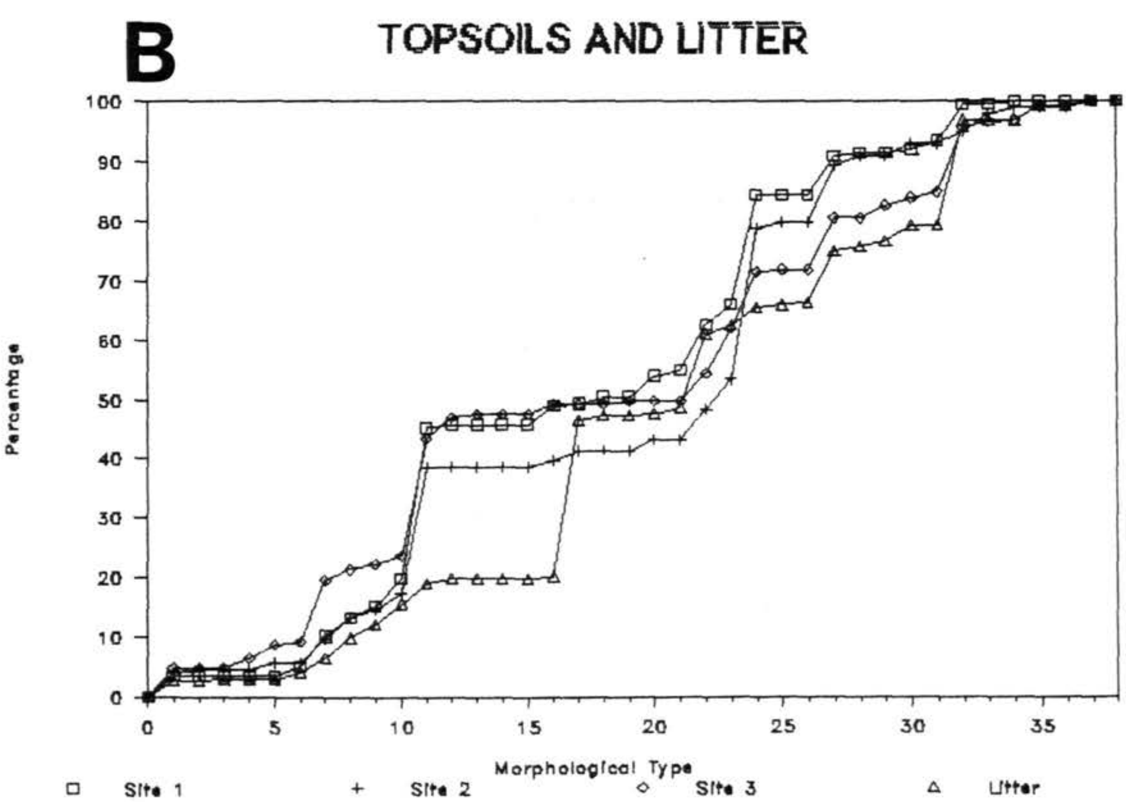
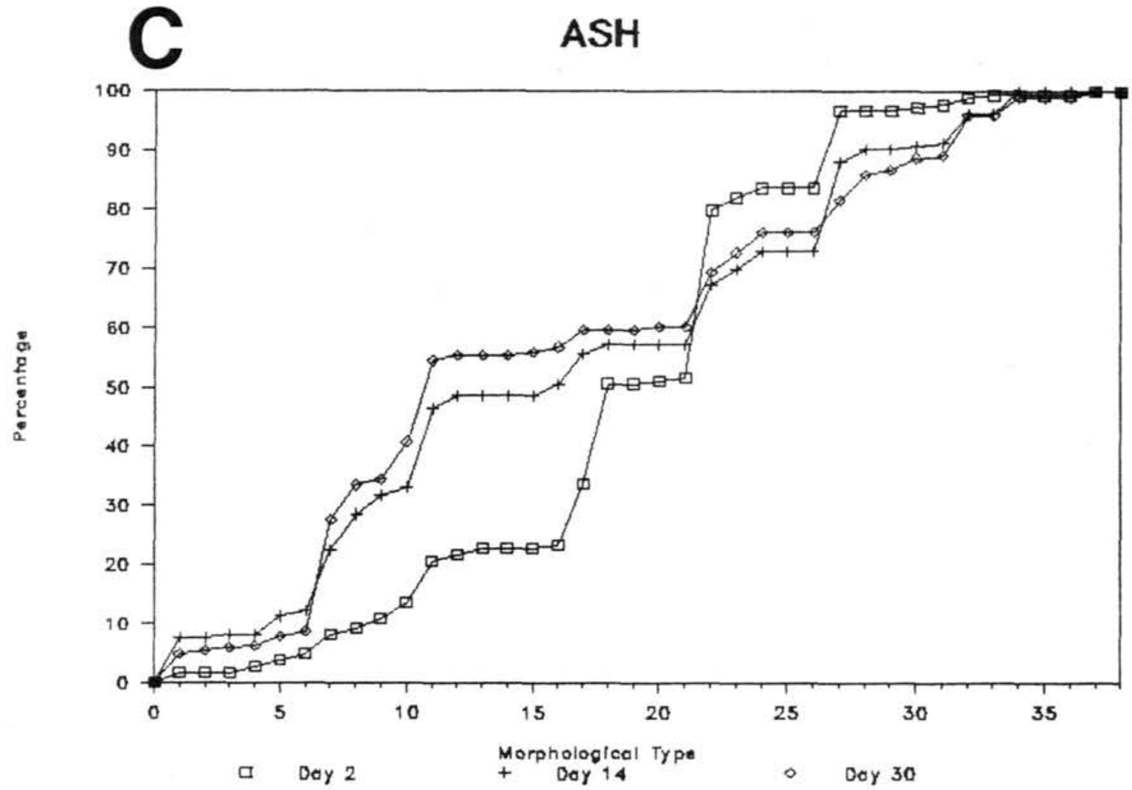


FIGURE 6.2: ECOLOGY RESERVE PLANT OPAL ASSEMBLAGES



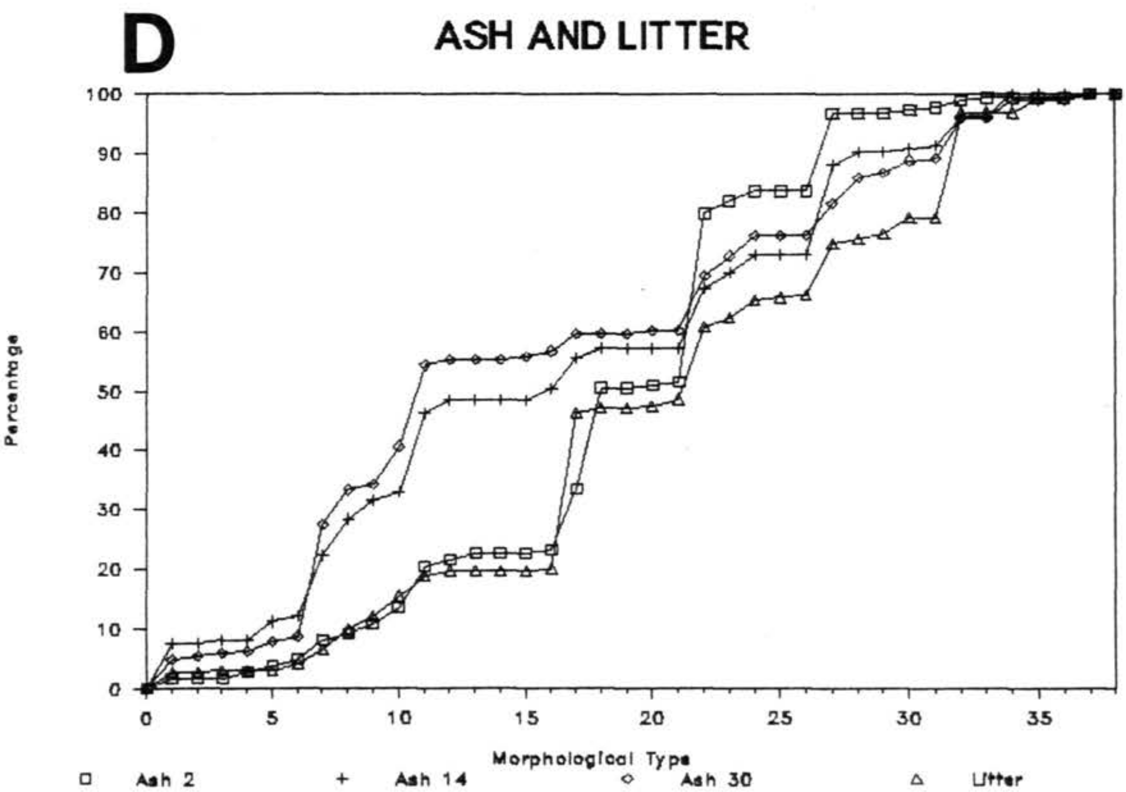


FIGURE 6.2: ECOLOGY RESERVE PLANT OPAL ASSEMBLAGES CONT'D

Table 6.8: KOLMOGOROV-SMIRNOV TWO TAIL TEST RESULTS: ECOLOGY RESERVE

Comparisons	D	Critical value a=0.05
Ash		
Day 2 & Day 14	0.27	0.14*
Day 2 & Day 30	0.34	0.14*
Day 14 & Day 30	0.08	0.14
Topsoil		
Site 1 & Site 2	0.14	0.14
Site 1 & Site 3	0.13	0.13
Site 2 & Site 3	0.10	0.13
Litter & Ash		
Litter & Day 2	0.22	0.13*
Litter & Day 14	0.30	0.13*
Litter & Day 30	0.37	0.13*
Litter & Topsoil		
Litter & Site 1	0.29	0.13*
Litter & Site 2	0.20	0.13*
Litter & Site 3	0.29	0.12*

* not drawn from same population

similar to those found in the Pilliga sites.

This disappearance of platey plant opal could be due to the plant opal breaking up, or to its removal from the system. The plates in the samples varied from large sheets containing many silicified cells (stomatal cells, epidermal cells, etc.) to smaller sheets which were perhaps only one cell or part of a cell wall (Plate 33). All were <2 um thick. It appeared that the plates were both becoming smaller and fewer through time between the litter and ash samples.

The characteristics of platey plant opal

In order to examine the fate of platey and rod-like material after leaf-fall and before incorporation into the soil after a fire, a count was made of 110 pieces of plant opal for each sample (litter and ash), recording its morphological type, whether it was multicelled or part of a single cell, its percentage carbon coating and its surface area if a plate and length if a rod.

A similar proportion amounting to over 60% of both the litter and the day 2 ash samples comprised platey plant opal (Table 6.9). However, a Students' t-test on the average surface area (d.f.= 142 at 5% level) showed that the underlying populations from which the samples were drawn were not the same. The day 2 ash consisted of the litter layer which was in place before the fire, plus contributions from the burnt canopy layer and the herb layer. Thus the litter layer plant opal morphology may not be completely representative of the material burned during the fire. However, assuming that the surface area of the additional material was similar to that already in the litter layer, the reduction in the mean surface area of the plates would indicate that there was considerable breakup of the platey plant opal during the fire. This could be compounded by the effects of the ashing in the muffle oven, although inspection suggests that this effect is minor (cf. sheets Plate

Table 6.9: PLATEY PLANT OPAL CHARACTERISTICS IN THE ECOLOGY RESERVE SAMPLES

Sample	% platey	multicelled (% of platey)	surface area of plates sq um	
	n=110		mean	sd
Litter	66.4	58.9	370,570	612,340
Day 2	64.5	87.3	56,440	55,640
Day 14	15.5	87.5	52,610	44,020
Day 30	8.2	66.7	31,190	25,860

Sample	% platey rods	length of rods & hairs um			% black inclusions
	n=110	n	mean	sd	n=110
Litter	2.7	7	259	100	0.0
Day 2	1.8	21	473	288	49.1
Day 14	1.8	52	317	209	7.3
Day 30	0.0	49	256	157	0.0

33A, B and Plate 34).

If larger, multicelled material broke up, it would be expected that a larger proportion of the plates would be smaller, single or part-celled plates and platey rods. Table 6.9 indicates that this is not the case. However, multicelled means any plate having more than one cell - in the case of the litter, the multicelled plates were very large indeed, as can be seen by their surface area distribution. Thus, breaking up of these cells would simply provide, in many cases, more multicelled plates. It could, then, be argued that break up would provide more, not less, multicelled plates in the day 2 ash; and indeed, the proportion of these plates rose 28%.

Large amounts of particulate material are removed in smoke (Khanna and Raison, 1986), and this was also observed during the fire. It would appear, from these observations, that a large number of the more fragile platey opal pieces in the ash are removed in this manner. Logically, the smaller exposed platey material would be more susceptible to removal in smoke. The increase in the smaller size range is then consistent with removal of smaller sizes during the fire and the break up of the larger pieces. Thus two processes are important in modifying the plant opal morphology during a fire - removal in the smoke and break up of exposed opal.

Processes modifying the assemblages after fire

After the first heavy rain most of the ash was observed to have di~~s~~sappeared from the hillslope. The percentage of plates remaining had declined to 15.5% but proportion of those being multicelled plates remained the same (Table 6.9). If the plates had simply broken up during this period, there would be a increase in the proportion of smaller multicelled plates in the day 14 sample as well as an overall increase in plates and platey rods and/or and increase in clay sized material. This is not the case. Table 6.6 shows no increase in clay sized material between the day 2 and day 14 samples, while Table 6.9 shows no increase in the proportion of multicelled plates. This would seem to indicate that the plates were removed during rain or in wind, and indeed, this is confirmed by the fact that the mean surface area of the plates remaining after the rain was not significantly different from that before the rain (Students' t-test, d.f.= 85, 5% level).

Between day 14 and day 30 the percentage of plates decreased to 8.2%, while the proportion of multicelled plates and the mean surface area of the plates decreased. This would indicate further break up of the material. During this period only 9.2 mm rain fell, which may not have been sufficient to initiate movement of the remaining material but the area experienced high evaporation and winds which may have contributed to the removal of some material. A slow down of movement could also be expected from the influence of plant growth at this time. The first seeds germinated 2-3 weeks after the fire (J. Howell, pers. comm.).

The proportion of rods fell between the litter and the ash. Between the day 2 and day 14 samples breakup of platey material into rods appears to be balanced by the removal of such material. The increase in length of rods and hairs (as well as the number available for counting) between the litter and the day 2 ash may be due to additions of extra leaf material during the fire contributing fresh, unbroken material.

Additional observations

Carbon coatings were observed on many of the particles of plant opal in the pinch sample from day 2 (Plate 34A), while fewer were to be found by day 14 (Plate 34B), and none were present in the day 30 sample

(Table 6.9), [Plate 35]. The main morphological class coated in the day 2 sample were the 2 dimensional sheets (plates), while in the day 14 sample the remaining coatings were mainly on more robust forms - rods and 3 dimensional sheets. Coatings are due to the incomplete combustion of carbon (Jones and Milne, 1963). No warping or twisting of plant opal was observed in any sample.

Summary of results

1. No evidence was found in the samples examined that the temperature of this fire was high enough to produce mineralogical or morphological changes in the plant opal apart from carbon coatings.
2. Results did, however, demonstrate the selective removal of platey plant opal fragments after a fire as well as the breakup of multicelled particles.

Conclusions

Litter on the hillslope contained an average plant opal content of 0.73%. This amount may be represented by the plant opal contained in the day 2 sample, which can be measured in the silt size range only (Table 6.6). In the day 2 sample 25% of the silt fraction comprised plant opal (7.95% of the total sample calculated by the % silt x % opal method). By day 14, this had been reduced to 13% of the silt fraction (4.35%), and by day 30 to 6% (1.35%). The reduction in total plant opal between day 2 and day 14 is 45.3% and by day 30 is 83%. Thus, remembering that most plant opal is to be found in the clay fraction, the amount of opal available to the soil on day 14 is 0.4% and on day 30 this is a mere 0.1% of the dry weight of the original litter.

These figures can only represent an order of magnitude, since we cannot tell what is happening to clay sized plant opal; however, it does demonstrate that, in an area where fire is an integral part of the environmental system, its effects on the plant opal cycle are considerable.

In a similar area to the Ecology Reserve Site, in Hawkesbury Sandstone at Narrabeen Lagoon, Lamb (1985) recorded accumulated litter masses of 2100 and 1900 gm^{-2} in areas unburnt for 9 years which he considered had reached equilibrium. Given that 0.7% of this litter is plant opal (14.7 and 13.3 gm^{-2}), after a fire in such an area the amount of plant opal available for incorporation into the soil is only of the order of 2.1 to 1.9 gm^{-2}.

Birk and Simpson (1980) examined litter accumulation and decomposition in periodically burned Jarra and Kauri eucalyptus forests, finding that if the natural rate of decomposition was slow, frequent fires would remove much of the total litter falling between fires. In Jarrah, if 50% of the forest floor burned after seven years of fuel accumulation, 22% of the total seven year input would be consumed. If all of the forest floor burned, 44% of the total input would be removed. In a Karri forest the figures after seven years accumulation would be 32% for a 50% forest floor burn. While these figures were used by the authors to point to the problems in regular hazard reduction fires, they also serve to illustrate the immense potential for the removal of particulate silica.

In the period between fire, normal incorporation processes by soil fauna can remove a great deal of litter. At equilibrium in the Pilliga State Forest sites, for example, between 1 and 3 g of plant opal is being incorporated into the soil each year. Given a litter half-life of 3 to 4 years, the amount of plant opal to be removed during a fire is considerable.

Deposition of the plant opal

The plant opal lost from the hillslope may either be transported away in creeks or deposited further downslope in depositional areas such as floodplains, lakes and swamps. There are indications in the case of the Ecology Reserve that some of the opal from the catchment is deposited in the flood plain.

Distribution of plant opal in Site ER3

The distribution of plant opal in the soil at Site ER3, situated in the floodplain of Mars Creek, was examined. The location of this soil (ER3) is shown on Figure 6.1. It was on the flood plain of Mars Creek below the road crossing, and the vegetation in the immediate area was mainly tall grasses. The soil comprised a surface layer of very coarse sand mixed with blue metal from the road and contained a large amount of charcoal. Below this was a series of layers which became more clayey with depth. A full soil description is given in Appendix B (profiles ER) and an analysis of the particle size distribution of each layer is given in Table G3, Appendix G).

Material and Methods

Plant opal was extracted from the silt fraction of samples taken at 100 mm, 250 mm, 600 mm, and 1000 mm depth using the method outlined in Chapter 7. The resultant proportion of opal (corrected for purity) was multiplied by the proportion of silt in each sample and graphed (Figure 6.3A). Examples of the plant opal found in the samples are shown in Plate 36A and B. The amount of clay in each sample is also graphed (Figure 6.3B).

Results

Figure 6.3A shows that the amount of opal in the silt rises to a maximum at 600 mm depth and falls again at 1000 mm. Since it is believed that a very large proportion of the plant opal is to be found

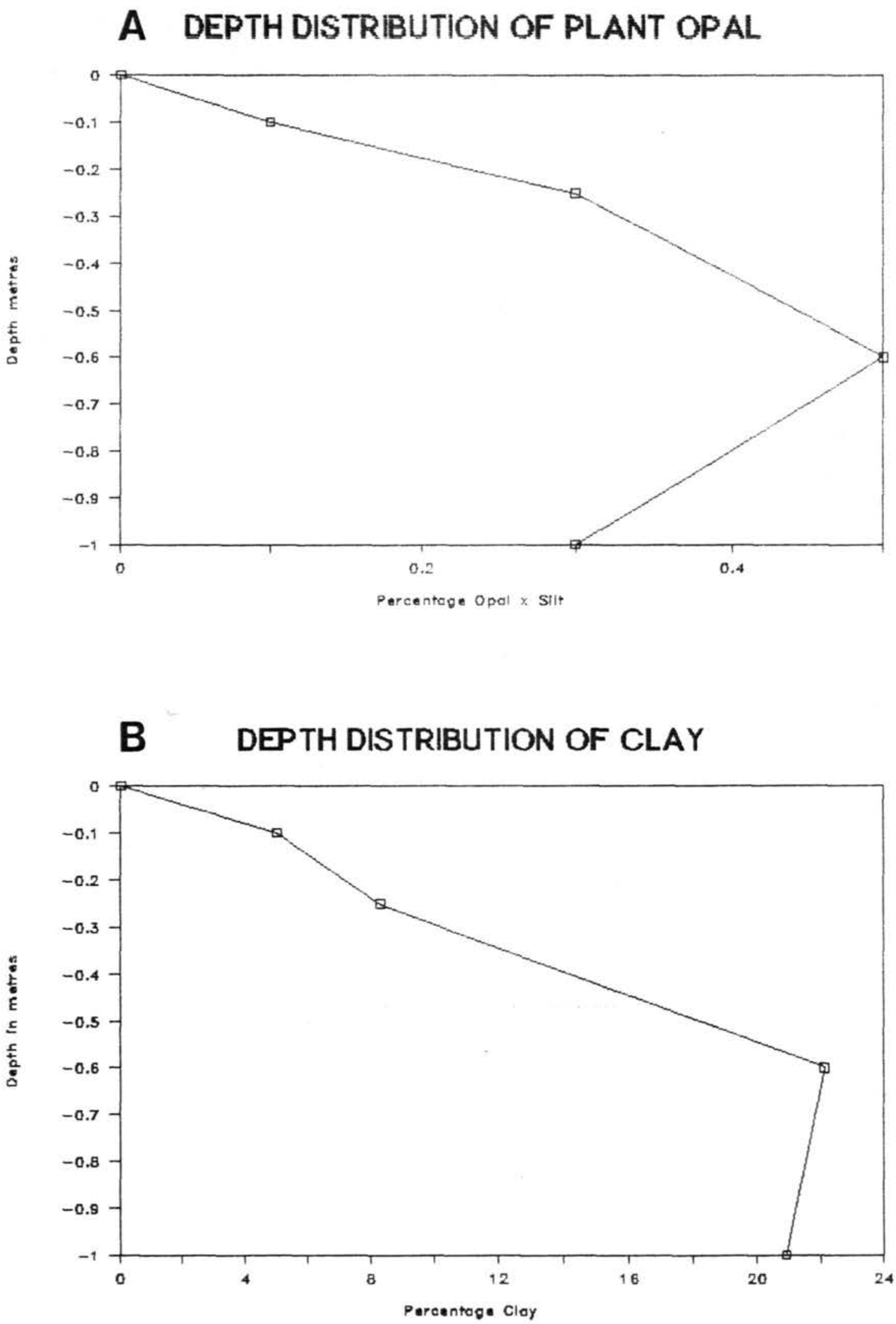


FIGURE 6.3: DEPTH DISTRIBUTION OF PLANT OPAL AND CLAY, ECOLOGY RESERVE PROFILE ER3

in the clay, it is interesting to see that the percentage clay in the samples similarly rises to a maximum at 600 mm. Thus these graphs show that the maximum amount of plant opal is indeed to be found at a considerable depth in this soil, contrary to expectations.

