

CHAPTER 4

MATERIALS AND METHODS

4.1 Introduction

A wide range of materials and methods were utilized in this research programme. Many glasshouse and field experiments shared common materials and methods. This chapter aims to facilitate ready access to the various methodologies employed. Where these were common to two or more experiments they are discussed in the general materials and methods rather than documented under specific experiments.

4.2 Possible Sources of Variation Between Glasshouse and Field Experiments

Glasshouse pot trials were designed to examine *inter alia*, substrate and species responses under optimum soil moisture conditions, and enhanced humidity and temperature using sieved substrate materials. In this context, pot trials have historically been considered useful guides to spoil management (Howard *et al.* 1977, Puchko 1977) as well as more traditional forms of soil management. Field experiments are generally designed to further clarify or confirm glasshouse results under variable field conditions and to develop broad scale, practical techniques. Consequently, it is important to recognise early in the research programme where possible sources of variation between glasshouse and field experiments are likely to occur.

Glasshouse experiments described in this study ran for a maximum of 184 days. While such studies have the advantage that variation due to environmental factors is considerably reduced, extrapolation of data to the field must be undertaken with caution, for several reasons.

Bell (1985) indicated that, for nutritional studies, one must firstly distinguish between the nutrient requirements for germination of

native plants, their establishment requirements and their maintenance needs. Glasshouse nutritional trials provide information on the needs of native species in the germination and early establishment phase; maintenance requirements are more satisfactorily obtained from field trials.

A second factor mentioned by Bell (1985) is that glasshouse experiments are conducted under optimum moisture regimes with pots being watered regularly to field capacity. Field conditions are generally substantially drier. It can be expected, therefore, that the growth rate and yield potential achieved in glasshouse trials may not be matched in the field. This factor will tend to decrease the amount of nutrient which must be added in the field to achieve maximum yields. On the other hand, leaching losses in the field could mean that higher levels of nutrients may need to be applied to achieve the same response as in glasshouse trials. In either case it could be expected that the relationship between glasshouse and field nutrient applications will depend on rainfall in the field and could vary for different substrates as a result of different substrate / water characteristics. For example, the high percentage of rock in many spoil materials can increase available water in the short term although, in the long term, water holding capacity would be reduced (Wilson 1985). The amount of rock present in substrates can vary considerably between substrates in the field and also between the field and glasshouse, the latter having a reduced rock component due to preparatory sieving.

The third factor which Bell (1985) considers must be taken into account in extrapolating pot trial results is the difference in fertilizer distribution between the pot and field. In all glasshouse trials undertaken in this study, the nutrients were mixed in solution and applied uniformly to the pot surface. All solutions were absorbed by substrates and no immediate loss occurred. In direct seeding field trials, fertilizer was generally broadcast on the surface in granular form and, in some cases, cultivated into the top 20 cm of spoil. Consequently, the availability of nutrients to newly germinated seedlings could vary considerably. Nutrient availability in the field may also be affected by surface drying which may restrict root growth in the surface layers. Nutrient availability could be

further affected by the tendency of nutrients, particularly surface applied P, to be bound in the top 2-3 cm of many spoil materials (Elliott 1982) and high rock volumes, which would tend to concentrate applied nutrients due to the reduced soil volume available to roots.

Although field trials ran for considerably longer periods than glasshouse trials, they were still of a relatively short duration (1-2 years) and care must be taken in predicting long-term growth from early response data.

Variation in seed lots both within and between glasshouse and field experiments can also occur as a result of inbreeding effects which are common in *Eucalyptus* spp. (Eldridge and Griffin 1983).

4.3 General Materials and Methods

4.3.1 Glasshouse Experiments

4.3.1.1 Nature of the Experiments

Table 4.1 provides a brief description of each glasshouse experiment, together with mean day-time and night-time temperatures and the source of spoil material.

Pot experiments can be divided into four groups. Experiments 1 and 2 examined seed germination and early growth. Experiment 3 contained no plant material and examined physical and chemical changes in spoil material over time. Experiments 4, 5 and 6 examined the early growth of planted seedlings. Experiment 7 examined the selectivity of pre-emergent herbicides on the germination of native species. Results and discussion are presented in these groupings.

Table 4.1 Description of glasshouse experiments together with mean day-time and night-time glasshouse temperatures, source of materials and duration of each experiment.