Discussion

This soil is better thought of as a series of layers deposited overbank by the creek in times of flood. The catchment is gradually becoming more built up as the area which the small Mars Creek drains changes from bush to orchards to urban in use. In particular, the Creek drains the area on which Macquarie University now stands. The road above the sampling point was paved about 12 to 13 years ago, and the blue metal used to pave it is found to about a depth of 200 mm. It is tempting to thus ascribe an accumulation rate of around 16 mm per year, thus placing the plant opal maximum as having occurred some 30 to 40 years ago during a period when the catchment was still largely in orchards and grass.

Summary of results

The topsoil contained only a small percentage of plant opal (Table G3); however, lower in this soil the percentage of both silt (15 - 17%) and the plant opal content of the silt (2-3%) rose considerably indicating a layering of material alternatively rich and poor in plant opal may be common in these conditions.

In addition, the plant opal which is in the clay size range is more soluble in water and it may contribute directly to the soluble silica in the soil. Khanna and Raison (1986) for example, reported increased concentrations of soluble silica in soil under an ash bed derived from forest burning which remained very high for at least three years. They reported high concentrations of silica in ash, but did not discuss the source of the soil concentration.

Conclusions

During and immediately after a fire a large amount of plant opal is removed, firstly in the smoke and secondly in wind and rainwash. The amount being removed may vary, depending on the intensity of the fire, the time since the last fire, and rainfall/wind intensity and timing after the fire. Selective removal of large, platey sheets appears possible during this time, changing the plant opal assemblage remaining.

In the observed fire, an 83% reduction of plant opal present in the litter awaiting incorporation into the soil, was found. Some of this opal may be deposited in the flood plain of the creek draining the catchment; much will be lost from the catchment.

In this chapter, fire has been indicated as one of the processes which is capable of generating a difference between the litter and topsoil plant opal assemblages. The use of plant opal extracted from litter to erect modern vegetation assemblages should then be approached with caution, since fire may considerably alter the assemblages in areas where it is common, particularly if the fire has been recent. A major problem also occurrs where soil age estimations have been based on calculations of the amount of plant opal present in litter or the standing crop of vegetation (e.g. Jones and Beaver, 1964 and, more recently, Kondo, 1988). In areas where fire is or has been common, such calculations, which at best are highly suspect, are even less accurate.

PART 2

LABORATORY AND FIELD TECHNIQUES

"A precise statement can be more easily refuted than a vague one, and it can therefore be better tested. This consideration also allows us to explain the demand that qualitative statements should if possible be replaced by quantitative ones by our principle of increasing the degree of testability of our theories. (In this way we can also explain the part played by *measurement* in the testing of theories; it is a device which becomes increasingly important in the course of scientific progress, but which should not be used [as it often is] as a characterizing feature of science, or the formation of theories, in general......" Popper, 1972: 356.

CHAPTER 7: SAMPLE PREPARATION AND ANALYSIS

Introduction

Part 1 was concerned with field observations and the laboratory analyses which supported or amplified those observations. In Part 2 the techniques used in the analyses are discussed.

This chapter examines the new and amended techniques which were used in this thesis for the sample preparation and analysis of plant opal. In particular, it discusses the problems involved in current methods of heavy liquid analysis and methods which may overcome these problems. It discusses the use of a new heavy liquid and outlines some of the new directions being taken to analyse opal. A brief review of sample preparation and analysis techniques and the methods used in this thesis which are not new, are covered in Appendix F.

Sample preparation techniques make use of the mineralogical, chemical and physical characteristics of plant opal. It is isotropic with a refractive index of 1.427-1.487, specific gravity of between 2.00 and 2.28 and a surface area of 14.4 sq.m/g (Jones and Milne, 1963). Much of the present utilisation of plant opal in paleoenvironmental and archeological work pre-suposes that the chemical and physical properties of plant opal are well understood, and this is not the case. Recognition of plant opal in sediments, dissolution rates in sediments, maturation, etc., have all been considered to some extent, but studies in these areas often throw up more questions than they answer. The very extraction of plant opal from both plants and sediments poses problems which, if left unresolved, must surely present difficulties when any morphological work is undertaken. Yet these appear to be unrecognised or overlooked by many researchers.

Extraction from vegetation

Two general methods of extraction of plant opal from vegetation have been common; usually referred to as "wet-way" and "dry-way". Early workers obtained plant silica by ashing the plant (dry-way) and examining the residue, the "ash picture", "spodogram" or "silica skeleton" (utilised for example by Lanning *et al.*, 1958; Lanning, 1960, 1961). The plant material is macerated with bleach or dilute hydrochloric acid, rinsed and ashed in a muffle furnace at 450-900°C for 6-12 hours. The ash residue is then washed in dilute hydrochloric acid, hydrogen peroxide or water and mounted on a slide using Canada Balsam or similar fixitive. If the opal is to be viewed in place, the final rinsing is shortened to reduce disturbance.

A recent development in the preparation of spodograms is that of Lanning (1980). The plant material is ashed between two glass slides. After ashing, one slide is removed and replaced with a cover slip after Canada Balsam has been added. This method gives an undisturbed sample. There are other refinements to the method which have been developed by individual workers; for example, Bowdery (1984) reports that Fujiwara crushes the ash to a fine powder, sonicates for 15 minutes then decants in water.

There are, however, problems with spodograms or dry ashing. The disadvantages of this method include the incorporation of larger quantities of accessory elements, dehydration, fusion of material and blurring of detail, warping and twisting, some crystallisation to a disordered cristobalite and the closing of pore systems resulting in a lower surface area when compared with plant opal obtained by wet-way methods (Jones and Milne, 1963). Pearsall (1979) found the dimensions of maize phytoliths shifted to a smaller size category on ashing. Thus dry-way methods produce chemical and physical changes in the silica of the plant often leading to mis-interpretation. As late as 1974, however, dry-way ashing was still being used to check for crystalline "artifacts" by Wilding and Drees who noted no differences in optical or diffraction analyses of tree leaf isolates thus prepared when compared with wet-way. They did find evidence of sub-microscopic crystallites of cristobalite in low order arrangement in X-ray diffraction analysis (XRD) of isolates prepared by both methods, and concluded that both cristobalite and alpha-quartz was synthesized in vegetation tissue. In view of the overwhelming published evidence to the contrary, this is a point which needs re-examining in the particular species used, since contamination can not be ruled out.

In the wet-way method, the surrounding plant material is digested by acids and oxidised. Various acids and oxidising agents have been used: Jones and Milne (1963) used nitric and perchloric acids; Geis (1973) used sulphuric acid and hydrogen peroxide. Both Geis and Lewin and Riemann (1969) commented on the dehydrating effects of concentrated sulphuric acid. Rovner (1971) recommended that for safety reasons a nitric-perchloric mixture should not be used, and recommended the use of Schulze solution. It is considered that the wet-way method separates out of plant material phytoliths which are essentially similar to those found in the plants.

Problems often encountered include tough or waxy plant tissues which do not readily digest and require pre-treatment by boiling or soaking in acetone or chloral hydrate solution (Pearsall, 1989).

The method used in the extraction of plant opal from plants in this thesis was maceration in concentrated sulphuric acid followed by oxidation in chromium tri-oxide. The length of time in sulphuric acid was kept to a minimum and no dehydration effects were noted. This method was found adequate for all plant and litter samples, and is outlined fully in Appendix F.

Extraction from sediments

While the separation of opal from plant tissues is relatively simple, separation from sediments is not. The clay fraction is believed to contain the majority of the plant opal (Wilding and Drees, 1974), and has a tendency to flocculate; thus separation from the whole sediment sample is increasingly difficult with increasing clay content.

In the sand to fine silt range heavy liquid separation of plant opal from soil material is used, making use of the low specific gravity of plant opal (1.5-2.3). The general method is to separate out the size fraction by the usual fractionation methods, remove any organic material using peroxide or by heating in chromic acid, removing salts, etc., and dispersing the soil by the use of Calgon (i.e. Wilding, 1967) or a 9% polyvinylpyrrolidone solution in ethanol (i.e. Wilding and Drees, 1974). The opaline material is then separated by the usual heavy liquid methods utilising a heavy liquid such as nitro-benzene bromoform at a specific gravity (SG) of 2.3.

Since the problems in the use of heavy liquids are many (expense, dangerous to health, difficult to reclaim, etc.), simple techniques involving floatation have been employed (Powers and Gilbertson, 1987; Hart, 1988a). These techniques are based on the platey nature of many plant opal morphologies and have been used with tracer grains by Powers and Gilbertson to provide absolute frequencies of phytoliths.

Two methods were used during the course of work on this thesis to concentrate plant opal in sediments; a simple flotation method and flotation using heavy liquids. The following steps are common to both methods:

(i) The sample of sediment is dried at a temperature of 80°C (this temperature is used for all drying unless otherwise stated). It is then passed through a -1 phi (2.00 mm) sieve to remove coarse sediments and organic fragments. A sample of about 20 - 30 g is then weighed out into a 500 mL clean glass beaker. Distilled water is used at all times.

(ii) About 200 mL 10% hydrogen peroxide is added to remove organic material. The mixture is left overnight then heated to remove most of the liquid. None of the sediments used contained appreciable carbonates, thus the extra step to remove these by adding hydrochloric acid was not necessary. It could be added here or after step (vi).

(iii) The sediment is then disaggregated using 10% Calgon (sodium hexametaphosphate) and left standing for 24 hours. If the sediment is particularly aggregated, a shaker is used to facilitate disaggregation.

(iv) The sand is then separated from the sediment by wet sieving with a 4 phi (62.5 um) sieve, then dried, weighed and stored.

(v) The remaining sediment is placed in a 1000 mL cylinder for decantation. In special cases the cylinder was placed in a constant temperature water bath and the fine fraction was analysed by pipette (Folk, 1980). Usually, however, the object is to remove the small amount of clay present in the samples to prevent flocculation problems. All material smaller than 8 phi or 3.9 um is removed, leaving a silt sample containing particles in the 4 - 8 phi size range (silt). The cylinder is filled with distilled water, stirred to distribute all of the sediment evenly throughout the column, and left for 1h 51m. At the end of this time, all particles coarser than 8 phi will have settled to a depth of at least 10 cm, and the material in the top 10 cm of the water column is siphoned off. This is repeated until the water in the top 10 cm of the column is clear after 1h 51m - this usually meant up to 15 repetitions. The siphoned material may be evaporated to dryness and weighed if an accurate estimation of size distribution is required; however, in cases where the silt sized material is required for morphological work, this step is omitted.

(vi) The remaining silt is placed in a beaker and most of the water evaporated off. It was found that the silt could be dried at 80°C without becoming caked, although in some cases acetone was used to complete the drying process.

(vii) The silt is then divided into weighed samples of about 1g for plant opal removal.

1. Simple flotation method

This method was used in the work on the Oxford Falls swamp. A sample of silt is shaken in distilled water, then allowed to stand for around 30 seconds to allow the quartz to settle. The plant opal suspended in the water is then poured off, left to stand overnight, most of the water carefully poured off and the remainder stored.

2. Flotation using heavy liquids.

(a) General Method

(i) The heavy liquid is prepared to a Specific Gravity of 2.3 (see below).

(ii) A weighed sample of silt (about 1 g) is placed in a 10 mL centrifuge tube, the heavy liquid added and the lid put on. It was found that if the tubes were then shaken several times and left overnight, problems with the silt not wetting up adequately and clumping were avoided.

(iii) The tubes are then placed in a Clements GS 200 centrifuge at 2,000 r.p.m. for 15 minutes. The light fraction containing the plant opal is then carefully pipetted off and placed in another centrifuge tube. More heavy liquid is added to the original sample and the whole process is repeated untill the yield is negligible.

(iv) Distilled water is added to the collected light fraction which is then centrifuged so that the plant opal is retained in the bottom of the tube. The centrifuging is repeated until the plant opal is clean (this may take 10-12 repeats). The plant opal is then stored in a glass or plastic vial in distilled water.

(v) The heavy liquid/water mix is reclaimed (see below).

(b) Heavy liquids used

(i) Zinc Bromide

Initially zinc bromide was used as the heavy liquid in this study since it is less risky to health than bromoform. To 400 g of zinc bromide in a clean beaker, 80 mL of distilled water is slowly added, stirring all the time. When the powder is completely dissolved, the specific gravity (SG) is tested by the use of a hydrometer and adjusted to 2.3 by the addition of either water or zinc bromide. The heavy liquid must be covered since it absorbs water. It can be recycled by evaporating to the desired SG.

While zinc bromide is not as deleterious to health as other heavy liquids, it does present other problems. It is acidic and zinc precipitates may form, particularly if it is added to water. This is initially avoided by carefully adding the water to the zinc bromide. The presence of carbonates in a sample is a problem unless the carbonates are neutralised by the addition of hydrochloric acid. It may then be necessary to raise the pH of the sediment by addition of sodium hydroxide.

(ii) Sodium Polytungstate

Sodium polytungstate is the generic name for an inorganic salt sodium metatungstate (SOMETU, West Germany). It has been used by paleontolgists successfully for separation, and was used for the heavy liquid flotation in this thesis from 1987 onwards (Hart, 1988b). Tungsten compounds are considered non-toxic, and the manufacturers claim that if it is accidentally ingested "all is secreted within twenty four hours."

To prepare a heavy liquid having a SG of 2.3, 722.5 g of sodium polytungstate is mixed with 277.5 g distilled water. The SG is adjusted as in method (i), and recovery is also by evaporation. The heavy liquid tends to loss water readily, and must be kept covered.

The two heavy liquids used in this thesis were compared to see if there was any difference in their ability to separate plant opal from the sediments. Subsamples of a sediment were prepared and treated with zinc bromide or sodium polytungstate. Slides of the resultant plant opal were prepared, plant opal counted and results compared.

Four slides were prepared from each treatment. Two hundred grains were counted on each, and the proportion of plant opal determined. The sodium polytungstate separation yielded an average of 170.00 grains of plant opal over four slides (s.d. = 10.52), while the zinc bromide yielded an average of 177.25 grains over four slides (s.d. 11.79). It was found that there was no difference between the two methods in efficiency of plant opal separation (Student's *t*-test, d.f.=3, a=0.05). This comparison is reported in Hart (1988b), a copy of which is in Appendix H.

Problems

The separation of sediment for plant opal analysis is based on Stokes Law for the velocity of a falling sphere. Many assumptions are made in so doing, probably the most significant being that the bodies being separated are assumed to be spheres, and this is not so.

Plant opal comes in many shapes, many of which are platey and/or elongated; thus Stokes Law is not applicable. The problems in normal sediment analysis using Stokes Law have often been pointed out.

> "From the foregoing discussion it appears that mechanical analysis today has, in a very broad way, its uses: the best that can be said for it is that it is better than nothing." Boswell, 1961:9.

Boswell also pointed out that results using a particular method should only be compared if the analysis has been done by the one worker from the one laboratory.

In an examination of the physical aspects of pollen processing methods, Jemmett and Owen (1990) found a lack of consistency in results from parallel samples. Losses of pollen were recorded during the decanting of supernatants and they cautioned "against strict adherence to a preparation "recipe" without monitoring possible pollen losses at different stages" (1990:205).

The blind adherence to a "recipe" is probably a problem in plant opal processing too. The published methods of previous workers appear to be followed without much thought as to what is being done to the sample and the possible effects this might have. The reproducibility of results needs examination, as the problems found by Jemmett and Owen in pollen processing - that of lack of consistency between parallel samples, differential losses during decanting supernatants and the problems of pollen morphology seem to be echoed in the processing of plant opal.

In order to examine some of these problems in plant opal processing, the material discarded at several points during processing was examined. Figure 7.1 shows the outline of processes samples are

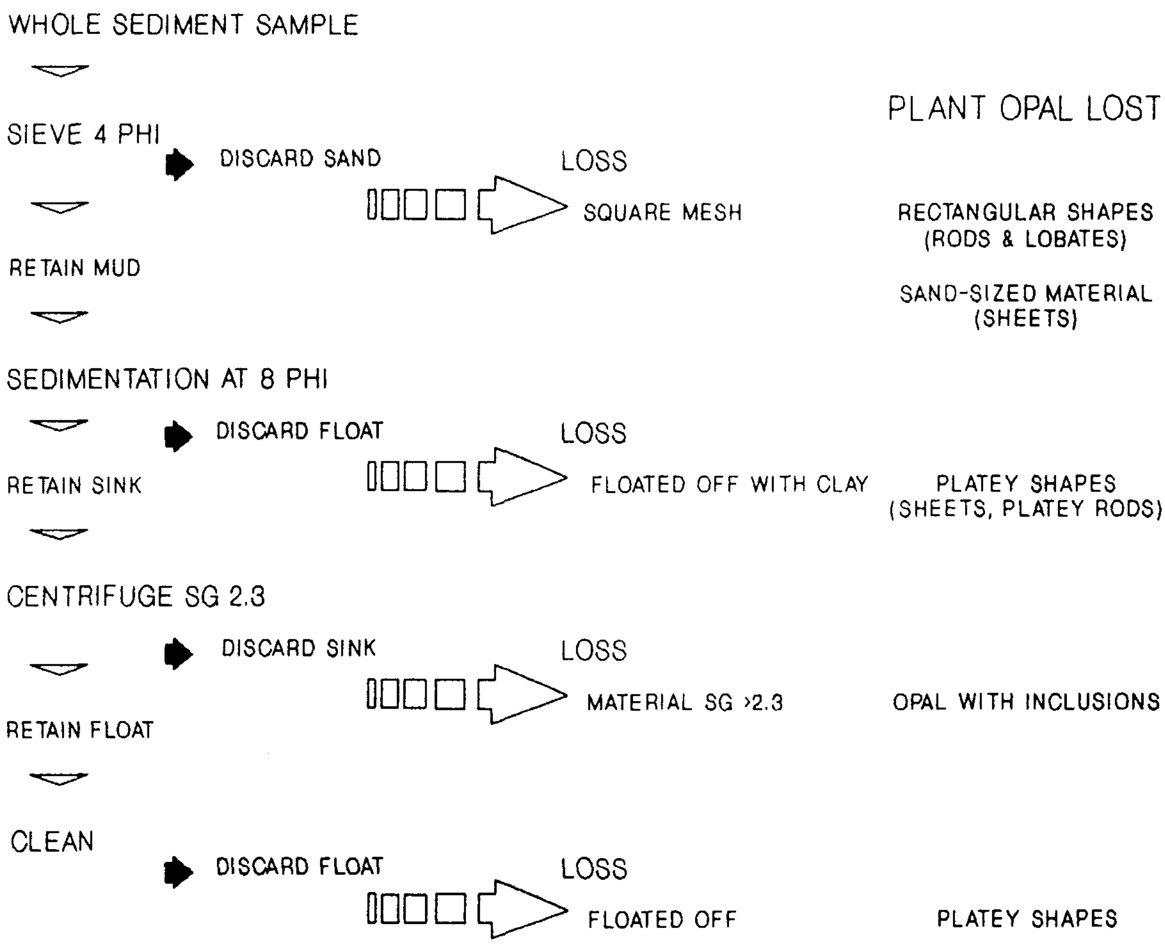


FIGURE 7.1: PLANT OPAL SEPARATION FLOW CHART STAGES AT WHICH PLANT OPAL MAY BE LOST

subjected to, and the places where there is potential for plant opal loss.

Study 1: The loss of plant opal during separation by heavy liquid

Method and Materials

During the separation of plant opal from pinch samples taken from the Site 6 broom plain (Pilliga), samples were retained at four places during the processing where there could be loss of plant opal of the size range being concentrated (i.e. the 4 - 8 phi or silt size). These were:-

1. During the wet seiving of the whole sample at 4 phi to remove the sand-size range. The sand at this stage was examined for smaller than 4 phi diameter plant opal particles.

2. During the sedimentation of the sample to remove the clay size particles. The material siphoned off was retained and examined for particles larger than 8 phi in diameter.

3. During separation by heavy liquid the sink comprises that material which has a SG greater than 2.3. This material was examined for opaline particles.

4. When the separated opal is cleaned, the supernatant is poured off. This was examined for opaline material.

In each case the material was retained and a subsample of it mounted for light microscopy as discussed below.

Results

1. Sand sized material

It was found that there were very few particles smaller than the 4 phi diameter. Over 1% of the sample was plant opal, mostly large multicelled plates (Plate 44A and B).

2. Clay sized material

This material was too small to count, however, as can be seen from Plate 44C, it contained many small pieces of opal.

3. Silt material with SG>2.3 (the sink)

The silt had no opaline material, indicating that the separation of this sample at this SG is successful.

4. Supernatant from cleaning separated opal.

A large amount of opal was present in this normally discarded water (Plate 44F), pointing to problems similar to those discussed by Jemmett and Owen (1990). While most of the opal was of the platey morphology there were morphologies present which might affect counts in assemblage work, thus it will be necessary, in future, to examine the causes of this problem.

Conclusions

In their work on pollen losses, Jemmet and Owen observed that losses in the supernatants were possibly due to pollen geometry. They experimented with variations in the suspension liquid (distilled water with or without detergent added to reduce the surface tension) with somewhat confusing results, and with differing centrifuge speeds which they showed did not significantly affect pollen loss. The above experiments with plant opal processing would seem to mirror the problems they found in pollen processing. The problem is compounded by the fact that plant opal contains, in addition to silicon, oxygen and water, impurities which considerably affect its colour, refractive index and specific gravity (Wilding *et al.*, 1977). It is obviously an area which requires further work.