Exp. No.	Description	tpho ⁺ (°C)	tnyc ⁺⁺ (°C)	Source of Substrates	Duration of Exp. (Days)
1.	N x P interaction Trial (Seed Germination)	29	22	H.V.No.1 Mine	98
2.	Soil Conditioner Trial (Seed Germination)	24	16	H.V.No.1 Mine	184
3.	Effect of Different Watering Regimes on the Physical and Chemical Characteristics of Substrates (No Plant Material)	30	19	H.V.No.1 Mine	11
4.	Nutrient Omission Trial (Seedlings)	31	24	C.S.R. Lemington Mine	126
5.	Nutrient Omission Trial (Repeat of Exp. No. 4 using different substrates and only <i>E. maculata</i> seedlings)	30	19	H.V.No.1 Mine	126
6.	N x P interaction Trial (seedlings)	24	16	C.S.R. Lemington Mine	120
7.	Pre-emergent Herbicide screening Trial (Seed Germination)	25	14	Ravensworth No. 2 Mine	169

⁺ $tpho = tmax - \frac{1}{4} (tmax - tmin)$

⁺⁺ $tnyc = tmin + \frac{1}{4} (tmax - tmin)$ (Richards 1985)

where $tpho$ = mean daylight temperature during the experiment

$tnyc$ = mean night-time temperature during the experiment

$tmax$ = mean daily maximum temperature during the experiment

$tmin$ = mean daily minimum temperature during the experiment

4.3.1.2 Potting Materials

Experiments 1, 2, 3 and 7 were conducted using free draining 200 mm or 150 mm diameter black polythene pots. Experiments 4, 5 and 6 used a closed pot system wherein each 200 mm diameter pot was lined with two plastic kitchen-tidy bags.

In each experiment, all pots were filled with the same weight of freshly sieved spoil. Only material passing through a 5 mm sieve was used in the experiments. For all experiments, a fresh batch of each substrate was collected and sieved at the mine prior to return to the glasshouse for potting. Samples of sieved material were taken prior to potting for physical and chemical analysis (see Chapter 5).

4.3.1.3 Plant Material

For experiments 1, 2 and 7 fifty seeds of either *E. maculata*, *C. glauca* or *A. salicina* were sown in each pot. A template was used to ensure that the seed was evenly distributed and sown to a depth of 2 mm. Seed viability data are shown in column 2 of Table 4.2.

For experiments 4, 5 and 6 ten seedlings of either *E. maculata* or *C. glauca* were planted into each pot. A template was used to ensure the even placement of seedlings. Seedlings were approximately 30 days old and approximately 6 cm high at planting and had been germinated and grown in standard Forestry Commission germination boxes under standard glasshouse conditions in the nursery. Dead seedlings were replaced for up to seven days after transplanting into pots.

Table 4.2 Field sowing rates and general seed viability data for native species used in glasshouse and field experiments and broadacre trials.

Experiments commenced in 1985		Field sowing details					
Species	No. viable seeds/kg	Field sowing rate (kg/ha)	No. viable seeds/ha	No. viable seeds/plot (10 m ²)	Field Exp. No.	Field Trial No.	Glasshouse ** Exp. No.
<i>E. maculata</i> *	101,500	1.28	130,000	130	1,2,3		1,2
<i>E. punctata</i>	168,500	0.38	65,000	65	1,2,3		
<i>E. cladocalyx</i>	130,050	1.00	130,000	130	1,2,3		
<i>A. salicina</i>	8,546	0.92	7,860	8	1,2,3		
<i>C. glauca</i> *	390,957	0.33	130,000	130	1,2,3		1,2
Experiments commenced in 1986							
<i>E. maculata</i> *	143,000	1.00	143,000	143	7	1,2	7
<i>E. maculata</i>	143,000	1.28	183,000	183	4,5		
<i>E. punctata</i>	7,000	1.00	7,000	7	7	1	
<i>E. punctata</i>		0.38	2,660	3 approx	4,5		
<i>E. cladocalyx</i>	94,000	1.00	94,000	94	4,5	2	
<i>A. costata</i>	66,200	1.00	66,200	66	7	1	
<i>C. glauca</i> *	822,500	0.30	247,000	247	7	1,2	7
<i>C. glauca</i>		0.33	271,425	271	4,5		
<i>A. saligna</i> *	19,481	1.00	19,481	N/A		2	7
<i>A. longifolia</i>	50,634	0.50	25,317		7	1	
<i>A. salicina</i>	8,546	0.92	7,800	8 approx	4,5		
<i>E. crebra</i>	325,000	0.5	162,500	N/A		2	
<i>E. gomphocephala</i>	68,000	1.0	68,000	N/A		2	
<i>E. tereticornis</i>	301,000	1.0	301,000	N/A		2	

* Used in glasshouse experiments at 50 seeds per pot

** Field data not relevant

Note: Seed viability data supplied by the Forestry Commission Seed Store, Coffs Harbour or Kershaw's Pty. Ltd., Sydney.

4.3.1.4 Fertilizer, Watering and Randomization

The potted substrates were lightly watered one hour prior to planting or sowing. Immediately after planting or sowing, fertilizer was applied in solution. The pots were again watered and watering continued as required to ensure optimum germination and/or growth.

For seedling experiments, fertilizer was added in three equal split dressings at 0, 6 and 12 weeks after planting into pots. All fertilizer was applied immediately after sowing in germination experiments. Analytical reagent grade chemicals (Selby) were used in all experiments.

All pots were re-randomized twice per week, and were kept weed free.

4.3.1.5 Monitoring, Harvesting and Analysis

For germination experiments germination normally commenced after approximately one week and plant emergence was recorded daily. Each newly emerged seedling was pegged with a toothpick to assist recording. At harvesting both the total number of germinants and the number of surviving germinants was expressed as a percentage and calculated as follows:

$$\text{Germination \%} = \frac{\text{Total number of germinants} \times 100}{\text{Number of seeds sown}}$$

$$\text{Survival \%} = \frac{\text{Number of surviving germinants at harvesting} \times 100}{\text{Number of germinants}}$$

Germination energy was also calculated and expressed as an index (Maguire 1962) as follows:

$$\text{Germination energy} = \frac{G_1}{Y_1} + \frac{G_2}{Y_2} + \dots + \frac{G_n}{Y_n}$$

where G = Number of seeds germinated on that day.

Y = Number of days since sowing.

At harvesting seedlings were separated from substrate by careful hand washing. Shoot sections were then cut off at the cotyledons. Roots and shoots from each treatment were then separately bagged and dried in a mechanical convection oven at 60° for 48 hours before weighing. *C glauca* seedlings were examined for root nodules.

For all experiments analysis of variance was undertaken as indicated in individual experiments, using the Statistical Analysis System (SAS) analysis of variance programme (SAS Institute Inc. 1985).

The following variables were analysed for variance where appropriate:

1. Germination per cent
2. Survival per cent
3. Germination energy
4. Total root dry weight (Total root dry weight in each pot measured)
5. Total shoot dry weight (Total shoot dry weight in each pot measured)
6. Total plant dry weight (Sum of 4 and 5)
7. Average plant dry weight (Total plant dry weight ÷ no. of plants in pot)
8. Root to shoot ratio
9. Average seedling height (Individual seedlings measured)
10. Spoil crust strength
11. Spoil pH
12. Spoil conductivity
13. Spoil gravimetric water content

4.3.2 Field Experiments

4.3.2.1 Location and Description

The location and description of each field experiment is given in Table 4.3. With the exception of experiment 6, all the experiments

Table 4.3 Location and description of field experiments and broadacre field trials

Experiment No.	Location (Colliery)	Description
1.	Hunter Valley No. 1 (H.V. No. 1)	Site preparation / mulch exp.
2	H.V. No. 1	Soil amendment exp.
3.	H.V. No. 1	Fertilizer rate / pellet exp.
4.	H.V. No. 1	Grass competition / herbicide exp.
5.	H.V. No. 1	Sowing time exp.
6.	Ravensworth No. 2	Pre-emergent herbicide exp.
7. *	Bloomfield	Japanese Millet competition exp.
Broadacre Field Trial		
1.*	Bloomfield	Comparison of growth of sown and planted trees together with a comparison of germination and growth on two substrates
2.	C.S.R. Lemington	Comparison of germination and growth on two substrates

* located in lower Hunter Valley

examined germination and early seedling growth. Experiment 6 examined the effect of pre-emergent herbicides on planted seedlings.

4.3.2.2 Experimental Design

Unless otherwise stated, all the experiments were laid out as fully randomized, factorial designs with three replications corresponding to blocks. For germination experiments individual treatment plots were 5 m x 2 m (10 m²) in size. All plots were separated by a 2 m buffer strip within blocks, and by 4 m buffer strip between blocks. All treatment plots were individually labelled after pegging. Planted seedlings were laid out as described in individual methodologies.

4.3.2.3 Site Selection and Details

Many of the experiments were replicated on a variety of substrate materials. Although frequently separated by up to one kilometre on individual collieries, an attempt was made to select sites with comparable aspect and slope. However, on some sites, e.g. at Hunter Valley. No.1, no such options were available.