Study 2: Contamination by charcoal

A further separation problem encountered was the contamination of samples with charcoal (see Chapter 6). It was found that the SG of charcoal coated plant opal ranged from at least 1.7 to 2.3. In most samples a little charcoal or carbon coated opal remained but in instances where fire was a recent event (the Ecology Reserve) and in depositional areas such as the Oxford Falls swamp, the amount of charcoal was more than a minor annoyance.

Materials and method

Initially, this problem was encountered during the Oxford Falls work. During the separation of plant opal from the core (OFC1), 2 parallel samples were taken; sample 1 from the darker area of the core (0-50 mm) and sample 2 from the lighter area under this (50-55 mm). Both were subjected to the normal separation method outlined in Appendix F, using zinc bromide as the heavy liquid. Before separation, both samples were oxidised using sulphuric acid and chromium trioxide to remove organic material. Both samples were then run through a series of centrifuge steps with the SG of the zinc bromide ranging from 1.5 to 2.0. Slides were made of the separated opal at each step, and the results examined under light microscopy.

Results

At the normal separation SG of 2.3, both opal samples were found to be contaminated with charcoal. This problem was much more severe in the case of sample 1.

Sample 1: Examination of the sink from at successive SGs between 1.5 and 2.0 showed plant opal and charcoal in both the sinks and the supernatants. Plate 32 shows material from the supernatant at SG 1.7. It clearly shows that plant opal remains in the charcoal at low SGs.

Sample 2: No further plant opal was removed in the supernatants at lower SGs.

The removal of charcoal

Charcoal is the result of the incomplete combustion of organic material. Decomposition requires a large amount of energy. In the separation reported above, treatment with acid and oxidation of the samples before separation did not remove the charcoal. Bowdery (1989) reports that phytoliths have been extracted from residual carbonised fragments in ash samples by incineration until white hot. In the extraction of phytoliths from peat samples, Huber (1987) heated samples at 350°C for 1 - 7 days to determine loss-on-ignition.

When this same problem was found in the Ecology Reserve samples, loss-on-ignition technique was used (see Chapter 6).

The coatings can be removed by heating in a muffle furnace in temperatures up to 560°C for 24 hours, and, provided no other organic material is present, the opal appears to be little affected. However, this method is not without possible problems, and care should be taken that the characterisitics being examined are not changed. This is another area which requires further experimentation.

Other analytical techniques

(i) Particle size distribution

The particle size distribution of plant opal in soil is of particular interest since much is suspected to be in the clay fraction. Few reports on attempts to erect a particle size distribution curve for disarticulated plant opal, either from the plant or soil are to be found in the literature. One which was reported and is of most interest to this thesis was a tribute to Geis in the Phytolitharien Newsletter (Vol. 4 (2): 1987) which reported work on the particle size distribution of plant opal derived from litter.

Geiss (1984) examined litter in two forest communities in Central Illinois; a mesophytic mixed and an oak-hickory forest community. Four replicate litter samples were removed. These samples were not decayed and were considered to approximate annual litter additions. Plant opal was extracted and the particle size distribution determined in a long test-tube. Unfortunately, most of the results of this experiment are not available (Rovner, 1989, pers. comm.), since the Tables referred to in the text are missing. However, Geis comments in the text that "over 90% of the opal from forest litter occurs in particle sizes finer than 20 microns" (1987:7).

In the course of this thesis, experiments involving the use of a Sedi graph 5100 with V2.00 software have been conducted to determine the particle size distribution of plant opal derived from grasses.

The Sedi graph measures particle concentration in a fluid using a finely collimated X-ray beam to measure the transmitted intensity of the suspension relative to the suspending fluid. As such, it uses Stokes Law, and is subject to the problems inherent in this (see above). To date, experiments using *Stipa* sp. plant opal in water have shown that this suspending fluid has too low a viscosity to provide useful results, and experiments are under way using ethylene glycol as the suspending liquid. In addition to this problem, the amount of plant opal required is large, and it is intended to use a spike in the suspension to surmount this problem.

(ii) Elemental composition of plant opal

While many of the characteristics of plant opal are well known, shape is the usual one which has been considered in investigations which use plant opal as a tool in paleoecology and archaeology. With the availability of more sophisticated instruments, however, it should be possible to consider the use of some of the other known variable characteristics of plant opal such as the presence of occluded elements.

Jones and Milne (1963) isolated the plant opal from oats and examined the elements contained with the silica, while Bartoli and Wilding (1980) determined elemental composition of plant opal in three forest species and two grass species. Elements searched for were Si, Al, Fe, Mg, Ca, Na, K, Mn, organic C and Ti and the amounts of elements other than Si present ranged between 0.1 and 5.6% by weight. These authors considered that the content of elements was probably due to the elements present in the cytoplasm of the cell during silicification.

It has been suggested that the elemental chemical composition of plants may be species related (Shkolnik, 1984). Iodine concentration is often a species characteristic among marine algae and the highest molybdenum contents in plants are in the families Fabaceae and Caesalpiniaceae and subfamily Mimosoideae. High and extremely high concentrations of boron is a common property of the angiosperms. It is also suggested that trace elements have influenced the evolution of species and that plant associations have similar concentrations of trace elements in them.

Thus, if the elemental composition of the plant opal reflects the elements present in the cell cytoplasm and the element or proportion of elements differ between species, plant opal extracted from the soil may be traced to the species or association of species giving rise to them. If, however, the elemental composition of the plant opal is a function of the sediment in which the plant is growing, the use of occluded elements in plant opal to "fingerprint" sediments then becomes a possibility.

Study 3: Probe analysis of plant opal

Materials and methods

Samples of day 2, 14 and 30 plant opal from the ash at the Ecology Reserve (see Chapter 6) were prepared for analysis by an ETEC autoprobe with 3 crystal spectrometers and a Link Energy Dispersive System. The probe measures elements other than hydrogen in parts per hundred. Plant opal suspended in distilled water was dropped onto a special glass slide and allowed to dry naturally. The sample was magnified (500X) and displayed on a screen to allow the operator to select part of the sample for analysis. A software program (Link Analytical ZAF4/FLS) presented a quantitative analysis of the composition of the sample.

Results

The results are presented in Table 7.1. The morphology of each plant opal specimen analysed is listed, and the analysis given in terms of oxides. The results are similar to those presented by Bartoli and Wilding (1980).

Study 4: Trace elements in plant opal

Preliminary work has also been undertaken to examine the possibility that trace elements outside the detection range of the probe are present in the plant opal. Such elements were envisaged to be present in very small (ppb) quantities. The inductively coupled plasma source mass spectroscope (ICP/MS) was used which, according to its manufacturers, has the following attributes:

1. Concentrations of elements are measured directly; it counts individual atoms.
2. Detection limits are said to be excellent.
3. Most isotope ratios can be determined directly from solution.
4. Sub-ng/mL detection limits for most elements.
5. Very small samples can be scanned.

This instrument has many advantages but probably the most important of these as far as plant opal analysis is concerned is the size of sample required. Removing several grams of sediment, purifying the opal and analysing this is a rather sledge-hammer approach to the problem. Using the ICP/MS a few plant opal pieces, or even one of a particular morphology, can be used, thus opening up new possibilities to be explored.

Table 7.1: PROBE ANALYSIS OF ASH SAMPLES, ECOLOGY RESERVE

SAMPLE #	MORPHOLOGY	% OXIDE SiO2	TiO2	Al2O3	FeO	MgO	CaO	Na2O	K2O	TOTAL
Day 2:1	bulbous 2D sheet	97.83	0.00	1.08	0.00	0.00	0.00	0.66	0.00	99.57
2	3D sheet	97.37	0.00	0.68	0.09	0.00	0.00	0.22	0.00	99.63
3	3D sheet	98.87	0.00	0.40	0.34	0.00	0.00	0.00	0.00	99.61
4	ridged rod	95.38	0.00	2.86	0.41	0.00	0.00	0.00	0.00	99.69
Day 14:5	rough 3D sheet	75.84	0.34	13.97	4.97	1.25	2.11	0.94	0.54	99.94
6	chrysophyte	83.03	0.00	2.85	0.34	2.80	4.31	5.98	0.67	99.96
7	rod	87.07	0.00	2.32	0.36	2.07	3.22	4.36	0.38	99.79
Day 30:8	2D sheet*	86.46	0.00	1.64	0.68	2.10	3.56	4.42	0.92	99.77
9	hair	87.94	0.00	2.06	2.27	1.70	3.06	2.51	0.27	99.81

* stomata

Table 7.2: PRELIMINARY VG PLASMAQUAD RESULTS, PLANT OPAL FROM SPECIES GROWING ON SAND MONKEY (SITE 10)

ELEMENT	MASS	SAMPLE Actinotus helanthi (flannel flower) leaf	stem	Triodia mitchellii (spinifex) leaf	stem	1-5cm soil	100cm soil	600cm soil
B	11	611	318	0	83	1	1	1
Na	23	5667	2909	65	300	*	*	*
Mg	24	1000	1182	2	133	*	*	*
Al	27	16889	24318	75	1350	322	120	189
P	31	*	*	*	*	*	*	*
Ca	44	1889	2545	184	600	*	*	*
Sc	45	167	45	0	0	2	1	3
Ti	46	2000	1273	35	267	69	33	27
Ti	48	1278	909	2	217	33	22	2
V	51	*	*	0	*	44	4	13
Cr	52	1389	1318	16	733	16	4	8
Mn	55	111	91	1	33	23	3	9
Fe	57	5500	7818	150	*	*	*	*
Ni	60	1167	2818	1	17	2	1	4
Cu	63	222	136	1	17	4	4	3
Cu	65	222	91	2	17	5	4	4
Zn	66	333	500	3	67	7	2	11
Zn	68	0	227	4	33	7	4	13
Ga	69	0	0	0	*	4	0	2
Ge	72	111	45	1	33	3	1	2
Se	77	1444	1500	9	533	4	5	7
Br	81	0	0	9	*	10	6	8
Y	89	0	0	0	*	0	0	1
Zr	90	0	45	0	0	1	0	0
I	127	389	227	0	50	1	0	0
Ba	138	56	45	0	33	7	2	34
Nd	146	0	0	0	*	0	0	3
Pb	208	222	409	0	83	2	1	2
Th	232	333	182	0	33	1	0	1

* No response calibration (quantity too large)
All data in ppb.
Internal standard used: In: Mass 115

Experiments have been and are being conducted using the ICP/MS at the CSIRO Division of Tropical Crops and Pastures, Cunningham Laboratory, Queensland. The analysis has been undertaken by Mr Alan Johnson of that laboratory; sample preparations by myself under the support of a 1989-90 CSIRO/Macquarie University Collaborative Research Grant and a 1991 Macquarie University Research Grant.

Materials and methods

The mature leaves of *Actinotus helanthi*, flannel flower (family Apiaceae) and *Triodia mitchellii*, spinifex (family Poaceae) were collected from the sand monkey (Site 10) in the Pilliga State Forests. Sediment from the underlying soil was also collected (5 cm, 100 cm and 600 cm). Plant opal was extracted from the leaf samples as outlined in this chapter, and this, with the sediment samples, was submitted to analysis by the ICP/MS.

Results

Results are tabulated in Table 7.2. These show that in addition to those elements which had been detected within the phytoliths previously by Jones and Milne (1963), Bartoli and Wilding (1980), etc., several elements previously undetected were present in very minute quantities (parts per billion). These elements were also found to be present in the sediment. The differences in element concentrations between the plant opal of the two species is of interest, as is the case where some elements (such as Sc, Zr) are present in the plant opal of *A. helanthi* but not in that of *T. mitchellii*.

Discussion

There are several problems which are at present being eliminated from the procedure outlined above in order to improve the reliability of the results. Purity of samples is a problem. While strategies to improve purity during the extraction stage including the use of plastic implements, the use of water for washing the samples which has been distilled using plastic distillation equipment only and many more washes of the plant opal product are obvious and may solve the problem in plant extracts, the problem in sediments is much more difficult. As discussed earlier in this chapter, the methods currently used for the analysis of plant opal in sediments do no more than concentrate them. No matter how carefully the separation is done, impurities in the form of other minerals will persist. When such a plant opal sample is submitted to the ICP/MS for analysis, these impurites will affect the analysis.

In the manufacture of monoclonal antibodies, biologists isolate one cell for cloning purposes. The method they use involves the picking up of the cell out of a culture. Such a method could be used to remove plant opal pieces with the advantage that a particular morphology could be selected. This is the next obvious step in the purification of plant opal from sediments. The establishment of techniques for purifying very small samples of material will be an important contribution since the continued use of the ICPS/MS at these high levels of resolution demands absolute purity of sample.

Summary

In this chapter the new or modified techniques used in this thesis to separate and analyse plant opal from plants and sediments have been discussed. The use of a new heavy liquid (sodium polytungstate) was compared with an older method, and the problems in separation techniques explored. Techniques which have not been used before in the analysis of plant opal - for particle size analysis and the determination of elemental composition have also been discussed and the significance of these advances in future work indicated.

The discussion of techniques continues in Chapter 8, where the litter study which formed a major part of this thesis is outlined.

CHAPTER 8: LITTER STUDY TECHNIQUES

Introduction

In Chapter 2 it was found that the amount of plant opal entering the Oxford Falls swamp was 4 $gm^{-2}y^{-1}$. While this contribution may be greater than from most vegetation communities, the actual amount over a wider range of communities is of interest. The production of particulate silica in the Pilliga field sites was examined by entering the cycle in the litter layer (Chapter 3). The litter from three forest communities was analysed for plant opal amount and morphology, and in order to do this, a major litter study was initiated in 1987 which continued for 153 weeks until September 1990. This Chapter outlines the techniques used, and presents and discusses the results. Appendix D discusses material peripheral to this study.

Litter

Litter is a layer of dead plant material which may be present at the soil surface or dead plant materials not attached to a living plant (Satchell, 1974:xv). Such a litter layer may or may not be clearly distinguishable from the underlying mineral soil layer.

There are several systems of nomenclature for the litter layer. The nomenclature widely used in Australia defines the O horizon as the upper part of soil above the mineral soil dominated by fresh or partly decomposing organic matter; organic matter levels must be greater than 30% if the clay fraction of the mineral soil is greater than 50% and greater than 20% if the mineral fraction has no clay. It is divided into the O1 in which the nature of the original vegetation is apparent to the naked eye and the O2 in which discrete parts no longer identifiable (Hubble and Isbell, 1983).

Very often the litter layer comprises the O1 only. In the Australian litter layer, humification is of minor importance and the litter layer is usually a distinct layer with a sharp boundary between it and the underlying mineral soil. The important process is comminution. There is a gradual decrease in size of material between the top of the layer and the base. The lower part of the layer is not humus in the Northern Hemisphere sense; it is not greasy to the touch. Australian litter layers are suprisingly dry. The main process within them is comminution by the mesa-fauna in preparation for incorporation into the soil.

In the study area, the main field sites' litters were as described above. The mallee litter layer was particularly interesting, since termites appeared to be bringing mineral soil to the surface of the layer and covering the litter before beginning to feed on it (Plates 18 to 21).

(ii) Amount

The mass of the litter layer is not well known in Australia but variation is believed to be substantial due to the non-random nature of tree spacing and litterfall accession, soil variability, bark haloes around trees, the stage of development of the forest, the length of time since fire, the nature of shrub and herb layer, etc.

Annual litter fall varies considerably between forest types (Table 8.1). Production also varies throughout the year; for example the East Australian leaf fall is maximum in spring and early summer. The component proportions vary also (see Table 8.2). In Australia the bark fall is high, often accounting for up to 30% of the total litter fall, depending on the species.

Incorporation into the soil

The source of material entering the soil is from debris on the surface and from roots. Some litter is buried soon after it reaches the surface, mainly by earthworms and termites, but also as corpses of

Table 8.1: ANNUAL LITTER FALLS FOR VARIOUS FOREST TYPES*

Forest type	Litter fall t/ha/y	Litter fall kg/ha/y
equatorial rainforests	5.5 - 15.3	5500 - 15000
warm temperate forests	2.9 - 8.1	2900 - 8100
cool temperate forests	1.0 - 6.9	1000 - 6900
arctic conditions	0.6 - 1.5	600 - 1500

* Williams and Gray (1974:611)

Table 8.2: Reports of amounts and components of annual average litter fall, Australia, since 1970

species	location	Litter in kg ha^{-1} y^{-1} leaf	twig	bark	total	Author
E. regnans	Wallaby Ck (Vic)					
mature forest stage		2737	1689	1403	7756	Ashton
spar " "		3721	2039	1094	6194	(1975)
pole " "		3141	1789	921	5868	over 5yrs
Total forest	as above					
mature forest stage		4016	2050	-	7756	as above
spar " "		3942	2476	-	7731	
pole " "		3446	2120	-	6606	
Euc/Angophora forest	Brisbane				1800-3600	Birk (1979b)
overstory		57-61%	)	)30%	85%	Birk
understory		12-21%	)	)	8-15%	(1979b)
E. marginata (jarrah)		50%		-26%-	2400-3100	Hatch (1955)
Angophora-Eucalyptus	Hawkesbury S. St.				2680	Hannon (1958)
subtrop E	Nth Stradbroke Island	44%	25%	23%	6400	Rogers & Westman (1977)
E. obliqua					3600-5500	Attiwill *et al.*, (1978)
E. obliqua & *E. baxteri*	S Aust		430		2330	Lee & Correll, (1978)
E. sp	Armidale NSW				2500-3700	Pressland (1982)
E. sp	Seal Rocks NSW				1670	Fox *et al* (1979)
A. costata-) *E. gummifera*)	Narrabeen Lagon	2970	1134	1080	5400	Lamb 1985
E. botryoides-) *E. robusta*-) *A. floribunda*)	NSW	4247	2235	522	7450	" "
E. maculata	NSW	43%	21%	20%	6840	McColl 1966 In Lamb 1985

organisms, faecal pellets, etc. This material is incorporated into the topsoil by various processes such as growth of organisms which defecate, die etc., mechanical disturbance of the soil by natural physical means (freeze-thaw for example), agricultural means or by soil animals and acretion by wind or water borne mineral and organic particles. It is concluded that practically all litter materials pass through macro and meso fauna in the initial phase of decomposition but the total amount of litter consumed annually is difficult to estimate.

A large variety of micro-fauna and large numbers of different species of meso and macro-fauna inhabit the litter. Initially, fresh litter is not palatable to saprophagous litter fauna, probably due to the presence of polyphenols. "It is generally accepted that the consumption of leaf material by the litter and soil fauna mainly results in a mechanical disintegration or comminution of the ingested material without much chemical change." (Jensen, 1974:94). Most fauna ingest easily decomposed carbohydrates and soluble nitrogenous compounds; termites may be an exception since they are able to digest cellulose and, in some cases, lignin (Lee and Wood, 1971). All of this activity by macro-fauna results in the exposure of more surface area of leaf litter for access by micro-organisms. Differences in structure and components, i.e. leaves etc., from various plants leads to different rates of decomposition in the same environment.

The study of the mobility of elements from the litter layer during decomposition has concentrated on those elements which are essential for plant growth - nitrogen, sodium, potassium, calcium, etc., i.e. nutrient losses. The loss of silica has been discussed by Remezov (1961), but is generally not considered.

The litter study

This study was conducted at three sites in the Pilliga State Forests; Site 7, the upper broom plain; Site 8, the mallee; and Site 12, the forest. Fire affected all sites in 1951, and parts of Site 7 were burned in 1966/67.

The aims of the litter study were two-fold. Firstly, the amount of litter accumulation, and the proportion of each component on an annual basis was required, since this would give an estimate as to the amount of plant opal entering the cycle via the litter layer. Secondly, the decomposition rates of litter in each of the three sites were of interest since this governs the rate of plant opal inclusion into the soil.

A. Amount of litter

Three types of litter collections were made to give an estimate of the amount of litter fall on an annual basis, the standing crop of litter (bulk collection), and to examine the question of steady state accumulation of litter (an initial and final collection).

A. Initial and final collections and annual litter fall

(i) Aims of study

The aims of this study were:

1. to measure annual litter fall and to examine its variation throughout the year,
2. to calculate the opal content of the various components of the litter collected in order to provide an estimation of the amount of plant opal entering the cycle through the litter layer and
3. to make two litter collections separated in time to ascertain if the litter was in steady state, i.e. if inputs were balanced by outputs.

(ii) Litter tray construction

Litter was collected in litter trays measuring 50 cm x 50 cm. These were constructed of four pieces of 2 cm diameter plastic electrical conduit joined by plastic right-angled elbows. This provided the frame of the tray. The elbows had a hole drilled through which a piece of wire was threaded. The wire then passed through the centre of a 10 cm long piece of conduit, providing the legs. The wire protruded out of the conduit, and was used to anchor the frame in the ground.

Terylene curtaining material was used to provide the tray, since it was both inexpensive and easily worked with. The curtaining was cut into squares 70 cm x 70 cm to make the tray. A hem 7.5 cm wide was sewn along each edge, and the corners of the tray were mitred to provide access to the hem for the frame.

In the field, each tray was assembled by threading a 50 cm long frame side through each of hems and joining them with the elbows to form a square tray. The wire was passed through the elbow and leg at each corner, and the tray placed on the ground. Each tray stood 10 cm above the ground with two sides alined north-south (Plate 22). One of the sides was labelled in two places with the number of the tray; at the mid-point where it could be seen and at the very end where the number was hidden under the elbow. This was found to be necessary as the numbers faded.

The advantages of this design were its ease in construction and assembly, and its lightness for carrying purposes. Litter in the tray was easily removed by hand, and the adhering particles brushed out using a small paint brush. It was found that the curtaining material began to deteriorate after about a year and, although the terylene was fairly strong, larger branches tended to rip it and replacement was necessary. This was both an easy and inexpensive task.

(iii) Litter collection

In May, 1987, an initial litter collection was made at each site and in May, 1991, a final litter collection was made. In each case, ten 1 m^2 quadrats were cleared at points radomly determined from within a 100 m x 100 m area. The litter was dried and weighed (Table 8.3) and in the case of the initial litter collection, several subsamples from each site were separated into components and weighed to establish the ratio of components for the mesh bag experiment.