4.3.2.4 Plant Material

In field experiments 1 to 5 a standard seed mix was applied and comprised seed of *E. maculata*, *E. punctata*, *E. cladocalyx*, *C. glauca* and *A. salicina*. Sowing rates and general seed viability data are shown for all experiments in Table 4.2.

Seed for each plot was mixed with fertilizer and further bulked immediately prior to sowing and thoroughly mixed with two litres of fine bran to assist even hand broadcasting. Where other additives were applied, e.g. in experiment 2, these were also thoroughly blended with the above mixture.

Seeds of *Acacia* were pre-treated by soaking in boiling water for one minute, 12 hours prior to sowing.

Six month old seedlings grown under standard nursery conditions were used in experiments 6 and broadacre field trial 1.

4.3.2.5 Fertilizer

Experiments 1, 2, 4 and 5 received standard dressings of N and P each at 10 kg ha⁻¹ as commercial grade fertilizers. Nitrogen was applied as ammonium nitrate (Nitram) while P was applied as single superphosphate. Other experiments received specific fertilizer treatments which are described for each experiment. Fertilizer was broadcast onto the surface of all plots and not incorporated by cultivation with the exception of field experiment 7.

4.3.2.6 Site Preparation

Field experiments 2, 3, 4 and 5, were cultivated with an agro-plough one week prior to sowing. Other experiments were deep ripped and/or scarified, as described.

4.3.2.7 Maintenance

Weed emergence, particularly on topdressing material was common. However, over the duration of the experiments all weeds were controlled by chipping or by the careful application of Roundup[®] (glyphosate). None of the experiments was artificially watered.

4.3.2.8. Monitoring and Analysis

Seedling emergence and mortality were assessed at regular intervals. Each newly emerged seedling was recorded and marked with a 20 cm white painted meat skewer. If the seedling died, the skewer was removed and the loss noted. Germination per cent was calculated as follows:

$$\text{Germination \%} = \frac{\text{Total number of germinants at final measure} \times 100}{\text{Number of viable seeds sown}}$$

Survival per cent was calculated as follows:

$$\text{Survival \%} = \frac{\text{Number of surviving germinants at final measure} \times 100}{\text{Number of Germinants}}$$

Germination energy was calculated in the same manner as in glasshouse experiments (4.3.1.5) with the exception that germination was measured monthly instead of daily. Consequently, field germination energy was measured over a much longer period than for glasshouse experiments.

Survival %, stem height and/or stem basal area at 2 cm above ground level was measured two years after sowing for experiments 1, 2 and 3 and one year after sowing or planting for experiments 4, 5, 6, 7 and broadacre trials 1 and 2.

Per cent ground cover was assessed for experiments 4, 6 and 7, as described in specific methodologies.

Rainfall and Stevenson Screen temperatures were recorded at Hunter Valley No. 1 Mine for the duration of each experiment. These results are shown in Table 3.3 (rainfall) and Table 3.4 (temperature).

Ground temperatures and gravimetric moisture contents were measured for particular field experiments and to characterize broad substrate types.

Two methods were used to measure above and below ground temperatures:

1. Above ground. Fixed maximum / minimum Brannan thermometers attached to a wooden peg 10 cm above ground level and housed inside a ventilated screen. Range = - 50°C to + 50°C.

2. Surface and below ground. Stem thermometers. Tel-tru thermometers (Model GT100R) with 20 cm stems. Range = - 10°C to + 110°C placed on the surface (unshaded) or inserted at 45° to horizontal to a depth 2 cm below ground surface. Stem thermometers were allowed to equilibriate for five minutes before an initial reading was taken. Temperatures were taken at various time intervals as described in specific materials and methods.

Gravimetric moisture content was measured by taking a core of soil to a depth of 2 cm using a 2.5 cm diameter glass tube. The sample was sealed and returned to the laboratory where it was weighed and then dried in a Gallenkemp Oven at 105°C for 24 hours. The sample was then re-weighed and the gravimetric water per cent calculated following Corbett (1969):

$$\text{Grav. H}_2\text{O}\%* = \frac{\text{Weight of Soil Wet} - \text{Weight of Soil Owendried} \times 100}{\text{Weight of Soil Owendried}}$$

* (Corbett 1969.)

For all experiments, analysis of variance was undertaken using the SAS analysis of variance programme (SAS Institute Inc. 1985).