In October 1987 the litter trays were established to collect litter fall. At each site the south-west corner of each of ten litter trays was located according to ten random grid references within an area 100 m x 100 m. The area was located according to a reference on the road, and the side nearest the road was parallel to the road but 10 m into the bush, thus avoiding road edge effects. Figure 5.1 shows the location of each area. In Site 12, trays 1, 3 and 5 were relocated after forestry operations destroyed them in the 81st week.

Over the following three years (153 weeks) the litter was collected from all trays on each of 12 trips to the Pilliga. In the laboratory, the litter was air-dried and sorted into components. Litter was sorted into leaves, twigs, bark, faecal material and residue. Leaves were half a leaf or more, although in the case of the upper broom plain, the leaves were so small as to be impossible in most cases to separate, and were included in the residue. Twigs were divided into those less than and those larger than 1 cm diameter. Faecal material included only that of macropods. Small faecal pellets were included in the residue which also included the remaining small pieces of material. The litter was then dried to constant weight at 110°C and weighed. The average and standard deviation for each component at each site was calculated, and the cumulative figures expressed in gm^{-2} (Table 8.4).

Table 8.3: INITIAL AND FINAL LITTER SAMPLING, PILLIGA SITES (weight in g/sq.m)

SAMPLE #	SITE 7 INITIAL	SITE 7 FINAL	SITE 8 INITIAL	SITE 8 FINAL	SITE 12 INITIAL	SITE 12 FINAL
1	355	350	2153	1646	904	2840
2	51	768	760	2680	1369	2526
3	202	921	3774	2037	635	334
4	118	1353	1109	969	1597	2137
5	585	762	881	1444	308	809
6	244	438	955	1468	891	1410
7	135	899	1130	1574	858	671
8	401	871	1015	742	1061	811
9	477	680	2472	906	1852	423
10	425	431	1608	2494	1602	384
Average	299	747	1586	1596	1108	1235
Std Dev	166	282	907	618	461	892

Initial sample: May 1987
Final sample: May 1991

Table 8.4: LITTER; CUMULATIVE, BY COMPONENT: PILLIGA SITES
(average: g/sq.m)

SITE	WEEK	COMPONENT							TOTAL	DATE
		Leaves	Twigs >1cm	Twigs <1cm	Twigs Total	Bark	Faecal	Residue		
Site 7:										
Upper Broom Plain	0	0	0	0	0	0	0	0	0	Oct 87
	8	1.14	0	18.13	18.13	0	0	23.69	42.96	Dec 87
	22	3.51	0	19.10	19.10	0	0	34.88	57.49	Mar 88
	29	3.67	0	22.20	22.20	0.22	0	38.31	64.40	Apr 88
	43	5.44	4.79	24.88	29.67	0.44	0.29	45.93	81.77	Aug 88
	51	5.98	4.79	29.42	34.21	0.44	2.11	60.25	102.99	Oct 88
	71	10.59	4.79	33.87	38.66	0.44	2.11	92.59	144.39	Feb 89
	81	10.76	4.79	35.25	40.04	0.44	2.11	104.44	157.79	May 89
	90	10.78	4.79	35.47	40.26	0.44	2.11	105.92	159.51	Jul 89
	101	11.53	4.79	36.38	41.17	0.49	2.11	110.24	165.54	Sep 89
	124	13.41	21.00	53.44	53.44	0.49	2.11	163.38	253.83	Mar 90
	137	13.72	21.00	62.26	62.26	0.49	2.11	169.02	268.60	Jun 90
	153	13.86	21.00	80.20	80.20	0.49	3.73	176.46	295.74	Sep 90
						ANNUAL AVERAGE TOTAL			100.5	
Site 8:										
Mallee	0	0	0	0	0	0	0	0	0	Oct 87
	8	19.45	0	3.89	3.89	7.51	0.24	24.13	55.22	Dec 87
	22	59.54	0	23.76	23.76	16.01	0.24	62.86	162.41	Mar 88
	29	67.74	0	25.51	25.51	20.02	0.24	71.29	184.80	Apr 88
	43	73.02	3.82	27.61	31.43	22.22	0.54	78.17	205.38	Aug 88
	51	84.96	3.82	37.39	41.21	25.74	0.54	94.42	246.87	Oct 88
	71	132.71	84.40	99.81	99.81	55.25	1.73	141.49	515.39	Feb 89
	81	138.21	84.40	101.25	101.25	58.54	1.73	157.03	541.16	May 89
	90	139.06	84.40	102.09	102.09	59.25	1.73	161.25	547.78	Jul 89
	101	145.26	84.40	110.70	110.70	63.45	3.37	167.56	574.74	Sep 89
	124	280.61	90.66	129.72	220.38	70.87	3.79	230.50	806.15	Mar 90
	137	286.92	173.21	178.87	352.08	78.81	5.39	253.78	976.98	Jun 90
	153	292.74	173.21	193.31	366.52	80.20	5.48	258.24	1003.18	Sep 90
						ANNUAL AVERAGE TOTAL			340.9	
Site 12:										
Forest	0	0	0	0	0	0	0	0	0	Oct 87
	8	27.24	11.30	16.93	16.93	0.19	0	28.74	73.1	Dec 87
	22	39.32	26.66	34.77	61.43	1.43	0	64.74	166.92	Mar 88
	29	42.09	31.68	37.05	68.73	1.55	0	68.32	180.69	Apr 88
	43	42.09	31.68	37.29	68.97	1.55	0.31	71.63	184.56	Aug 88
	51	48.61	31.68	48.48	80.16	2.70	0.31	104.76	236.55	Oct 88
	71	95.34	31.68	72.26	103.94	2.98	0.31	167.80	370.38	Feb 89
	81	97.59	31.68	78.99	110.67	3.72	0.39	179.77	392.14	May 89
	90	97.75	31.68	79.07	110.75	3.84	0.39	180.73	393.46	Jul 89
	101	99.37	31.68	82.15	113.83	4.50	0.39	186.23	404.32	Sep 89
	124	143.96	53.33	107.9	161.23	11.89	0.76	272.25	590.09	Mar 90
	135	147.52	53.33	112.58	165.91	11.89	2.28	308.37	635.97	Jun 90
	153	149.60	69.59	115.86	185.45	12.31	2.66	322.70	672.72	Sep 90
						ANNUAL AVERAGE TOTAL			228.6	

From these figures can be derived the average annual litter fall at each site. The total weight of each component was expressed as a percentage of the total litter at each site (Table 8.5).

Since the litter trays only measured litter from plants above 10 cm tall, a collection of the biomass under 10 cm tall was made during the spring of 1990. Ten random 50 cm x 50 cm quadrats were located within each 100 m x 100 m area and all of the biomass under 10 cm tall clipped. The material was dried and weighed, and converted to an average weight per square metre. The material gathered was assumed to represent the total annual growth of the herb layer (Table 8.6).

Results

(i) Litter fall patterns

The distribution of litter fall through time at the three sites is illustrated in Figure 8.1. All show a marked lull in the amount of litter falling during the winter months. Similar reports are to be found in the literature (Birk, 1979, Lamb, 1985). Figure 8.1 shows the partitioning of this litter fall by component. Leaves from Site 7, the upper broom plain, are very small and contributed mainly to the residual component. However, those which were large enough to be separated as leaves demonstrate a less marked seasonality in fall than the leaf component at the other sites. Bark was a major component in the mallee only. *Eucalyptus viridis* sheds bark fairly constantly throughout the year with a slight summer high, providing a halo of bark around each mallee clump.

Lamb (1985) examined litter fall in two eucalyptus woodlands near Sydney in a humid climate, and found that in seasons of below average rainfall, litter falls, which peaked in summer, showed a more pronounced peak. He found wood and twig fall highest in late summer and bark in early spring. Similar results were obtained by Birk (1979) in a

Table 8.5: PERCENTAGE OF TOTAL LITTER FALL BY COMPONENT

	COMPONENT				
Site	Leaves	Twigs	Bark	Faecal	Residue
7	4.7	34.4	0.2	1.3	59.6
8	29.2	36.5	8.0	0.5	25.7
12	22.2	27.6	1.8	0.4	48.0

Table 8.6: SITES 7 (BROOM PLAIN), 8 MALLEE AND 12 (FOREST): BIOMASS UNDER 10cm HEIGHT

Sample No	Site 7 Weight g	Site 8 Weight g	Site 12 Weight g
1	1.31	0.91	12.56
2	2.96	3.58	12.89
3	4.30	3.26	17.76
4	6.71	2.26	12.45
5	10.32	0.72	8.34
6	8.41	0.97	22.92
7	5.31	6.87	19.56
8	2.96	1.01	8.23
9	8.21	5.96	12.89
10	7.01	1.13	13.35
Total	57.50	26.67	140.95
Average	5.75	2.67	14.10
SD	2.72	2.11	4.44
Av/m-2	23.00	10.67	56.38

Quadrats 50cm x 50cm

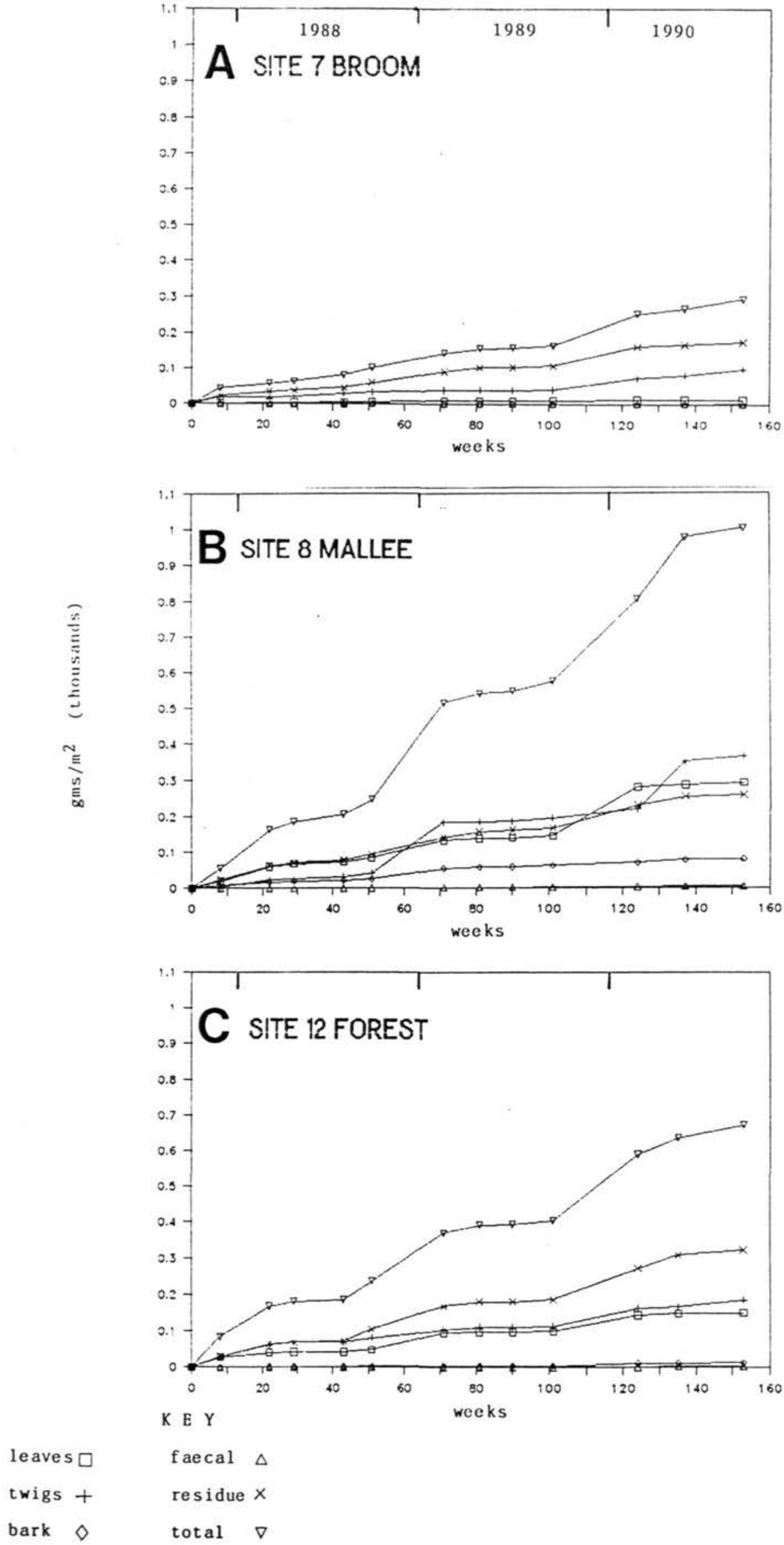


FIGURE 8.1: CUMULATIVE LITTER BY COMPONENT

euclayptus forest near Brisbane (subtropical climate), with overstory leaf litter strongly correlated to the overall litter fall, while understory litter mainly fell in spring. In this environment, grass fragments were found to fall a little heavier in late spring and summer.

Table 8.4 gives a summary of annual litter fall for the three sites by component and Table 8.5 displays this as a percentage of the total litter fall. In all three sites, the twig-fall was a dominant component. Both Birk (1979) and Lamb (1985) found that leaves provided the main litter component in the eucalypt forests studied, while wood, although an important component, provided about half the leaf component. Leaf-fall and twig-fall were about even in importance in the mallee and forest. The reason for this difference may be the differences in both climate and species.

All sites demonstrated distributions which were explicable in terms of the vegetation at each site. For example, Site 8 (mallee) had a high bark component due to the species in this community shedding bark prolifically. This component is probably under estimated by these figures since the trays did not sample adequately the bark haloes around each mallee tree.

(ii) Annual litter fall

Annual litter fall for the three sites was estimated to be 1005 kg/ha/yr for Site 7 (upper broom plain), 3409 kg/ha/yr for Site 8 (mallee) and 2286 kg/ha/yr for Site 12 (forest). This is the material caught in the litter trays only; when the estimated annual addition from the herb layer below 10 cm height is added, this becomes 1235 kg/ha/yr (Site 7), 3516 kg/ha/yr (Site 8) and 2850 kg/ha/yr (Site 12).

Climate over all sites is semi-arid. Williams and Gray (1974:611) reported annual litter falls for various forest types (Table 8.1). The nearest category to the climatic zone for the Pilliga State

Forests is the warm temperate forests. While Site 12 would fit into the litter fall range for this environment as given by Williams and Grey, Sites 7 and 8 fall into the cool temperate forest category.

Table 8.2 summarises litter studies in eucalyptus forests in Australia since 1970. The upper broom plain (Site 7) annual litter fall approximates to that of Fox *et al.* (1979) at Seal Rock in New South Wales. The vegetation examined at Seal Rock, however, was a tall eucalyptus open-forest. Both the mallee (Site 8) and forest (Site 12) have litter falls similar to published figures for drier areas of Australia (Hatch, 1955; Hannon, 1958; Lee and Correll, 1978).

(iii) The initial and final litter collections

Table 8.7 summarises the initial and final litter collections in each site plus the initial litter collection from a 1971 fuel equilibrium study initiated by the Forestry Commission in the East Pilliga State Forest, near the sites discussed in this thesis.

The equilibrium study was started in September 1971, 20 years after the last known fire over the plots (November 1951). The plots were established in white cypress pine (*Callitris glaucophylla)* with a *Casuarina luchmanni* understory. Forty plots were established with differing treatments, the aim being to establish the fuel weight on the forest floor after 20 years with annual re-sampling see if the litter had attained steady state, i.e. annual additions to the litter layer were balanced by the material entering the soil or being lost to the system. Unfortunately, the annual resampling figures have not been located.

The average total fuel weight of the study sites was less than 2.5 t/acre (6726 kg/ha) and included all litter and vegetation to 20 ft (6 m) tall. The litter layer including all vegetation below 3 ft

Table 8.7: INITIAL AND FINAL LITTER WEIGHTS (kg/ha)
PILLIGA STATE FOREST SITES

	Site 7		Site 8		Site 12		Fuel Study*	
	initial	final	initial	final	initial	final	initial	final
Average	2990	7470	15860	15960	11080	12350	6108	6298
sd	1660	2820	9070	6180	4610	8920	785	725
yrs accumulation	19		36		36		20	
1/2 life of litter	3.9y		2.4y		3.2y			

* Forestry Commission Study, total biomass under 3m

(0.9 m) weighed 2.48 t/acre (6,226 kg/ha). The length of time taken to accumulate this was 20 years. This study is difficult to equate with the present litter study since it was a fuel accumulation study which included the biomass. The nearest treatment is one which removed all litter and vegetation to 3 ft in height. It showed that equilibrium had been reached 20 years after fire. While the initial litter collections in the present study did not include the vegetation to 0.9 m high, some comparisons can be made with Site 12.

The forest (Site 12) had an initial litter collection of 11,080 kg/ha, accumulated over 36 years (since the last fire). Both this site and Site 8 (mallee) appear to have reached steady state, while the upper broom plain, with only 19 years accumulation from the last fire, is not in steady state.

B. Bulk Litter Collection

Bulk collections of litter were made from the Pilliga sites in September 1990. The purpose of this collection was to give an estimate of the plant opal in storage at each site in the litter layer and to provide assemblages of plant opal characteristic of the litter layer (see Chapter 3).

Within a 100 m x 100 m area at each of four sites in the Pilliga (Site 7, upper broom plain; Site 8, mallee; Site 9, lower broom plain; and Site 12, forest), 10 random collections of litter each 1/16 m^2 were made. All collections were made in areas last affected by fire in 1951 in order that they might be comparative. The samples were combined into one sample for each site, separated into components, dried and weighed, and weight in grams per square metre calculated for each site.

The percentage of the total litter in storage represented by each component is shown in Table 8.8. As with the annual litter fall, twigs provided the main input. When compared with the annual litter

Table 8.8: PERCENTAGE OF LITTER STORAGE BY COMPONENT

	COMPONENT				
Site	Leaves	Twigs	Bark	Faecal	Residue
7	0.0	14.0	2.5	2.1	81.4
8	13.0	23.7	5.7	1.2	56.4
9	0.6	21.5	0.6	1.0	76.3
12	8.3	50.9	0.2	0.3	40.2

Table 8.9: PROPORTION OF EACH LITTER COMPONENT IN EACH MESH BAG

Component	Site 7		Site 8		Site 12	
	g	%	g	%	g	%
leaves	0.2	1.0	1.0	5.0	1.5	7.5
twigs	4.6	23.0	5.0	25.0	5.5	27.5
bark	0.1	0.5	1.5	7.5	0.1	0.5
remnants	15.1	75.5	12.5	62.5	12.9	64.5

proportions by component (Table 8.5), leaves comprise a lesser part of the storage than they do in the annual litter fall, there is a higher proportion of twigs in storage in Site 12 than in the litter fall, bark accumulates at Site 7, and faecal pellets accumulate at all sites. This would seem to indicate a preference for leaves, generally, to decay rapidly, and the remainder to vary in decay rates in response to differing environmental factors.

The total litter in storage in the litter layer at each site in kg/ha was:

Site 7, upper broom plain:	6,293 kg/ha
Site 8, mallee:	12,011 kg.ha
Site 9, lower broom plain	4,886 kg/ha
Site 12, forest	17,311 kg/ha

C. **Mesh bag study**

(i) Aim of the study:

The aim of this study was to provide a litter half-life (i.e. the time taken for half of the litter to decay) for each of the 3 sites; Site 7, the upper broom plain, Site 8, the mallee and Site 12, the forest. These could then be used to examine the rate of incorporation of litter and hence plant opal into the soil and to compare the litter incorporation rates with other communities previously examined in Australia. The mesh bag method was used since it was reported by Lamb (1985) to be time saving and it was intended to make comparisons with the results gained in his study (conducted near Narrabeen Lagon, N.S.W. close to the Oxford Falls Swamp site). In addition, Woods and Raison (1982) point out that the use of a decomposition constant is difficult due to the assumption that the mass of accumulated litter is constant with time being frequently unjustified due to frequent burning. Further discussion on the techniques available is to be found in Appendix D.

(ii) Method:

In each of the three sites 30 mesh bags were tethered to the ground. The bags were 200 mm x 200 mm, of nylon mesh with an aperture size of 1.5 mm x 1.5 mm (Plate 31). Each bag was numbered and 20 g of dried litter from the site inserted.

The proportion of each litter component in this 20 g was calculated from the results of the 1 m^2 initial litter collection. Since this initial litter collection took several months to completely sort and dry, the number of samples used to calculate the initial litter proportion for the mesh bags was 4 or 5. The litter components used in each case were leaves, twigs under 1 cm diameter, bark and remnant material (see Table 8.9). They were collected from the litter trays at each site, dried, sorted, weighed and inserted into the bags.

The mesh bags were transported to the sites in plastic bags. The material left in the plastic bags after the mesh bags were layed out in the field, was taken back to the laboratory, dried and weighed. This amount was then subtracted from the original mass, giving the "mass in field". The mesh bags were laid on ground cleared of litter by hand sweeping, so that each bag lay on highly organic soil.

The bags for Site 7, the upper broom plain, were laid out in December 1987, while the 2 remaining sites' bags were installed in March 1988. The experiment ran to September 1989, (a total of 99 weeks for Sites 8 and 12, and 101 weeks for site 7), when it was terminated due to a combination of disturbance by wild pigs and some bag decay. A total of 27 bags were retrieved from Site 7, and 24 from each of the remaining two sites.

Three mesh bags were collected each field trip, transported back to the laboratory in plastic bags and the material initially air-dried in the plastic bags. The soil and litter adhering to the outside of the mesh bags was then carefully removed and discarded. The material inside the mesh bags was removed and oven dried.

It was found that the amount of sediment inside the mesh bags was very large indeed, and the litter was separated from this material by flotation. The sediment was dried and weighed (except for the July 1989 field trip, when it was accidentally discarded). Table 8.10A shows the average percentage of litter remaining through time, and Table 8.10B shows the mass of sediment in each mesh bag on retrieval, in each case averaged over the three bags except in incidents where the bags were too damaged to be included.

Results

Figure 8.2 shows the percentage of the original mass of litter remaining over time. The results were often highly erratic due to the amount of sediment remaining intimately mixed with the litter in some cases after flotation. However, the results do show that the mallee (Site 8) lost litter fastest, followed by the the upper broom plain and the forest. There was less litter removed in the colder months, and overall about 30% of the litter was removed in the approximately two years. The half life of the litter at each site was calculated from regression equations using the method of least squares (Mendenhall and Ott, 1976) [Table 8.10A and Figure 8.2] using a LOTUS 123 program.

The half lives calculated for the three sites were 3.9 years (Site 7), 2.4 years (Site 8) and 3.2 years (Site 12). Since the upper broom plain is not in steady state the half life calculated is probably an underestimate.

While none of the regression lines goes through the 100% value at time = 0, the upper broom plain and the mallee lines are reasonably close. The scatter of figures for the forest, however, leads to these

A: LOSS IN WEIGHT, LITTER

SITE 7: Broom Plain

time (weeks)	% original mass remaining	Regression Output:	
0	100	Constant	101.8895
43	93.04	Std Err of Y Est	4.900522
51	85.26	R Squared	0.796067
71	87.4	No. of Observations	7
81	86.64	Degrees of Freedom	5
90	79.74		
101	68.64	X Coefficient(s) -0.25745	
		Std Err of Coef. 0.058274	
		Regression equation	Y=101.8895-0.25745X
		Half life of litter	3.9 years

SITE 8: Mallee

time (weeks)	% original mass remaining	Regression Output:	
0	100	Constant	95.27037
14	96.64	Std Err of Y Est	4.021768
21	85.03	R Squared	0.894119
35	81.46	No. of Observations	8
43	78.2	Degrees of Freedom	6
63	71.88		
73	64.54	X Coefficient(s) -0.35724	
82	66.22	Std Err of Coef. 0.050187	
99	64.578	Regression equation	Y=95.27037-0.35724X
		Half life of litter	2.4 years

SITE 12: Forest

time (weeks)	% original mass remaining	Regression Output:	
0	100	Constant	91.63473
14	86.61	Std Err of Y Est	7.108106
21	79.07	R Squared	0.611932
35	81.39	No. of Observations	9
43	82.05	Degrees of Freedom	7
63	82.05		
73	61.6	X Coefficient(s) -0.24907	
82	71.07	Std Err of Coef. 0.074969	
99	73.77	Regression equation	Y=91.63473-0.24907
		Half life of litter	3.2 years

B: AVERAGE MASS OF SEDIMENT IN BAGS ON RETRIEVAL

	SITE 7: Broom Plain			SITE 8: Mallee			SITE 12: Forest		
Date	Time (wks)	av mass sediment	gain per week	Time (wks)	av mass sediment	gain per week	Time (wks)	av mass sediment	gain per week
Oct 1987	0	0	0						
Dec 1987	8	0.31	0.0388	0	0	0	0	0	0
Mar 1988	22	2.59	0.1177	14	*		14	*	
Apr 1988	29	5.48	0.1890	21	3.58	0.1705	21	1.09	0.0519
Aug 1988	43	*		35	4.52	0.1291	35	*	
Oct 1988	51	5.88	0.1153	43	2.09	0.0486	43	0.27	0.0063
Feb 1989	71	3.51	0.0494	63	17.64	0.2800	63	0.64	0.0102
May 1989	81	21.77	0.2687	73	6.08	0.0833	73	2.73	0.0374
Jul 1989	90	*		82	*		82	*	
Sep 1989	101	26.03	0.2577	99	26.86	0.3276	99	11.58	0.1170
average/week			0.1481			0.1732			0.0446
grams/year/sq.m			192			225			58

Table 8.10: MESH BAG RESULTS (BAGS 200 mm x 200 mm)

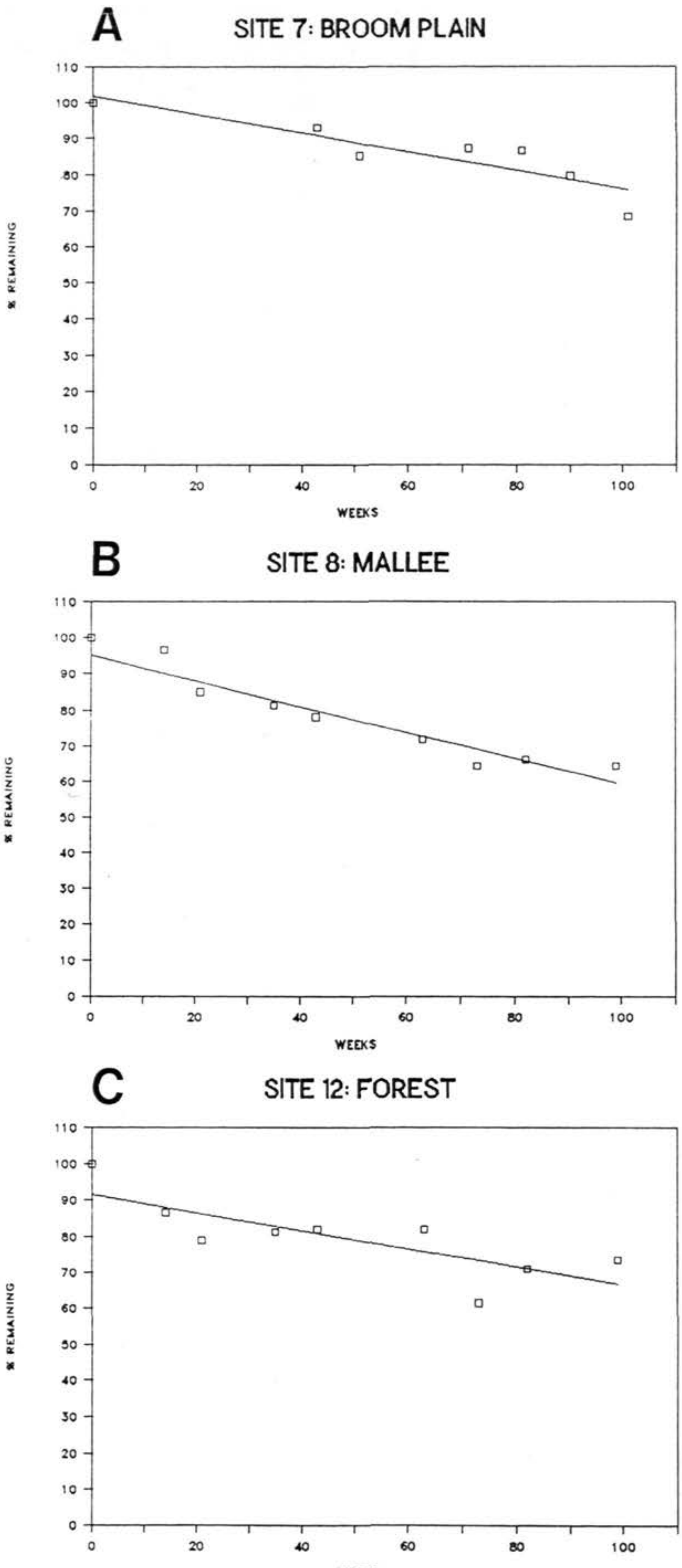


FIGURE 8.2: REGRESSION ANALYSIS OF MESH BAG WEIGHT LOSS, PILLIGA SITES

figures being treated with caution.

Figure 8.3 shows the sediment gain inside the bags over time. Again highly erratic, it never-the-less points to a considerable accumulation, particularly in the last few months, with the sites gaining sediment amounts again in the order mallee, upper broom plain, forest, i.e. in the same order that they lost litter.

Discussion

Published figures for the half-lives of litter in Australian forest communities are given in Table 8.11 and, in those studies which used mesh bags, varies from 1.8 (Lamb, 1985) to 4.9 (Hatch, 1955, quoted in Lamb, 1985). No comparable figures could be found for the communities examined in the present study, however the figures obtained (2.4 - 3.9 years, see Table 8.10A) fall within the published figures limits although they should be treated with caution, particularly those from the Forest (Site 12).

A second estimation of decomposition of litter is given by "k", the decay constant (Birk and Simpson, 1980). This is the proportion of the forest floor which turns over annually, and is calculated from the equation:

$$k = L/X_{ss}$$

where L = rate of litter fall

X_{ss} = litter mass at steady state

Thus for both Site 8 and Site 12 k is 0.21/yr if the initial litter collection weights are used (Table 8.7), and 0.28 (Site 8) and 0.13 (Site 12) if the bulk litter collection figures are used.

There are, of course, many assumptions in these calculations. Steady state conditions are required; this condition appears to be met by the two sites. The model on which the equation is based assumes litter does not vary year to year and that the proportion represented by

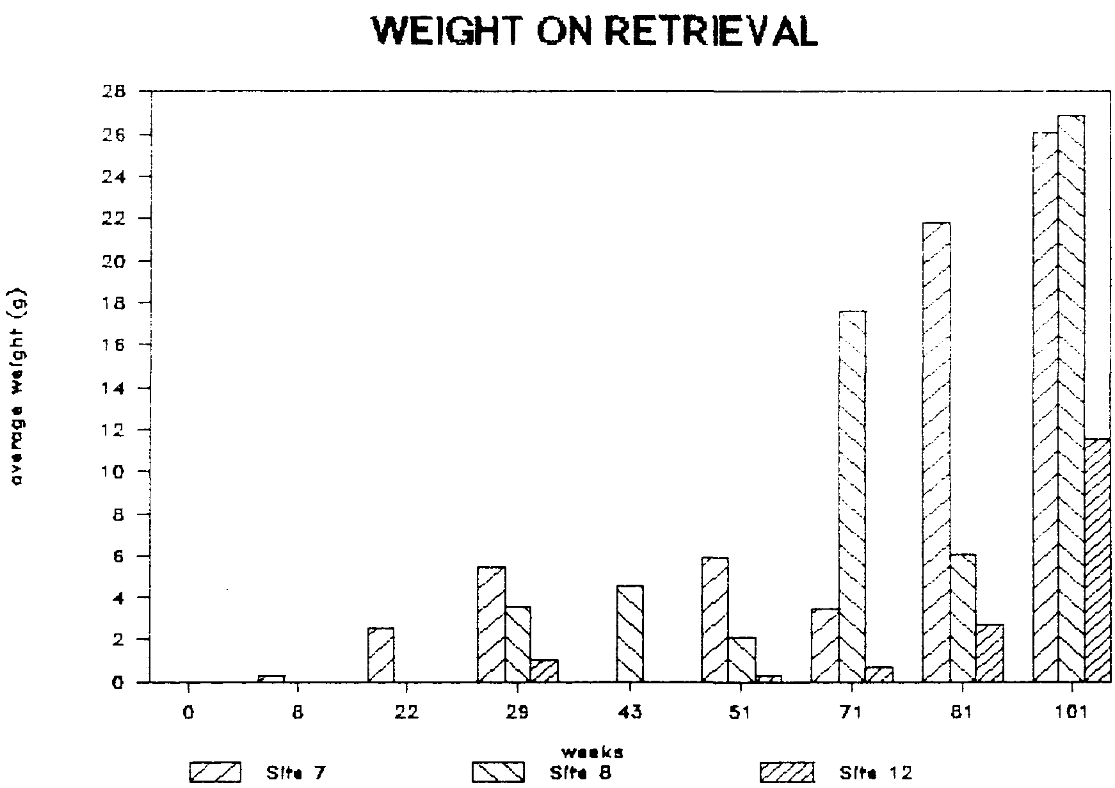


FIGURE 8.3: AVERAGE SEDIMENT GAIN IN MESH BAGS PILLIGA SITES

Table 8.11: Measurement of half lives of litter

species	location	half life (yrs)	method	author
Euc sp	Armidale			Pressland 1982
continuous forest		2.14	above ground collection	
discontin. forest		0.98		
sub trop E.	Nth Stradbroke Island	2.9		Rogers & Westman 1977
E. obliqua & *E. baxteri*	S Aust	2.91	above ground collection	Lee & Correll 1978
Angophora costata-) *E. gummifera*)	Narrabeen Lagon	2.7	mesh bag/accum litter	Lamb 1985
E. botryoides-) *E. robusta*-) *A. floribunda*)	) NSW	1.8	" " "	" "
E. marginata	W. A.	4.9		Hatch 1955 In Lamb 1985

k turns over annually, something which has not been tested (Birk and Simpson, 1980). Birk and Simpson indicate that loss may decrease with age as material is progressively buried, that is the decomposition rate is uneven throughout the litter. Woods and Raison (1982) point out that decomposition rates obtained by this method may not agree with mesh bag methods, and this appears to be the case in this study. However, these figures do give some comparative indication of the amount of litter which is incorporated into the upper layers of the soil annually.

It should be noted that this study proceeded for only 2 years. Lamb (1985) points out that different components of the litter decay at differing rates. Woods and Raison (1982) comment that mesh bags probably give an accurate measure of the early decay rates, but this should not be extrapolated to predict long-term turnover of litter, since rates of litter decomposition may increase substantially after the initial 1 or 2 years. From inspection of the data, this is difficult to assess, but it would appear that none of the sites reached this point. However, it appears that the accumulation of sediment was increasing towards the end of the experiment.

It should also be noted that the material used in the litter bags was taken from actual litter fall caught in the litter trays. Thus the hebaceous layer is not represented in this sample, nor are the larger than 2 cm diameter branches.

As noted by Lamb (1985) contamination from soil and litter debris was large, and the bags were destroyed by animals, notably wild pig. This was particulary noticeable in the later months after a dam had been built about 1 km away for the retention of water for fire fighting. The pig population in the field site area rose dramatically due to the presence of water, and the bags were increasingly prone to damage. The mesh also appeared to be considerably weakened after nearly 2 years of exposure, and the termites were, with increasing regularity, making holes through the mesh when building galleries.

It was the contamination by termites that was the most productive when it came to addition of sediment to the interior of the bags, and this addition was measured, since it appeared that it would give a minimum measure of the amount of material the termites were adding to the surface when foraging for food. The average amount added to the surface per annum at each site was calculated as:-

Mallee (Site 8):	225 g/m2/yr	(maximum 425 g/m2/yr)
Broom plain (Site 7):	192 g/m2/yr	(maximum 335 g/m2/yr)
Forest (Site 12):	58 g/m2/yr	(maximum 152 g/m2/yr)

These figures would give a turn over rate for the top 10cm of the soil at each site as:

Mallee (Site 8):	261 years
Broom plain (Site 7):	306 years
Forest (Site 12):	1,014 years

with a minimum of 138 years. These figures are based on a bulk density of the top soil of 1.7 g/cc, the average of published bulk densities for solodized solonetz soils in Stace *et al.*, (1968).

These figures do not, of course, include the amount of material that the termites bring above ground and deposit in the branches and trunks of dead, dying and living trees, thus they can only be taken as a minimum. They have startling implications for the genesis of the soils (see Appendix A).

Conclusions

While the litter study's primary purpose was to provide estimates of plant opal available for cycling it also provided information concerning the variation in litter fall, storage, composition and decomposition within the Pilliga State Forests.

The rates of decomposition of the litter were particularly interesting since not only did they show that the mallee litter was both accumulating and decaying at a faster rate, but they supported the observation that soil fauna were highly active at this site. The addition of sediment to the mesh bags was also highest at this site; another indication of faunal activity.

The litter study places the results of the investigations into the amount of plant opal in the litter and soils in the Pilliga sites into perspective when considered along side other environments in Australia and throughout the world. The assemblage studies which followed required an examination of the classification of plant opal and the erection of plant opal keys and techniques for assemblage studies. These are discussed in the next two chapters.

CHAPTER 9

THE MORPHOLOGY OF PLANT OPAL

Part 1: Plant opal keys

Introduction

Chapters 9 and 10 of this thesis deal with the morphology of plant opal. Much of the phytolith research in the archaeological and paleoenvironmental areas has been directed at finding either a species specific phytolith; a single shape diagnostic of a plant species; or an assemblage of phytoliths diagnostic of a vegetation community. Its aim has thus been the identification of plant species or communities, utilising the plant opal. The basis of such identification is reviewed in this Chapter, while Chapter 10 discusses assemblage studies and the details of the plant opal Keys erected for use during this work.

Classifications and Keys

Many "classifications", for example of plants for medicines, are not classifications at all, but Keys.

> "Identification schemes are *not* classifications. The procedure of identification is based on deductive reasoning. Its purpose is to place an investigated individual into one of the classes of an already existing classification. If one succeeds, one has "identified" the specimen. Identification deals with just a few characters .... By contrast, classification, as now conceived, assembles populations and taxa into groups, and these, in turn, into even larger groups, this process making use of large numbers of characters." Mayr, 1982:147.

In her discussion on the systematics (classification and taxonomy) of phytoliths, Mulholland (1987) stated that classification of phytoliths should follow certain principles if they were to be used for archaeology. These were that classification should be "based to a large degree, if not entirely, on phytolith morphology" and "the classification should correlate disaggregated phytoliths to the plants in which they formed, at as specific a level as possible" (1987:67). If these two conditions are indeed necessary, then what is being achieved is not a classification of plant opal but a key for the identification of plants.

There have been several phytolith keys erected in the last 10 years and all have as their aim the identification of plants (Brown, 1984; Piperno, 1987; Mulholland, 1987). However, the majority of the identification schemes which have been erected have been referred to as "classifications". Thus there are two important points which need to be stressed; firstly, there are no classifications of plant opal; and secondly, the keys which have been erected are for the identification of plants, not plant opal.

Plant opal identification

Introduction

To date, the characteristic used in plant opal keys has been morphology. Plant opal can be divided initially into two morphological classes; regular repetitive shapes (often referred to as phytoliths) which provide a cast of the cell in which the silica was deposited or formed (as in the case of spheres) as buddings off a silicified cell wall, and irregular shapes which are usually the plate-shaped encrustations of cell walls. The size of the plant opal, once dissagregated, ranges from sand to clay-size, with the majority falling into the fine-silt to clay size range (Wilding and Drees, 1973).

The subsequent classes defined may be based on the cell which the silica body occupies; epidermis, haircell, mesophyl, etc., (e.g. Piperno, 1986), or on the disarticulated shapes; dumbbell, cross, trapezoids, etc., (e.g. Brown, 1986). In most cases the former have been favoured by those entering phytolith research from the botanical direction and who are mainly interested in examining the plant opal while in place in the plant, its location, rate of growth, etc., (e.g.

Sangster, 1970a). Those who have dealt with the plant opal in its disarticulated form, for example archaeologists and pedologists (e.g. Baker 1959a, b, c), use shape classes.

The question of the relative advantages of either type of class is difficult since both have their uses. However, the obvious result is the difficulty in communication between, say, botanists and pedologists, and this is compounded by the complication of keys which are a mixture of both. Piperno (1987) for example, considered that such keys which name major categories according to their origin in the plant and adds morphological categories at a lower level, would be of more practical use; however phytoliths have a wide variety of forms, and each form can vary between plants and within the one plant in response to factors such as maturity, availability of silica, and stress. The difficulty lies in tracing the disarticulated shapes back into the plant.

The uses of dissarticulated plant opal

(i) Gross differences

The presence/absence of plant opal has been used as a climatic indicator in many studies, particularly those which deal with continental dusts found in deep sea sediments (Folger *et al.*, 1967; Parmenter and Folger, 1974; Maynard, 1976). Parmenter and Folger (1974) looked at Atlantic sediment cores containing phytoliths, identified them as being derived from grasses, and used them to argue for aridity in the Sahara during Glacials. On land, Pokras and Mix (1985) used evidence from bio-siliceous material including phytoliths in tall grass savannah in Africa as evidence for spatial variability in Late Quaternary climates.

The differences between forest and grass plant opal include the fragility of the former and the forms in the latter which appear to be specific to grasses. These properties have been used to distinguish forest/grassland chronologies and to provide supporting evidence in paleo-climatic studies (for example Witty and Knox, 1964; Verma and Rust, 1969; Wilding and Drees, 1971).

(ii) The species specific phytolith

In archaeology, the discovery of a phytolith diagnostic of cultivated cereals or vegetables has been long pursued. Pearsall (1978), used the cross-shaped silica bodies found in maize to support arguments for the cultivation of maize at an archeological site location. Cross-shaped phytoliths are in Twiss *et al's* (1969) Panicoid class (discussed below), and are common in many species of grasses. Of the grasses examined by Pearsall, four genera including *Zea* produced cross-shaped phytoliths. The percentage of cross-shaped phytoliths in each of four size categories - small, medium, large and extra large (with dimensions given), was used to separate wild grasses from maize. Nine races of maize were examined, and Pearsall concluded that maize could be distinguished by the large or extra large cross-shaped phytoliths it produced.

Moore (1978) in a letter to Nature, briefly reviewed Pearsall's work on maize phytoliths, and concluded that more extensive numerical analyses of modern maize phytoliths were required to test the reliability of the size criteria erected. Rovner (1983) also sounded a warning note, pointing out that large cross-shaped phytoliths have been found in other species, and that there is a possibility of bias due to moisture regimes. A full discussion of the maize phytolith is to be found in Piperno (1987).

Bozarth (1986) reported distinctive phytoliths derived from bean pods and the cucurbit rind which formed part of a reference collection of domestic plants made during the examination of prehistoric villages in the central plains of the United States. In particular Piperno

(1987) considered the phytoliths from the cucurbita to be distinctive at the level of genus.

In paleoenvironmental reconstructions the species specific phytolith would point to a particular species and hence environment. In the sedges the hat or cone-shaped "Cyperaceous type" of Mehra and Sharma (1965) was a conical hat on an organic pad projecting from the inner tangential walls of living epidermal cells. It has also been reported as being specific to the Cyperaceae by Norton (1967), who stated that it had not been reported outside the Cyperaceae at that time. Piperno (1987) also reported conical and hat-shaped phytoliths in the Palmae, Cyperaceae, Marantaceae and Orchidaceae. In the Cyperaceae the conical-shaped phytoliths, according to Piperno, are distinctive and separate from others:

> "All species in the Cyperaceae contribute these distinctive phytoliths, often in high numbers."(1987:93).

However, in one small sample of Australian vegetation, this shape has been found in at least one dicotyledonous species (Chapter 2).