The following variables were measured in the field where appropriate:

1. Germination per cent
2. Germination energy
3. Survival per cent
4. Plant height (cm)
5. Basal area (cm²) at 10 cm above ground level

For each experiment average germination energy, average height and average basal area were first determined by dividing the totals for each plot by the appropriate number of trees. This was done for all trees and on an individual species basis. Because variables 3 and 5 were not normally distributed a log_e (X + 1) transformation and an arc sine (or

angular transformation) were respectively applied. Unless otherwise stated the seven variables thus analysed were :

- (1) Germination per cent
- (2) Survival per cent
- (3) Average germination energy
- (4) Average height
- (5) Average basal area
- (6) Log (to base e) transformation of average basal area plus one
- (7) Angular transformation (arc sine) of percentage of germinated seedlings surviving at final measure.

Analyses were done (a) on a per tree basis ignoring species (i.e. means over all species) and (b) on an individual species basis (i.e. means for each species).

Because these experiments are field trials at different sites (different substrates types) the analysis in all cases has been treated as a nested or hierarchical one (i.e. like a split plot) with substrates coming first, then treatments and then (for analysis (b)) species. For Field experiment 1, because site preparations are also split plots, there is a further hierarchy, i.e. substrate / site preparations / mulch type / species. For Field Experiments 1, 2 and 3 heavy rain damaged some plots four months after sowing. Consequently, the damaged area of each plot, expressed as an estimated percentage of the total plot area, was used as a pseudo-variate in a covariance analysis in order to compensate for the effect of storm damage.

4.4 Specific Materials and Methods

4.4.1 Glasshouse Experiments

4.4.1.1 Glasshouse Experiment 1 - Effect of Nitrogen, Phosphorus and Substrate on Germination and Early Growth

Design and Treatments. A single replicate 2 x 3 x 4² factorial design including :

- (i) 2 species :- *E. maculata* and *C. glauca*
- (ii) 3 substrates :- Topdressing (1), Sandstone (2), Shale (3)
- (iii) 4 rates of nitrogen (N) as ammonium nitrate at: nil (N0), 5 (N5), 10 (N10) and 20 (N20) kg N ha⁻¹
- (iv) 4 rates of phosphorus (P) as sodium dihydrogen phosphate at: nil (P0), 5 (P5), 10 (P10) and 20 (P20) kg P ha⁻¹

The experiment comprised a total of 96 pots.

Analyses of *E. maculata* foliar nutrient levels for N, P, Ca, Mg, K, Na, Mn, Zn and Fe were undertaken for the treatments. NO P0, N5 P0, N10 P0, N20 P0 and N20 P20 at the Forestry Commission's Wood Technology and Forest Research Division. Seedlings from each pot were bulked prior to analysis.

The experiment was sown on January 25th, 1985, and was harvested after 98 days.

4.4.1.2 Glasshouse Experiment 2 - Effect of Soil Amendments on Germination and Early Growth

Design and Treatments. A 2 x 3 x 5 factorial design with two replications including:

- (i) 2 species :- *E. maculata* and *C. glauca*
- (ii) 3 substrates :- Topdressing (1), Sandstone (2), Shale (3)
- (iii) 5 soil amendments :-
 - Gypsum (CaSO₄) at 3 t ha⁻¹
 - Gypsum at 6 t ha⁻¹
 - Alginure[®] at 0.75 t ha⁻¹
 - Terrasorb 600[®] at 1 t ha⁻¹

- Control

The composition of each material is as follows :-

Gypsum - 20% calcium (Ca) as calcium sulphate (Ca at 0.6 and 1.2 t ha⁻¹ which is equivalent to gypsum at 3 and 6 t ha⁻¹ respectively)

10% sulphur (S) as calcium sulphate (S at 0.3 and 0.6 t ha⁻¹ for gypsum at 3 and 6 t ha⁻¹ respectively)

Terrasorb - Water absorbing granules consisting of hydrolyzed starch polymers

The material has claimed aerating and soil conditioning capability.

Alginure - An organic colloid derived from seaweed with the following composition :

<u>Element</u>	<u>ppm</u>	<u>kg ha⁻¹ (approx.)</u>
Calcium	19,400	14.6
Potassium	19,800	14.9
Nitrogen	14,670	11.00
Sodium Chloride	27,000	20.3
Phosphorus	2,600	2.0
Aluminium	4,620	3.5
Magnesium	2,156	1.6
Potassium Iodide	700	0.5
Iron	220	0.16
Manganese	38	0.03
Zinc	42	0.03
Copper	4	negl.
Molybdenum	1	negl.
Vanadium	1	negl.