Many researchers have reported phytoliths from grasses which are specific at least to the level of tribe (Twiss *et al.*, 1969). Piperno (1978) reported short-cell phytoliths from grass epidermis including the dumbbell, cross-shaped and saddle-shaped as having restricted distribution within the Gramineae. Other distinctive phytoliths which she disscussed included cystoliths (outgrowths from the cell walls), stegmata cells giving rise to taxinomically significant phytoliths in the palms (Palmae), phytoliths from leaves of *Musa* spp. (bananas and plantains) which may be genus specific and surface ornamentation which might be significant (Piperno, 1987:65). All such significant phytolith shapes must be viewed with extreme caution since their significance may be limited geographically, and, indeed, further examination of other species in the area may produce similar forms.

(iii) The Twiss *et al.* classification

Much work in the 1960s and 1970s was directed at attempting to establish a link between shapes of phytoliths observed in the soil and the plant from which each was derived, in the hope of being able to use unique phytolith shapes as being diagnostic of a particular plant species. A very influential paper by Twiss, Suess and Smith was published in 1969 in which a morphological classification of grass phytoliths was erected and used to distinguish between phytoliths of the short and tall grass prairies of the United States. Subsequently this was followed by a number of papers based on the classification or slight modifications of it, and it has, in more recent years, provided the basis for the establishment of diagnostic phytolith assemblages (Rovner 1983).

Twiss *et al.*'s. (1969) classification itself had its roots in much earlier work, and quotes the research of Smithson (1956, 1958), Baker (1959), Prat (1936), Metcalf (1960) and Folger, Burckle and Heezen (1967), which in turn are based on the poineer work of Ehrenberg (1847).

Folger *et al.* (1967), sampled a North Atlantic dust fall during an intense dust storm approximately 500 km from Africa. Examination of the dust collected during this storm showed that the two most abundant biogenic components were freshwater diatoms and opal phytoliths. They discussed Ehrenberg's 1847 classification of 34 forms of "Phytolitharia" which he found in dust given to him by Darwin, and went on to divide the phytoliths in the North Atlantic dust fall into 4 classes; Rod, Dumbbell, Barrel and Capstan/Hourglass.

Twiss *et al.* (1969) also looked at Smithson's 1956 and 1958 works. The 1958 work considered the relationship between grass opal and that opal appearing in the soil. In this paper, Smithson distinguished rods, hats, fans, dumbbells, and tried to distinguish between tribes of grasses. However, none of Smithson's subsequent work with Wynn Parry, which was very important to this question of identification, was examined by Twiss *et al.* For example in their 1966 paper, Parry and Smithson examined the 3-dimensional shapes of silica bodies in 31 grasses and 10 cereals. They divided the silica into silica bodies and silica not occurring as solid bodies, and sub-divided into costal rods, hats, long cells, hairs, etc. They noted the great variation in shape, not only between species, but also within species, even on the same part, and the variation in association and abundance due to maturity.

Baker's work was also referred to in Twiss *et al.* (1969). They did not, however, examine Baker's work on assemblages of phytoliths in soils (1959b). In this paper, Baker examined the phytoliths found in two soils; a swamp sediment and a basaltic soil 90 miles apart. The phytoliths from the 5 - 50 um fraction of the topsoil (0-7 in) were used, and the percentage distribution by count of shape types contrasted. Rod and nondescript forms dominated both assemblages. Baker made no attempt to examine the phytolith content of the vegetation at each site, yet reached the conclusion that any difference between the phytolith assemblages in the soils was due to differences in the vegetation, a reasoning which, although not substantiated, has been used by most workers since.

The paper by Baker referred to under the heading "Previous Classifications" in Twiss *et al.* (1969) is the paper "Fossil Opal Phytoliths and Phytolith Nomencalature" (1959c) which sets out which of those mineralised bodies in plants should be called phytoliths. The paper did not "propose a nomenclature for opal phytoliths based on the anatomy of grasses and allied plants" (Twiss *et al.* 1969:110) and Bakers subsequent classifications were not based on the anatomy of any plants.

Both Prat (1936) and Metcalfe (1960) which were cited in Twiss *et al.* (1969), recognised three major taxonomic grass groups - Bambusoid, Festucoid and Panicoid. The last two were divided into several tribes on the basis of leaf structure. Silica bodies and their arrangement in the mature leaf was only one of the properties used in classification.

The Twiss *et al.* (1969) classification was part of that erected by one of the co-authors, Suess (1966), in his Masters Thesis (which was supervised by Twiss). Suess's classification was erected by the examination of spodograms and one class at least relied on being able to observe the placement of the silica bodies in the plant. It was by no means a complete classification of all the plant opal which is to be found in grasses. Only part of the classification was used in creating vegetation regions and assigning sediment and dust phytoliths to classes - the regular silica bodies cuboid and dumbbell.

Twiss *et al.* (1969) looked at the epidermal cells of leaves in 27 common grass species and proposed 4 classes distinguishing 3 groups of sub-families of the Gramineae. They noted that

> "In grass taxonomy the shape, frequency, and distribution of silica bodies in short cells of the mature leaf are combined with other properties of the epidermis in identifying species and genera" (1969:110).

Twiss *et al.* differentiated four classes of grass phytoliths which had equal classification status despite the fact that the Festucoid appeared to correspond to a subfamily containing four tribes (Prat 1936) while the Chloridoid and the Panicoid appeared to correspond to tribes within the one subfamily, Panicoideae.

> "Because saddle-shaped bodies are distinctive and common in atmospheric dust originating in the "short grass" region of the western prairie, we have given it (the Chloridoid class) equal

rank with the other morphological classes" (1969:111).

The Bambusoid subfamily of grasses has silica bodies which resemble all of Twiss *et al.*'s types, thus while "the bamboos and related genera are clearly distinct from all other grasses" (Twiss *et al.* 1969:110), their opal bodies clearly are not. The classifications' Elongate class contained five types which were "diagnostic of the Gramineae as a whole but do not possess any subfamily or tribal characteristics" (1969:112). It is interesting to note that even in the limited number of species (27) examined by Twiss *et al.*, *Zea mays* contained both Festucoid and Panicoid silica bodies.

The classes are described in two-dimensional terms as circular, oblong, etc.; thus when actually allocating an individual phytolith to a class, a certain amount of judgement must be exercised since, for example, "In side views of individual grains, Chloridoid phytoliths cannot be distinguished from Panicoid forms" (1969:112). It should be emphasised that, in the opinion of the authors' "a morphological classification of silica bodies can be applied to grass taxonomy only with caution" (1969:112). The classification discussed three-dimensional objects in terms of two-dimensions, and when the third dimension is added (as in Scanning Electron Microscope (SEM) studies) even more problems in allocation of class occur.

Clearly then, this was a key erected to assist in distinguishing between dust sources, thus those phytoliths which were to be found in the dust were of importance. The characteristic used was shape. It by no means set out to include all plant opal, not even all grass plant opal. It was a key erected to identify dust sources, yet it has been accepted as a general classification of grass phytoliths.

Even at the time of its development the problems in this scheme were obvious. It did not include all grass plant opal; there were many anomalies apparent; and there were difficulties in assigning the silica bodies to a shape class. While there have been some acknowledgement of the problems in the scheme particularly when used outside the Great Plains of the United States (Pearsall, 1989), it has been accepted almost without question by many workers in many different fields, and has been used by others as the basis of more complex "classifications". Rovner (1983) lists many such studies. Of the ability to distinguish between grass taxonomic groups using the Twiss *et al.* classification he states that

> "This basic differentiation has been confirmed by several more recent studies of North American grasses (Blackman 1971; Bonnett 1972; Rovner 1971)" (1983:230).

It is worthwhile to go back to these works to look for this confirmation.

Rovner (1971) examined the use of phytoliths in paleoecological reconstruction. He examined reference collections of phytoliths from four deciduous trees, nine dicotyledonous herbs, five conifers, seven Gramineae (four Panicoid class and three Poacoid class [Festucoid]) and four other monocotyledons. He found that Twiss *et al.*'s oblong rectangles and oblong rectangles with sinuous margins (Festucoid) appeared in both Poacoid (Festucoid) and Panicoid grasses, and commented that many other shapes seen in his study were not included in Twiss *et al.*'s types. Even the dumbbell; "a most significant taxonomic type" was "not without its redundancies" (1971:355). Hardly a confirmation of the classification.

Blackman (1971) certainly did not agree with the classification in the way Rovner (1983) would have us believe. Rovner implies that the shapes of phytoliths alone can be used to assign each to a grass subfamily. Blackman clearly states that this is not possible.

"However, there are limitations to the use of silica bodies alone, in taxonomic and identification studies. Many diverse species contain silica bodies some of which are rather similar in shape. Such phytoliths differ only in their dimensions, and are therefore recognized only by a large number of detailed measurements. Very few grasses contain silica bodies which are unique in shape. On the other hand, silica body patterns are associated with tribes or even genera of grasses. In situations where only one representative of the tribe or genus is present, the study of the silica bodies can then be of more direct use in identification" (1970:769).

Blackman is clearly stating that it is in arrangement on the living plant that silica bodies may be used for identification purposes, and not on the individual disarticulated phytolith shapes which form the basis of Twiss *et al.*'s classification. Palmer (1976) observed that "the use of grass phytoliths presents certain limitations in that the only feature that one can ascertain with dispersed silica bodies is shape" (1976:1732). By utilising fragments of grass epidermis, she was able to see the association of bodies - the distribution and occurrence of silica bodies is important in the identification of grasses. This is a very important point which appears to have escaped most workers in this field. Never-the-less, it is one which need stressing.

Blackman (1970) also pointed to the atypical deposition of silica which may account for much of the silica content of grasses. She did, however, say that the shape and relative abundance of silica bodies in soil was useful provided a preliminary survey of the grasses concerned had been done with good results; a fairly guarded statement.

Bonnett (1972) found:

"The particles of plant opal recovered from the soil by the various investigators cited and found in soil samples examined could be identified as coming from grasses. The species represented in the samples of plant opal could be identified as to subfamily but not, with certainty, as to species". (1972:31).

Bonnett used sediment samples provided by other researchers and presented no rigorous treatment of these samples to demonstrate this contention.

(iv) Silica in the non-grasses

Less interest was shown, initially, in plant opal in plants other than grasses; possibly due to the opal being smaller, more fragile and less in quantity.

The morphology of sedge (Cyperaceae) phytoliths has been examined by Metcalf (1971), Mehra and Sharma (1965), Raeside (1970) and Ollendorf, Mulholland and Rapp (1987), all of whom confirmed the presence of a cone-shaped phytolith common to the Cyperaceae.

Silica in timber has been described variously as inclusions, aggregates, concretions, corpuscles, bodies, deposits, echinulate bodies and various combinations of these terms, e.g. "corpusculate aggregates " (see Amos 1952:7). Gonggrijip (1932) described the silicification of certain types of wood that were resistant to *Teredo* attack, and Amos (1952) tabulated more than 400 siliceous timbers belonging to 32 botanical families and recorded the presence of silica in Gymnosperms such as *Araucaria* and *Podocarpus sp*. Brydon, Dore and Clark (1963) looked at silicified asteroschlereids in Douglas Fir and identified them in soil material. In addition to the work of Amos (1952), the silica deposits in wood in Australia have been examined by Bamber and Lanyon (1960) and Scurfield, Anderson and Segnit, (1974).

Wilding and Drees (1971, 1974) used the SEM to examine forest opal in soils and obtained reference opal isolates from deciduous tree leaves. Identification of the opal was on a morphological basis. They identified rods, spheres, etc., and in some cases related them to various cells within the plants although not to a particular species. These studies demonstrated the great advantages of using the SEM in identifying the small phytoliths.

A most extensive morphological examination of silica in deciduous angiosperms was undertaken by Geis (1973), although other isolated studies had been made by Metcalfe and Chalk (1950) and Verma and Rust (1969). In Geis's study, morphological and quantitative data was given for 36 deciduous tree and shrub species occurring in forest communities in central USA. Silica in the cells of leaves was examined in the *in situ* position, and the pattern of silica deposition was found to vary widely between plant taxa and to be random in most species examined. Nearly all components of leaf tissue were present as silicified replicas, but silica was concentrated mainly in cell walls and lumina of epidermal cells, epidermal hairs and hair bases. A classification was erected based on cell type, and patterns of silica deposition examined and illustrated. Geis concluded that "comparable analogs of the epidermal cells, epidermal hairs, and hair bases of deciduous forest species are lacking among the phytoliths described for the Gramineae" (1973:118).

Silica in timber has also been examined by Welle (1976) and Kondo (1977). Welle (1976) surveyed 75 families, 440 genera and 1,300 species of neotropical woods using thin sections and the Scanning Electron Microscope (SEM). He found silica occurred in about 300 species, many of which had been considered non-siliceous. Kondo (1977) found that conifers tend to have simple, rectangular prisms or irregularly shaped polyhedron phytoliths while broad-leafed trees contained rather more diverse shapes.

(v) Recent advances

The classification of Twiss *et al.* (1969) is at the basis of all recent developments in identification and assemblage work in disarticulated plant opal. Mulholland (1982) split the classification into ten categories, Collins *et al.* (1980) into twenty-five. Mulholland's 1986 classification contained nine major categories which mixed the categories of Twiss *et al.* (1969) with those of Metcalfe (1971) and other botanical classifications.

Brown (1984, 1986) provided a Key specific to the United States Midcontinental Plains grasses. Brown (1984) noted within-species variation and suggested that phytolith analysis required larger counts and more extensive sampling than pollen analysis. He concluded that problems would continue in trying to separate species and more research was needed. The 1986 Key is an expanded Twiss *et al.* classification of eight major classes of phytolith shapes. Brown examined four sub-families, sixteen tribes, fifty-two genera, one hundred and twelve species and commented:

> There is a troublesome lack of correspondence between phytolith shape taxonomy and grass taxonomy" (1986:67).

Mulholland (1987), in her discussion on phytolith classification, pointed out that when the objective is archaeological research and one is dealing with dissaggregated phytoliths, these can only be identified through morphology and not by orientation in the plant as in Metcalf (1960). Since the object is correlation to plants, relevant characteristics need to be identified to achieve this objective.

Mulholland stated that her more recent classifications are based on the type of cell silicified - i.e. she considered that this can be identified even in phytoliths which have been disarticulated and which may have been within sediments for some time. This classification's major categories are the trichomes, stomata, buliform cells, epidermal groundmass cells, rods, rectangles/squares and silica bodies (formed inside a silica cell). This last group, the silica bodies, were then further divided on the basis of shape. Qualitative morphological characteristics only were used since Mulholland found that measurements such as length and width, when analysed statistically, yielded a continuous range of variation.

Piperno 1987, presented two types of keys in an Appendix to her book on Phytolith Analysis. The first was her own where phytoliths were keyed by their origin in New World Tropical plants, the remaining were morphological grass keys.

The specialised Keys presented were Brown's (1984) Central North American Grass Key (discussed above), and Piperno's Lower Central American Grasses Key which comprised two major classes (short cell phytoliths [crosses, bilobates and irregular short cells with three or four lobes] and mesophyll phytoliths) and is a specialised key for Panamanian Panicoid and Bamusoid grasses. In contrast to that of Brown (1984) this Key stressed three-dimensional shape, size and proportion of each class in the sample. Such keys are essentially regional, and may serve for a particular type of study only.

The General Phytolith Key for plants of the New World Tropics (Piperno, 1987) was heirarchical comprising three tiers. The phytoliths were divided into seven classes; segmented hairs, nonsegmented hairs, hair bases, cystoliths, epidermal or subepidermal tissue - phytoliths arising from solid plugs from interior of cells, epidermal - phytoliths arising as large incrustations of cell walls or chunks of silica filling the entire cell, and origin in plant tissue unknown. The tiers were:

1. Origin in plant.
2. A variety of morphological characteristics including armed/nonarmed (in the case of hairs), shape, multicelled, silicification, surface ornamentation.
3. Again, a variety of morphological characteristics as in 2.

Thus there was no consistant pattern to the morphological characteristics chosen to define a level.

The most recent developments in the field of phytolith assemblage studies are in the area of stereology. Russ and Rovner (1987) used commercially available software (computer based image analysis) to process and analyse closely related phytolith populations. To use computing power, however, there is a need to reduce the numbers of parameters examined, and this may not be acceptable with more complex shapes. The problem of placing a particular phytolith into a category was discussed by Mulholland (1986) who found that under SEM different views of the same phytolith were being classed as separate categories. Even with computer aid, this is a problem which will remain difficult to resolve. Rapp (1986) reported that the Archaeometry Laboratory was working on a morphological classification of phytoliths using a large data base and building up a computer based non-cladistic classification.

Pearsall (1989) reviewed the grass phytolith "classifications" including Twiss *et al.* (1969) and examined the limitations which have become evident in this classification when applied to areas outside that for which it was erected. It must be emphasised that all keys are, indeed, regional, and perhaps the biggest mistake a worker in the field can make is to use a key outside the region in which it was erected. This in itself leads to problems of a major kind in countries such as Australia, where very little identification work has been attempted which is not firmly based in the work of overseas researchers. Bowdery (1989) points out that fragmentation in research effort has led to a great diversification in classification and nomenclature, resulting in subjective classifications containing descriptions of shapes which may not be consistent between classifications (1989:182). Since very few people are at present engaged in any form of phytolith research in Australia, the use of phytoliths in archaeological and paleoenvironmental reconstructions is in its infancy. In particular, the time required to erect a worthwhile reference collection for any particular site, severely curtails research in this direction.

Plant opal keys and classification

While still in the plant, plant opal may be used as part of a key to identify the plant. Once it has become disarticulated and part of the sediment, any attempt to key or classify plant opal must do so on the basis of its being a soil constituent in its own right. While a key may be developed on the basis of shape alone, a classification of plant opal must utilise as many of its characteristics as possible, including elemental composition as discussed in Chapter 7.

In the course of this thesis keys for the identification of plant opal were erected. Their aim was to identify plant opal morphological "type", ultimately to construct assemblages for comparison of the shapes which survive in sediments, are affected by fire, etc. What has been attempted is not the identification of plants but the elucidation of processes in the vegetation/litter/soil complex. The keys and the analyses for which they were used are discussed in Chapter 10.

Conclusions

Although workers in the archaeological and paleoenvironmental fields have attempted to classify plant opal, these attempts have produced keys, not classifications. The reasons for this appear to lie in misconceptions as to the basis of the classification process. Phytolith keys have been largely developed for the grass families and can be shown to have their origins in the 1969 paper of Twiss *et al.* Very little work has been done outside the grasses, and few schemes for identification of plant opal take into account the vast amount of plant opal in sediments which is not grass derived.

Major problems exist in using the keys in the areas for which they were erected. These include variability of a particular shape within the one plant and between plants of the same species, and very importantly, the variation in shape of a single disarticulated phytolith when viewed from different angles (Prat, 1948:343; Parry and Smithson, 1964:173; Twiss *et al.*, 1969:112).

Even more problems arise when the keys are used outside the area for which they were erected; never-the-less studies using the Twiss *et al.* classification or derivitives thereof are to be found in the literature of areas outside the Great Plains of the United States (e.g. Hawaii; Pearsall and Trimble, 1983).

The phytolith keys which are at present in use have the primary purpose of identifying plants, not phytoliths. What is being used is disarticulated plant opal which has lost its association with the host plant. In all but a few instances (silicified stomata, for example), to be able to identify the cell from which the plant opal came is optimistic. To take this a step further and hope to identify the plant family or species from which it came is optimistic in the extreme. To some extent this has been at least partially acknowledged in the assemblage studies of recent years; but the basis of these is open to question, and will be discussed in Chapter 10.

CHAPTER 10

THE MORPHOLOGY OF PLANT OPAL

Part 2: Plant opal assemblages

Introduction

The analysis of plant opal so as to provide data which will illuminate aspects of archaeology and paleoenvironmental reconstructions is probably the most often used aspect of plant opal study. Piperno discussed the "fundamental attributes that underlie their [i.e. phytoliths] applications to Quaternary paleoecology" (1987:131). These were:

1. the irregularity of their production in the plant kingdom; however, "when secreted by plants they take on manifold shapes and sizes that are faithfully replicated in species and identifiable in the collection of phytoliths isolated from soils and sediments" (1987:132);
2. although shared shapes between related and unrelated taxa are common, "Many plants do contribute a single phytolith type recognized by distinct shape" (1987:132); and
3. phytoliths in sediments are a function of the vegetation at the site and are extremely stable.

All of these fundamental attributes and hence the value of plant opal to paleoecology can be challenged. Faithful replication is not an attribute of plant opal; on the contrary each shape is an individual, yet point 1 suggests that they are not only identical but that each shape is readily identified in sediments. Points 1 and 2 refer to species specific phytoliths, a concept which must be approached with extreme care; and point 3 is a broad sweeping statement which can clearly be refuted on the basis of the evidence provided in this thesis. While the stability of plant opal under certain circumstances is not disputed, the many processes which intervene between plant, litter and soil and which may vary the plant opal assemblage are clearly not being taken into consideration.

Phytolith assemblages for the identification of vegetation

The present emphasis in studies utilising plant opal is on the analysis and interpretation of phytolith assemblages which are used as the basis for intra and interregional comparisons of vegetation (Piperno, 1987). Piperno states that such studies involve "the tabulation and quantification in percentages, absolute numbers, or ratios of all morphological variants observed in a sample" (1987:132).

An assemblage encompasses those classes of the Key which are found in any one sample. "A plant phytolith assemblage consists of shapes present in a particular plant specimen and the relative abundance" (Mulholland, 1986:123). However, just what the soil phytolith assemblage represents is a little more complicated.

As an initial split, since grasses produce significantly more biogenic silica than forest species, this gross production difference is often detected in the soil and indicates the difference in flora. Rovner (1983) points out, however, that problems include that this observation is based on use of the silt fraction of soils while the clay fraction may contain over 75% of the plant opal; i.e. this observation may not be true for the entire soil phytolith assemblage. So the first problem encountered is that of observing the entire phytolith assemblage in soils, and, at present, there are no techniques which readily enable us to do this due to the problem of working with the clay fraction.

Sampling and processing problems are commonly encountered. Pearsall and Trimble (1983) reported on the phytolith content of fifty-eight archaeological and fourteen control soil samples from the Waimea-Kawaihae road corridor, Hawaii. Difficulties were encountered with the flocculation of clays and phytoliths; in obtaining a clean sample on centrifuging; contamination; and in interpreting the results.

Many studies which seek to identify past environments on the basis of plant opal assemblages, do so by establishing a reference assemblage from the sediment in which the vegetation of the modern analoque of that environment is growing. In so doing they assume that:

1. the modern vegetation communities are similar to those of the past;
2. the processes affecting the plant opal before and after it is incorporated into the sediment are also the same
3. and that the assemblages erected for modern vegetation communities are indeed representative of that community.

This last point indeed incorporates part of the second. What is assumed is, that for any vegetation community, given similar bedrock, climate, topography and as many other factors which can be held constant, the plant opal assemblages will be sufficiently similar to enable their past analogue to be distinguished. And, of course, the converse - that different plant communities will be readily identified.

The problem of modern vegetation

A major problem still to be adequately addressed is the relationship between the modern vegetation and the plant opal in the sediment on which it grows. Both botanical and pedological factors influence the sediment plant opal assemblage. The botanical include species diversity and proportion, relative amount of plant opal production and its size range; the pedological horizontal and vertical displacement of the plant opal and dissolution rates.

The erection of modern phytolith assemblages has often been with reference to the sediment assemblage only. Piperno states:

"The processes that underlie the production, taxonomy, dispersion, and preservation of phytoliths interact in extremely complex and yet very poorly understood ways to create what we know as fossil phytolith assemblages. Often we cannot even begin to evaluate many of the factors in the past that may have contributed to variability in plant species and plant population production, dispersion and solubility. However, it is possible to ameliorate considerably the difficulties of interpreting past phytolith records by circumventing the intermediate effects of these processes and studying only their end product. We achieve this goal by constructing modern phytolith spectra from the surficial or "modern" soils underneath different kinds of plant communities." (1987:149).

It is then assumed that by matching this assemblage to paleoassemblages that the paleovegetation community can be delineated. Obviously the modern is biased in favour of the few plants which produce copious, robust plant opal (such as the Gramineae). But this is only the botanical side - what of the pedological?

This problem has been partially addressed in some of the earlier studies which looked at gross phytolith distribution with depth. Jones and Beaver (1964a, b) related differences in plant opal content in Illinois soils to differences in internal drainage. They argued for greater silicate weathering in both well and poorly drained soils. Their findings were not verified in a similar examination of plant opal content in toposequence members in Ohio by Wilding and Drees (1971) who noted a large range around the mean obscurring any subtle differences. They did, however, equate higher quantities of plant opal with larger periods of prairie vegetation and "more stable geomorphic surfaces" (1971:1009) which one takes to mean less erosion of topsoil.

The problem of plant opal dissolution in soils has been examined (Wilding and Drees, 1974; Bartoli and Wilding, 1980; Bartoli, 1985) generally finding higher dissolution of forest plant opal when compared with grass opal due to its more fragile nature.

What has not been considered is the uneven distribution of processes both between sites and through time which may further bias the plant opal assemblage in the litter and soil. In the Australian environment these processes include fire and the action of soil fauna.

Examples of assemblage studies

Piperno (1987) provided an analysis of plant opal from a modern soil from beneath a mature tropical vegetation on Barro Colorado Island, Panama, comparing phytolith assemblages from the top-soil with assemblages from the vegetation. The site had been well researched as to species composition and abundance, and Piperno had spent seven years erecting a reference phytolith collection of over 1000 species. Vegetation variation was represented by three units; old and younger forest and swamp. In the soil assemblages swamp formation was represented by an increase in oil palm (*Elaeis oleifera*) phytoliths, disappearance of some tree phytoliths and the appearance of diatoms and sponge spicules. The question of transportation of the phytoliths across boundaries was addressed by reference to some species specific phytoliths and it was concluded that "most phytoliths do not move more than 20m from their primary depositional loci" (1987:162).

Phytolith solubility was examined by comparing the phytolith production in the vegetation with the forms recovered from the soil. Two classes of phytolith were found to be abundant in the vegetation but barely present in the soil; hair cell and hair base phytoliths. The contribution from factors such as poor preservation or low production were very difficult to unravel. Over-representation of some forms was also a problem.

Despite this, Piperno concluded that

> "phytolith assemblages from soils of a tropical moist forest exhibit close relationships to the standing vegetation, hence corresponding patterns (Webb, 1974) between modern phytolith spectra and modern plant cover have been established" (p166).

The evidence is difficult to assess, but appears to be largely based on the presence/absence of a few distinctive phytoliths. Obviously such a large study could not be presented completely and possibly it is only notable exceptions which have been highlighted in discussion. However, as presented it is not convincing. Never-the-less the study does demonstrate that an immense amount of time (and hence research funding) is needed to sample vegetation communities and that these communities need to contribute distinctive and durable phytoliths to the soil. Such a large amount of information requires sorting both by experienced workers and computer programs.

The idea that the plant opal assemblage can identify past vegetation provided the basis for the phytolith analysis of geological sediments from Panama by Piperno (1985). She examined vegetation changes from 11,300 BP to the present by analysis of the phytolith assemblages in a core from Gatun Lake, and identified mature tropical forest, mangrove, fresh water swamp and slash and burn agriculture.

In this study, the phytolith assemblages representing different lowland tropical vegetation communities were distinguished. Piperno then claims

> "They can then be applied to the interpretation of future phytolith sequences from geological sediments in the tropics and used as general indicators of different types of vegetation." (1985:13)

Twenty samples from 4 different cores were processed. Phytoliths in the cores were placed into groups by reference to a modern comparative collection of tropical species, using shape and surface decoration. However, Piperno lists distinctive forms from various families, and then goes on to assess the changes in the cores by reference to these distinctive forms.

For example, during the first period examined, 11,300 BP to 9,000

BP, the phytolith samples contained an abundance of shapes considered indicative of the Palmae and Marantaceae families. Grass and sedge phytoliths, again distinguised by distinctive forms, were not observed "a pattern that one would expect from a mature forest situation" (1985:15).

> "This is then what a tropical forest can look like in phytolith diagrams - high numbers of palm and Marantaceae phytoliths, fewer forms from other forest species, and rare grass phytoliths." (1985:15).

While the main findings of the study were confirmed by supporting pollen evidence, the evidence from the phytoliths is not convincing by itself. All of the assumptions listed at the beginning of this section have been made in this study, yet there is little evidence which supports them. In the very simple grassland communities in the Great Plain of the United States where these ideas originated in the work on grasses (Twiss *et al.*, 1969), such relationships may be locally sustainable. Forest communities are much more complex, their production of robust forms is less, and many of the comparisons between the modern vegetation and the ancient are based on assumed distinctive and unique morphology or distinctive life forms such as diatoms. The pollen evidence may be very necessary to confirm these trends.

Phytolith assemblages from archaeological samples were compared with surface phytolith assemblages in a study from Hawaii (Pearsall and Trimble, 1983). The control samples from modern surface soils were correlated to known vegetation communities by way of surface pinch samples. The analysis of phytolith percentages from the pinch samples was used to erect a model which was then used to interprete the phytolith assemblages in the archaeological columns. The basis of comparison was the shift from non-grass to grass large-cell phytoliths which was dependent on canopy cover, and the short-cell grass phytoliths which were related to the grass vegetation. This use of short-cells was found to be problematic for interpretation.

The study concluded that the percentage of grass short-cells in the soil sample is not directly translatable into percentage cover of grass species. It also pointed out that the lack of short-cells and dominance of large cell non-grass phytoliths in an archaeolgical sample could be interpreted either as arid or semi arid conditions, or open, bare ground.

The question arises in all such studies as to the representativeness of any reference phytolith assemblage. Given several similar sites under one set of environmental conditions and vegetation communities, are the assemblages erected at each site indeed similar (and how is this measured)? Is this assemblage different from those of other vegetation communities in the same environment? These are some of the questions which were explored in Part 1 of this thesis.

Plant opal assemblages

Rovner commented that the

> "fundamental usefulness of opal phytolith analysis is obviously predicated on the ability to identify and, if possible, quantify the original flora that produced a soil phytolith assemblage." (1983:228)

It is, however, possible that plant opal assemblages without any floral implications, can shed light on processes in the soil, litter and vegetation.

The Keys erected in the course of this thesis were primarily based on the requirement that they be used for the identification of plant opal morphological types in sediments; i.e. disarticulated opal shapes. A morphological approach was taken, ultimately, after around six attempts over four to five years, in a Key which, in most classes, reflected shape and a non-botanical background. The exception to this was the prickle or hair class. These are readily identified as such in sediments, thus the terms have been retained. Other classes have their origin in the morphological classes of other authors'; for example the lobates, saddles, double outlines and cones of grass keys, all of which are obvious in sediments. All comparisons between assemblages have been made using the same Key.

Two Keys are used in this thesis. The first was erected for the initial examination of assemblages at Oxford Falls, and as such was based only on the shapes observed in the samples. The final arose out of many subsequent attempts, and was used for all of the remaining morphological work.

There is no large background reference collection since the plant opal was not being referred back to plants. Comparisons between sediment and litter assemblages constituted the main use of the Keys and the question of species assemblages has been examined once only; in the acacia. The main Key was erected on the basis of some 800 SEM micrographs of plant opal from many species and sediments in the field areas and after several years of both SEM and light microscopic examination.

The first Key

Only plant opal actually observed in the samples examined were placed in the Key. The shapes of plant opal were described as follows:

1. Sphere aggregates

   Many smaller spheres making up a spherical aggregation of around 10 um in diameter.

2. Spheres

   Single spheres up to 10 um in diameter.

3. Compound spheres

Groups of two or more spheres growing out of each other.

4. Cups

    Concave bodies, 2-10 um in diameter which often contained a sphere or spheres.

5. Stomata

    Silicified stomata.

6. Fan-shaped

    Solid fan-shaped plant opal.

7. Rods

    Rods 1-15 um in width and ranging considerably in length. These were further divided according to surface or shape characteristics into smooth, marked, dendriform or spiked.

8. Sheets

    Divided into thin (<1 um) and thick (>1 um) sheets and further divided on the basis of shape and surface characteristics. Thin sheets were scrolled (rolled up), plain or bulbous surfaced; thick sheets were regular or irregular in shape.

Orientation of the plant opal

Parry and Smithson pointed out that if the dumb-bells in leaves became displaced, "the impression that each consists of a linkage of two spheres is seen to be incorrect" (1957:975). In their paper dealing with opal shapes in British grasses (1964) they emphasised the necessity of considering three-dimensional shape, presenting several models of phytoliths demonstrating their three-dimensional shape in relation to the leaf surface. Blackman (1971), presented drawings of silica bodies as seen in surface preparations showing their outline shapes. She used two outlines - one representing the outer edge of the body and the inner line representing the inner edge. Ridges along the inner surface, since they appeared as a line down the centre in surface view, were represented by such a line, and represented the area of greatest thickness of silica. Oblique views of the bodies were also presented in some cases.

Twiss *et al.* (1969) presented their classification on the basis of the outline which appears when the silica body is as it occurs in the leaf (i.e. as a two-dimensional shape). While the three-dimensional shapes and the problems they presented were becoming well known, the shape defined in disarticulated silica bodies was with reference to the growth position, which required a certain familiarity with this and the ability to be able to identify each disarticulated silica body with its position in the plant. While this may have been possible for some grass silica bodies, in particular the grass short-cells, when description of unknown bodies is required this presents problems.

This problem has been to some extent eased by more rigorous definitions of the orientation of plant opal in the disarticulated form. Mulholland (1989) outlined such an approach. She defined the *base* as the largest broad, relatively flat face, and the side opposite the base, no matter what its shape, as the *top*. When the plant opal had its base or top parallel to the microscope slide it was in *top view* or *planar view*. She defined the *cross-section* as the outline of the body in top view. The *sides* were the longest pair of opposite faces connecting the top and base, and the *ends* the other pair. Thus a body can be oriented in *top view, planar view, side view* or *end view.*

Mulholland then went on to define 3 main groups of silica-bodies according to their rectangular or square base cross-section shape (lobates, etc.), as short cylinders or truncated and beveled cones (her rondels) and the distinctive three-dimensional shape of the saddle.

These descriptions are for the identification of silica bodies in grasses. However, a similar approach might be taken in describing all silica. In the following Key, the orientation of the three-dimensional bodies where necessary is described as in Mulholland (1989), that is in top, planar, side or end view. The cross-section is also described.

The final Key

The final Key, which was used throughout most of the thesis apart from the initial study at Oxford Falls, comprises an aggregational or exclusive hierarchy in three sections.

The classes in the first section comprise those shapes which have generally been considered to be grass derived. However, while it is difficult to shake off the genetic implications of these shapes, no such implication is made here, particularly since the aim of the key is not to identify plants but morphological type. The main classes, lobates (further subdivided), saddles, double outlines and cones have not been further subdivided on a basis other than overall shape, since they were not observed to be ornamented as were the remaining classes. This lack of ornamentation may be of considerable importance.

In the second section each class is divided into three levels. The first is based on basic three-dimensional shape; rods, spheres, etc. The second is based on shape attributes also; thick or thin, single or compound, etc. The third level is based on ornamentation of the surface; smooth, ridged, etc. The exception in the shape classes are the hairs and prickles which are botanical in origin, but readily identified as such. The third section comprises two classes; the unclassified opal and opal of other biological origin. The unclassified opal comprises pieces of opal which do not readily fit into the classes above. Because of the two and three-dimensional sheet classes, these are few and are usually broken pieces of other classes. The opal of biological origin comprises the diatoms, etc.

The aim of the Key was to identify plant opal of a morphological type. The requirement to identify plant species, which is common to all other plant opal Keys, was not a consideration.

Plant opal Key for the identification of morphological type

1. **Lobates**:

Plate 1A-F

Cross-section: lobate; side-view and end-view: truncated pyramid

Bilobates (dumb-bells): Two lobes of equal size connected by a shank.

Polylobates: Three or more lobes of the same or various sizes connected by a shank.

Crosses: Four equal sized lobes connected to a shank.

Lobate are in Twiss *et al's.* (1969) Panicoid class, and are generally considered to be derived from the leaf epidermis of gramineous plants; a silica body. Sangster (1968), examined lobates in three-dimensions and confirmed the earlier model of Parry and Smithson (1964). The term "polylobate" is derived from Brown (1984), and "crosses" from Pearsall (1978).

2. **Saddles**

Plate 1G

A solid body with two convex edges; cross-section has two opposite convex edges and two opposite concave edges.

In Twiss *et al's.* (1969) chloridoid class, [see also Brown (1984) and Mulholland (1987)].

3. **Double Outlines**

Plate 1H

Solid bodies having either a central depression or raised top giving a double outline in top view. The term is from Brown (1984).

4. **Cones**

Plate 2A-C

Solid body with circular or eliptical basal cross-section, rising to one or more apice. Side-view truncated cone or cone.

Cones are the hats and scutiform opals of Parry and Smithson (1964, 1966). Twiss *et al.* (1969) placed these in the Festucoid class; the cross section is circular to oval, (the "rondels" of Mulholland (1987) and in three-dimensional shape they are cone-like.

5. **Rods**

Plate 2D-H, Plate 3A-G

Three dimensional elongated body with straight or near straight sides. Rectangular in cross-section; the length is at least twice the width. The end-view is circular to non-circular.

Thick (>5um diameter), circular and non-circular end-view and Thin (<5um diameter)

Ornamentation:

smooth: No ornamentation

spiked: Regular or irregularly spaced thin protruberances on surface.

jigsaw: Thick protruberances in line down sides, often in two opposing lines which then interlock with neighbouring rods.

ridged: Thick or thin ridges running diagonally around the rod.

rough: Small protruberances or indentations completely covering the rod.

Platey rods: Flat elongated bodies

Ornamentation: Jigsaw (as above) and sinuous (at the edges adjoining the adjacent rod).

These are silicified long cells in grasses, silicified bundle sheath parenchyma cells and vascular elements in forest species (Wilding and Drees, 1974). In grasses, Parry and Smithson commented that the rods may be "trough- or boat-shaped (1964: 172). The platey rods are the long cells of Parry and Smithson (1964).

6. **Sheets**

Plate 3H, Plate 4, Plate 5A-C

Tabular in shape. Cross-section if regular will be rectangular, triangular, etc. End and side-views are rectangular or trapezoidal. May comprise part of one or more than one cell and may have various ornamentation on each piece.

2D, regular and irregular shapes. Thin (<2um thickness).

Ornamentation:

honeycomb:	Irregularly and regularly shaped depressions on the plate. Includes ridged, sinuous cell walls.
plain:	No ornamentation but may have a rough surface on a very small scale.
perforated:	Regularly sized and placed holes through the sheet.
bulbous:	Tabular body with one or more bulbous projections on the upper surface, often each projection surrounded by smaller "satellites".

Sheets 3D, regular-shaped (bulliform) and irregular shapes. Ornamentation may be smooth, rough or honeycomb as described above.

Sheets, 2D, are continuous sheets of opaline silica often showing impressions of long cells such as wavy patterns, etc., (Parry and Smithson 1966). Piperno (1987) described polyhedral and anticlinal epidermal phytoliths.

Sheets, 3D, are bulliform cells in the grass leaf whose function could be motor or water storage. Regular 3D shapes are also present in large numbers in forest species, although their function is not known.

7. **Hairs or prickles**

Plate 5D-H, 6A

long (>30um), short (<30um), and

gourd-shaped.

These are the trichomes of Parry and Smithson (1964, 1966), and Bonnet (1972). They comprise macro-hairs (unicellular) and micro-hairs (bicellular). Prickle hairs are hookshaped or straight (after Metcalfe 1960). Epidermal hairs are often hollow socketed.

8. **Spheres**

Plate 6B-H, Plate 7A

Basic shape is spherical in any orientation.

Single and Compound. These range in diameter from >1um to 50um with an average diameter of between 10 and 20um. Compound spheres are groups of two or more spheres which appear to be either attached at the circumference or growing out of each other. While many groups are composed of spheres of approximately the same size, some comprise individuals of varying sizes.

Ornamentation:

Smooth: No ornamentation.

Spiked: Slender elongated protruberances over surface.

Rough: Roughened surface.

Indented: Small well spaced pits over surface.

Verrucose: Wormlike protruberances all over.

Spheres were described by Wilding and Drees (1974) as "vesicular infillings in cell lumen or vesicles budding from a silicified cell wall" in deciduous tree leaves (1974:296). Those with verrucose surfaces include cystoliths; spherical and elongated silicified growths from epidermal and sub-epidermal cell wall with a nodular surface.

9. **Unclassified (fragmented) plant opal**

Plant opal which is recognisable as such, but not placed in any of the above classes although it largely includes fragments of them. Usually not many pieces of opal are placed in this class as the 3-dimensional and 2 dimensional sheets contain subclasses into which most will fit.

10. **Other biogenic opal**

Plate 7B-H

Biogenic silica from a known scource other than plants: diatoms, sponge spicules, etc.

In this thesis, the key outlined above was used as a basis for the erection of assemblage curves which would be representative of all of the plant opal shapes in the sample being examined, and which could be compared with other, similarly erected curves. The aim of the assemblage studies was to examine similarities and differences between the litter and soil plant opal in samples under various vegetation communities, and thus to evaluate the basis upon which phytolith assemblage studies are erected; and to examine the use of plant opal for examining processes in the litter and soil.

PART 3

IMPLICATIONS

"Accordingly I assert that we do not start from observations but always from *problems* - either from practical problems or from a *theory which has run into difficulties.* Once we are faced with a problem, we may begin to work on it. We may do so by attempts of two kinds: we may proceed by first attempting to guess or conjecture a solution to our problem; and we may then attempt to criticize our usually somewhat feeble guess. Sometimes a guess or conjecture may withstand our criticism and our experimental tests for some time. But, as a rule, we soon find that our conjectures can be refuted, or that they do not solve our problem, or that they solve it only in part; and we find that even the best solutions - those able to resist the most severe criticism of the most brilliant and ingenious minds - soon give rise to new difficulties, to new problems. Thus we may say that *the growth of knowledge proceeds from old problems to new problems, by means of conjectures and refutations.*"
Popper, 1972:258.

CHAPTER 11: OLD PROBLEMS

Introduction

The starting point for this study was a body of literature on the subject of plant opal; mainly written this century in English, although it included a few French, German and Japanese papers. As outlined in Chapter 1, the literature clearly divided the study of plant opal into three areas - botany, archaeology and paleoenvironmental research, and, to a lesser degree, pedology. While a line of enquiry branching out into each of these areas could be traced back to a few early papers, it became most obvious that, although some research had been conducted into agricultural and pedological aspects of plant opal in Australia in the 1950s and 1960s, there was no unbroken thread of plant opal study in this country as there was in others.

Research

While much of the pioneering work into the physical characteristics of plant opal was undertaken in Australia during the 1960s (Handreck, 1968; Handreck and Jones, 1967; 1968; Jones and Handreck, 1965; 1969), this work was not followed up as similar research was in, for example, the United States, by interest from other researchers. Most of the important botanical research was done, and continues to be done, overseas (e.g. Smithson and Parry, 1958-66; Sangster, 1968-91; etc.). Most of the archaeological research is centred on the United States (e.g. Rovner, 1983); some pedological work is being undertaken in Japan (e.g. Kondo, and Sase, 1986) and Europe (e.g. Bartoli, 1985). In a review of microfossil characteristics for use in archaeology in Australia, Selkirk and Adamson pointed out that:

> "the production of phytoliths by local plants and their distribution in sediments is also a neglected field of research" (1982:198).

Some Australian researchers have utilised plant opal in overseas projects, for example in Papua New Guinea (Wilson, 1985) and in Africa (Williams, Assefa and Adamson, 1986); however, the concentrated research efforts in the archaeological area in America, for example, have no equivalent as yet in Australia, although this applied research area is beginning at the Australian National University (Bowdery, 1984, 1989).

The research areas have tended to become more specific through the years, with the problems each area examines becoming further isolated. What they have in common, however, is that although the characterisics of plant opal (refractive index, specific gravity, mineralogy, thermal properties, colour,) were initially explored, there has been one characteristic overwhelmingly emphasised, and that is shape. The botanists are looking at how opal is laid down in plants and the archaeologists and environmentalists are attempting to use shape to identify past vegetation. With only a few exceptions, the use of plant opal in the field of pedology or indeed in the earth sciences is not considered.

The aims of this thesis

In Chapter 1 it was suggested that research into the three current areas of investigation into plant opal was still in its infancy due to fragmentation of effort, technical difficulties, its costliness in terms of both time and money and unawareness as to the potential of plant opal studies. Two initial areas of research in Australia were proposed; the examination of the relationships between vegetation communities and opal assemblages in the soil, and the cycling of plant opal through the biosphere and soil.

This thesis aimed to examine current trends in plant opal research overseas and their applicability, and to evaluate the use of disarticulated plant opal as a sediment constituent in its own right. Part 1 outlined the field studies undertaken to achieve these goals while Part 2 outlined the field and laboratory techniques used in the analysis of plant opal assemblages and the question of plant opal keys. In Part 3 the main points arising from the studies are reiterated and their wider implications examined in more detail.

The initial problem

Very few scientists in any field in Australian science are familiar with plant opal. The lack of continuing research into plant opal in Australia means that the opportunity to see plant opal, apart from in photographs and micrographs, does not exist. One must, as it were, start from the beginning with extraction techniques. While it was clear from the work of Baker, Jones, etc., in the 1950s and 1960s that Australian plants contain plant opal in much the same range of shapes and in the same sort of quantities as those from elsewhere in the world, very little subsequent work, in the light of current ideas, has followed in Australia.

While it is becoming obvious from the trends in the literature that the species specific phytolith is at best a localised phenomon~~a~~, the idea that an assemblage of phytoliths representing a particular vegetation community could be erected is being actively pursued overseas, despite the basic assumptions on which this idea rests appearing to have been little investigated. This thesis questions these basic assumptions and examines the uses of plant opal outside the fields of archaeology and paleoenvironmental research.

Initial observations

The three initial studies (Chapter 2) were implemented in order to make comparisons between the plant opal reported in the literature and the plant opal present in the Australian enviroment.

The first study examined the relationships between the plant opal assemblages in a swamp sediment and the assemblages in the dominant plant species growing in and around the swamp (Hart, 1988a). It demonstrated the complexity of such comparisons, even in simple communities, and pointed to the presence of processes within the sediment leading to the survival of some morphological forms being favoured above others. It also demonstrated that a phytolith considered species specific to the Cyperaceae was present in several of the species surrounding the swamp and led to a further examination of this phytolith in other species, particularly the *Acacia* (Hart, 1990).

The examination of the Cyperaceae type phytolith in the second study illustrated the need for very detailed descriptions of phytolith morphology, particularly if they are purported to be diagnostic of a vegetation type and are to be used for this purpose. Published micrographs of the Cyperaceae type phytolith (Figure 2.3) and descriptions of the phytolith may not be sufficient.

Chapter 2 demonstrated the problems in plant opal studies and in particularly in importation of ideas from other environments. What is not widely enough understood is that the Australian environment is indeed different. Separation from the influence of the northern hemisphere occurred sufficiently far back in geological history for Australian fauna and flora to follow developmental lines unlike those of any other continent. For example, of the more than 1,200 species of *Acacia*, more than 700 are native to Australia. Pedley (in Simmons, 1981) described the "rather tight knit 'Australian' group of *Acacia*;

> "In recent classification *Acacia* has been regarded as consisting of three subgenera. One has bipinnate leaves with spines in their axils. The flat-topped trees associated with the giraffes and elephants in the African landscape are *Acacia* of this subgenus, but there are also species in Asia, South America, and perhaps a dozen in northern Australia.......The second subgenus consists mainly of prickly woody vines or small trees widely

> spread in the tropics but with only one one Australian species.....The rest of Australian species belong to the third subgenus, members of which usually have no thorns or prickles and leaves modified to phyllodes.........A few species of the group are found outside the Australian region..."(Pedley, 1981:10).

Thus, any future botanical work in plant opal within the plant must proceed on the basis that the plant opal produced by Australian species may differ significantly in morphology from similar species elsewhere.

The initial observations at Oxford Falls were made to examine the basic question of shape in relation to species and sediment. The results led to a range of problems emerging, which required further consideration:

1. The whole question of plant opal morphology including classification, keys, species specific phytoliths, etc.
2. Why were there differences between the shapes present in the vegetation and the shapes present in the sediment? What processes were responsible for these differences? Was the difference due to the vegetation or to processes occurring before or after the plant opal was incorporated into the soil and would this affect the assemblage idea's basic premises?
3. How much silica is cycled through the terrestrial biosphere? How is it distributed in the soil?

The differences between the vegetation and sediment plant opal assemblages were then explored in the Pilliga field sites. The plant opal cycle was entered in the litter storage and investigated to see if the litter plant opal assemblages were different from those of the sediment, exploring by the way the basic premises raised by the assemblage ideas. The questions of how much and where were a matter of observation and measurement.

Assemblage Studies

The hypothesis that, holding other environmental factors constant, like plant communities produce like plant opal assemblages in the sediments in which they grow, is at the basis of many of the paleoenvironmental reconstructions utilising plant opal (e.g. Piperno, 1985). This hypothesis was tested in the Pilliga State Forests (Chapter 5).

A plant opal assemblage consists of those shapes present and their frequency. The problems surrounding classification of plant opal and the erection of keys was discussed in Chapters 9 and 10. The erection of a plant opal Key for the identification of plants is usually based on many years of careful study into the morphology of the plant opal in the plants in the particular area in which the work is being done. Such keys are user-oriented and regionally specific.

This is an area of research which is in its infancy elsewhere in the world. Most of the studies thus far incorporate the information from phytolith studies with that obtained from a parallel pollen study (Piperno, 1985) or with other cross-disciplinary evidence (Pearsall and Trimball, 1983). The plant opal evidence doesn't come from the assemblages themselves, but from the morphology of phytoliths within the assemblages which experience in the environment has led to the belief are specific to species in the extant vegetation (Piperno, 1985). Thus the identification of plant communities is based on the special cases, not the complete assemblage in these instances. What is not realised is that once the plant opal has left the plant, it's botanical basis is lost.

<u>In this thesis the plant opal assemblages were being erected to examine similarities or differences in plant opal **shapes**, not **plant**</u>

communities, thus the botanical background was not essential. What was being examined was the reproducibility of and differences in the assemblages, and assessment of this could be based on a key erected to identify plant opal morphological type.

The reproducibility of assemblages between samples is an important consideration in this work. The assessment done (between two upper broom plain Site 7 samples) showed that at this level of complexity the assemblages erected were very similar (similarity expressed statistically using the Kormogorov-Smirnov two sample test on pairs of cumulative frequency curves). Similar tests showed that, between the four broom plain, the degree of likeness between the plant opal assemblages in the topsoil differed. While one possible explanation which might be suggested for this is a difference in species distribution between the communities which necessitates a more rigid definition of "like" vegetation; the point to be made here is that while such definitions are able to be made for extant vegetation, the difficulties in doing so for past vegetation communities are very great indeed, if not impossible.

However, another possibility for the differences between the broom plain assemblages was that processes such as those initiated by fire or fauna might change the assemblages and indeed might do so differently from site to site; and it was this idea which was explored in the later part of Part 1. This required a change of approach to plant opal, and the implications of this are further explored in Chapter 12.

Conclusions

The use of plant opal as a research tool for the identification of plants and hence environment is at present the main area of plant opal research outside the strictly botanical. There a several points to be made in evaluating this research:

1. What is not being realised is that there is no classification of plant opal. Many schemes purporting to be classifications exist, due to a basic misunderstanding of the classification process. A classification of plant opal must incorporate <u>all</u> plant opal and use many characteristics, not just shape.

2. The erection of plant opal keys which concentrate at present on the grasses, is user specific, or at best area specific, and probably must remain so. The erection of a large number of keys is the outcome of such fragmentation, with the result that cross-related research is at best difficult. Assemblage studies, based as they are on regional plant opal keys, are also specific to an area. In addition, the more complex the key the more complex the resultant assemblage studies will become.

3. The present use of plant opal assemblages to identify plant communities relies on the presence within the assemblage of the "special case"; the supposedly species specific phytolith or the biological opal from diatoms, sponges, etc. The assemblage as an object of study, is not being utilised.

4. The use of a plant assemblage from the sediment under a vegetation community as an modern analogue for use in the identification of past vegetation communities is based on the supposition that the past and present plant opal assemblages are related in a consistent manner, and this has been shown to be unsustainable. It has been shown in this thesis that the plant opal assemblages from the topsoil under like vegetation communities are not alike, and that the plant opal assemblages from the topsoil under differing vegetation communities are not sufficiently dis-similar. No account is being taken of the

variation in processes in the plant/litter/soil complex both between sites and through time nor of the problems in identifying the host plant or community from the disarticulated plant opal in the sediment.

It is this last point which is the most crucial, since it is based on observations which clearly indicate these problems.

> "Observations play, however, an important role as *tests* which a hypothesis must undergo in the course of our [critical] examination of it. If the hypothesis does not pass the examination, if it is falsified by our observations, then we have to look around for a new hypothesis. In this case the new hypothesis will come after those observations which led to the falsification or rejection of the old hypothesis. Yet what made the observations interesting and relevant and what altogether gave rise to our undertaking them in the first instance, was the earlier, the old [and now rejected] hypothesis."
> Popper, 1972::346

CHAPTER 12: NEW PROBLEMS

Introduction

The differences between plant opal assemblages from the litter and the underlying topsoil pointed to some process or processes within this area capable of selectively removing certain types of plant opal. The analysis of this problem involved a shift in the view held of plant opal, with a consequent change from the problems formerly examined to a new set of difficulties.

Rovner defines opal phytoliths as "inorganic biogenetic plant particles of microscopic size" (1971:343); Bartolome *et al.* defines them as "microscopic, translucent particles that occur in plants and persist for long periods in soils" (1986:217). The emphasis in these and indeed all definitions, apart from on the smallness of the particles, is on their origin in the plant. It is this view of plant opal which produces a barrier to thinking of it as having significance as an independent soil constituent of some importance. As such, plant opal may provide illumination on problems other than those concerned with plants. It was this alternative view of plant opal which coloured the process studies in Part 1.

The cycling of plant opal

Investigation of the plant opal cycle began with the measurement of the amount and morphology of plant opal entering the cycle via the litter layer in three vegetation communities in the Pilliga State Forests. When the litter collected in the trays was considered, around 1% of the total litter production by dry weight was found to be plant opal. The proportion of grasses and herbs in the layer under 10 cm height added another 10% of that weight, thus, while the plant opal contribution of trees and shrubs is considerable, it is the degree of grass cover which is important as far as total plant opal available in any site is concerned. In the Pilliga, the more open forest (Site 12) which supported a greater variety of herbs and grasses than either the mallee (Site 8) or the broom plain (Site 7), produced the most plant opal available to enter the soil.

The comparisons of assemblages at each site provided interesting common shifts between the litter and soil which pointed to processes in the litter layer or the topsoil. A breakup of the opal is to be expected and was demonstrated; however the process which may be of most importance in the Australian environment in removing plant opal before it has a chance to enter the soil is initiated by fire.

The role of fire in the cycling of plant opal

Researchers are becoming aware that the role of fire in the Australian environment is both dominant and underestimated. While its role in the production of aspects of the Australian flora is widely known, fire's lesser known effects include those which contribute to the formation of soils. Mitchell and Humphreys (1987) showed that the formation of litter dams after fire plays an integral part in the formation of texture contrast soils (see also Appendix A). One of the effects of fire is to reduce the litter layer and part of the canopy (depending on the fire's intensity) to ash and charcoal which is then acted upon by both wind and rain.

Previous work on the effects of fire in the litter layer and the underlying upper topsoil had been undertaken in the Sydney area by Blong *et al.* (1982) and Mitchell and Humphreys (1987). Observation in these studies centred on the effects of fire on slope processes. Blong *et al.* measured sediment yield after a moderate bushfire and highlighted the magnitude of the loss. They noted the large amount of charcoal which was removed from the slope during the first few rain events. Mitchell and Humphreys examined the movement of the sediment on hillslopes after fire, concentrating on the morphology and composition of the litter dams and the sequence of events which are common under these circumstances, and demonstrating a process by which the the topsoil extending down to several centimetres below the surface on a slope may move. This introduction of processes initiated by fire into the Bishop *et al.* (1980) model for the formation of texture contrast soils which has been well explored in Eastern Australia, is important and far reaching in its effects.

The Ecology Reserve study

The vital period for the litter layer is during the fire and in the first rain event after fire. Observations and sampling in the Ecology Reserve Site demonstrated that the removal of plant opal in the fine ash by wind and water, and in the charcoal by slopewash, has important consequences in-so-far as the amount and morphology of the remaining plant opal is concerned.

The amount of plant opal in soils has been used to provide an estimation of the age of the soil (Jones and Beavers, 1964; Wilding, 1967; Kondo, 1988). In some estimations, the amount of plant opal in the soil is divided by the annual production rate to give an annual accumulation rate for the soil or sediment (Kondo, 1988). Kondo compared the ^{14}C ages of soils (quoted from Kata *et al.*, 1986) with ages obtained using this method and found this showed comparatively good agreement up to 5000 y B.P. However, the problems inherent in this method include the calculation of the amount of plant opal in the soil given the problems with isolating it from the clay fraction, and the processes such as fire which might remove large quantities of vegetation and hence plant opal.

The mobility of plant opal

The lateral mobility of plant opal in dust, water, etc., has long been recognised. Some researchers have considered that, in the area in which they are working, the lateral movement of plant opal may be only up to a few metres (e.g. Piperno, 1987). Piperno stated that the geological phytolith record may be fairly localised since she considered long-distant transport of phytoliths not to be a problem except under certain conditions where they might become a major component of dust. What is not realised is that this question of lateral mobility becomes important if selected shapes are moved as has been demonstrated in this thesis.

Vertical displacement has only been suggested by "nonphytolith specialists" who have

> "not benefitted from sufficient practical experience with phytolith extractions from soils and correlations with other site data to offer judgements on phytolith movement. Just as seriously, they are not aware of or have not carefully evaluated the very substantial body of evidence accumulated over the last 30 years showing that phytolith illuviation is not a problematic factor" (Piperno, 1987:147)

Piperno referred to the work of Jones and Beavers (1964), where plant opal found at considerable depth (to 30 in) in a profile was considered to be due to the nature of loess deposition, and to the vertical stability of phytoliths in the archaeological context. However, the possibility of the vertical movement (pervection) of plant opal over a short distance in a very short time frame has been demonstrated (Chapter 4) and such mobility, even over a short distance, is capable of biasing the plant opal record.

The mixing by soil fauna is a process which also cannot be ignored. The movement of plant opal by soil fauna is selective since parts of plants or particular species which they find palatable are moved into the areas of the soil they occupy; that is their nests, faunal channels and sheeting in the case of termites as was demonstrated in Chapter 4. These activities may lead to different plant opal assemblages occurring in various soil layers leading to interpretation problems. Thus, the actual location within a layer where samples are removed for plant opal analysis is of importance in material where species such as termites are operating. This is a particular problem which may prove to be crucial in the Australian environment where termites are capable of moving vast quantities of vegetation into their nests, utilising their faecal material in their nest and channel construction and depositing faecal pellets in their channels. While this might be an inconvenience in archaeological research in areas where soil fauna are highly active, such activity might be turned to advantage where the effects and influence of soil fauna are being studied.

More far reaching consequences

The observations outlined above have opened up several interesting areas of investigation. All are interconnected and stem from the nature of plant opal itself. The characteristics of plant opal include its shape and those mineralogical, chemical and physical properties which stem from its composition. Shape has been used to produce phytolith keys whose purpose is to identify plants or communities of plants. This has been, indeed, the only use to date for this attribute.

Shape was used in the main part of the investigations conducted in this thesis, but in an entirely different way. The keys erected had a purpose which had very little to do with identifying plant or vegetation communities. Shape was used in the keys to identify plant opal morphological types. This is a very important distinction. The end product was a shape which had certain characteristics, which, although very simple, could be used to erect assemblages of plant opal morphologies. The differences between the assemblages demonstrated the effects of processes going on in the litter and soil. The plant opal was used in process studies.

A study of the morphological types present in the litter and underlying soils in the Pilliga demonstrated that the assemblages of plant opal they produced differed (Chapter 5). The differences included those which could be attributed to the mechanical breakdown of plant opal, and those for which no immediate explaination offered itself such as a reduction in platey morphologies between the litter and topsoil. While such morphologies could be broken down to a size smaller than could be detected, or dissolved, the possibility that processes within the litter layer and soil selectively removed these morphologies was explored. The processes which have been shown to be initiated by rain following fire (sheetwash moving ash and charcoal on hillslopes) were considered to be a possible source of this selective removal.

As was discussed by Mitchell and Humphreys (1987), transportation in slopewash moves graphite and mica on hillslopes where fire has removed the retarding effects of vegetation. These are minerals with platey morphologies; they have a very low sphericity thus transportation, which tends to sort material according to size, shape and specific gravity, selectively removes them. Plant opal is also a mineral, often having low sphericity and a lower specific gravity than, say, quartz. In particular, much of the plant opal is platey in morphology, and when exposed on a slope after fire, acts as any other platey mineral when transported in slopewash.

The morphological types present in the plant opal assemblages on the soil surface immediately after a fire and at successive intervals for the following month were examined in Chapter 6. In addition to overall comparisons between the assemblages, the surface area of platey plant opal material was compared. This study demonstrated a reduction through time of the more platey plant opal morphologies which were exposed on the surface by the fire in ash and charcoal, and that this removal was initiated by rainwash on the slope during rain. The reduction in the amount of plant opal available for incorporation into the soil was considerable. The study demonstrated the details of a stage in removal of material from a hillslope after fire which was described in other studies. Blong *et al.* (1982) and Mitchell and Humphreys (1987) both recorded layers of ash and charcoal immediately after bush fires which swiftly disappeared after rain. This layer has now been shown to include much of the plant opal material which had been accumulating within the litter on the slope since the last fire, rendering it unavailable for incorporation into the soil.

But shape is only one attribute of the plant opal. The remainder hinge on its composition, and the properties stemming from this have been used to identify the material as opal (mineralogical properties) and in separation techniques (specific gravity). Very little use of them has been made except for dating (electron spin resonance, thermoluminescence, etc.). Techniques used in this thesis for the separation of plant opal from sediments have been critically examined and the loss of plant opal during separation discussed. The future refining of these techniques to avoid such losses is of considerable importance.

The variability in properties of plant opal such as refractive index and specific gravity are well documented (e.g. Peinemann, Tschapet and Grassi, 1970; Jones and Beavers, 1963). These variations are due to composition, and may tell us something about the host plant growth conditions (sediment composition, nutrients). The composition of plant opal has been examined (e.g. Jones and Milne, 1963), but the advent of instruments capable of more detailed analysis presents an opportunity to explore the elemental composition of plant opal as a characterisitic which may serve to facilitate their use as a "finger-print" in sediment.

The use of plant opal to "fingerprint" a sediment; to give it a unique signature which enables it to be traced stratigraphically; is not possible if morphology alone is used. The contemporary topsoil on one hillslope in the Pilliga is one stratigraphic unit (Chapter 3 and Appendix A), but the plant opal assemblages within it were shown to vary. While the variation was in some part due to the differences in contemporary vegetation which ranged from broom plains through mallee to forest, the differences in processes at each site also lead to differences in plant opal assemblages. The broom plains had areas of more recent fire than the mallee and the forest, and the mallee soil fauna were very active. Thus, using the shape characteristics of the plant opal alone will not serve to fingerprint the sediment. What is needed is an examination of a characteristic of the plant opal other than morphology.

Such a characteristic must be capable of grouping the plant opal from one sediment body apart from the plant opal from another sediment body. To date, the elemental composition of plant opal appears to present the most fruitfull line of enquiry in this respect (Chapter 7). Too much emphasis on morphology in contemporary plant opal studies has led to the other properties of opal such as their elemental composition or other uses for their morphological characteristics becoming of minor importance. One of the major problems preventing such use of plant opal is the problem of isolating a pure sample; yet there exist methods which need to be explored and in which a cross-disciplinary approach is required (Chapter 7).

Conclusions

This thesis presents an alternative way of looking at plant opal; an earth science view. It accepts the status of plant opal as a mineral in its own right, and acknowledges that, once it is in the sediment, its botanical ties become attenuated. The large contribution plant opal makes to the soil is not fully understood and the opportunities it presents as a component of soil and other sediments are yet to be realised. As a soil constituent, the variety in morphology alone affords opportunities for plant opal to be used in process studies.

The importance of fire as a factor influencing processes in the Australian environment is only slowly being realised. Its importance as a modifier of nutrient cycles has been well documented, but the silica cycle is little considered. Fire removes a large amount of the plant opal from the local cycle and transfers it elsewhere in the environment. It is selective in its effect, changing both the amount and morphology of the plant opal entering the soil. In addition, the difficulties in concentrating plant opal in sediments for examination is compounded by the prevalence of carbon coatings on the plant opal, causing its specific gravity to vary enormously. This is a problem which must be taken into consideration, particularly in the Australian environment.

The mobility of plant opal is affected by other processes including pervection through soil voids, particularly those created by soil fauna and the periodic dessication of soils. While the distance moved by the opal may not be great, it is a process which, in some instances, needs consideration. The movement of plant opal by soil fauna has also been demonstrated. This too has far reaching consequences on the nature of assemblages and the concentration of plant opal in various parts of the soil. The use of plant opal in examining the ecology of soil fauna is an area of research which might be profitable. The closer study of other plant opal properties will, in the future, suggest many other uses.

Implications for research

During the period of renewed research into plant opal which took place in the 1950s and 1960s, Australian researchers made valuable contributions to the investigation of plant opal characteristics. However, possibly due to the need to concentrate their research efforts elsewhere in a continent having such unusual flora, a short European history and a small population, the botanical aspects of plant opal in the Australian flora have been almost totally neglected. The possibility that such studies may open up new areas of botanical research is very strong.

While the implications for botanical research are positive, those for the user-oriented applications are not. The connections between the extant vegetation, the plant opal within a sediment and the past vegetation are too tenuous. However, the possibility of using plant opal as a marker of processes between plant, litter and soil has not been observed before. This presents an exciting new area of earth science research.