All soil amendments were applied in granular form, evenly spread over the surface of the pot and lightly worked into the surface of a pot to a depth of 5 mm.

Fertilizer (N10 P10) was added in solution immediately following sowing at 10 kg ha⁻¹ each of N and P in the form used in glasshouse experiment 1.

Sowing occurred on March 24th, 1985 and the experiment was harvested after 184 days.

4.4.1.3 Glasshouse Experiment 3 - Effect of Different Watering Regimes on the Physical and Chemical Characteristics of Substrates

To ascertain how seedling germination is affected by soil physical and chemical properties in the one experiment is difficult due to the need for destructive sampling. The approach adopted of drawing relationships between germination responses observed in glasshouse experiment 1 and trends in the following experiment where soil samples were subjected to similar moisture regimes was considered a reasonable compromise. This approach has been previously used by Emmerton (1983). The assumption inherent with the approach is that the seedlings do not affect soil properties within the duration of the experiment.

Design and Treatment

The experiment was designed as a split-plot over time. A three replicate 2 x 3 x 6 factorial experiment incorporating two soil moisture regimes, three substrates and six sample times using 150 mm top diameter, free draining, black plastic pots containing the same substrates used in glasshouse experiments 1 and 2.

Two moisture regimes were imposed. All pots were brought to field capacity on day 0. Half the pots were then allowed to dry without further watering. The other half were brought to field capacity at approximately 5 p.m. daily for the first five days of the experiment. Water was applied to moist filter paper placed on the surface of each pot in order to minimize surface disturbance. The filter paper was removed immediately after watering.

Measurements were undertaken at approximately 8 am on days 0, 1, 2, 4, 7 and 11. Measurements were taken immediately prior to watering up to day 5. Pots were re-randomized daily.

Materials and Methods

Each pot was filled with 1.7 kg of substrate at field moisture content. All substrates were obtained 'fresh' from site as described in Section 4.3.2. Fertilizer was not applied.

The pH of local town water at commencement of the experiment was 7.9; electrical conductivity was $7.7 \mu\text{S cm}^{-1}$.

The soil strength at the surface was used as a measure of soil crusting. Measurements of soil crusting, gravimetric soil moisture, pH and electrical conductivity were taken at approximately 8 am on days 0, 1, 2, 4, 7 and 11. An additional measurement of pH and conductivity was taken prior to wetting (day -1). Particle size analysis was taken prior to wetting and after 11 days.

All measurements were taken on surface material to a depth of 5 mm. The gravimetric moisture content sample was obtained by gently screwing a 25 mm diameter glass tube into the surface and removing a 5 mm deep core. Samples used to measure pH, electrical conductivity (EC) and particle size analysis were obtained by stripping the entire surface using a fine chisel to a depth of 5 mm and thoroughly mixing the stripped material.

An index of surface strength was obtained prior to surface stripping using a pocket penetrometer ('Soil Crete') with a 5 mm diameter flat tip which was inserted into the soil to a depth of 5 mm. Five measurements were made on each pot on a random grid basis, avoiding edge effects near the perimeter of the pots.

As the sampling methods for penetrometer resistance and soil moisture were destructive, a new pot was used for each substrate, for each watering regime and for each time interval. Consequently, the experiment consisted of a total of 18 crust strength and moisture readings for six time intervals; a total of 108 pots. Surface hardness and gravimetric moisture were analysed using the SAS analysis of variance computer program (SAS Institute Inc. 1985). Since pH and conductivity were not replicated, the three-way interaction term was taken as an estimate of error and the analysis done on this basis. Due to insufficient replications, analysis of variance was not undertaken for particle size analysis.

The experiment ran for an 11 day period between 15th March and 25th March, 1986.

4.4.1.4 Glasshouse Experiment 4 - Effect of Omitting Essential Nutrients - C.S.R. Lemington Colliery

Design and Treatments. A single replicate 2 x 3 x 9 factorial experiment incorporating:

- (i) 2 species :- *E. maculata* and *C. glauca*
- (ii) 3 substrates :- Topdressing (10), Sandstone (11), Shale (12)
- (iii) 9 fertilizer treatments :-
 - (1) All nutrients minus nitrogen (minus N)
 - (2) All nutrients minus phosphorus (minus P)
 - (3) All nutrients minus potassium (minus K)
 - (4) All nutrients minus magnesium (minus Mg)
 - (5) All nutrients minus sulphur (minus S)
 - (6) All nutrients minus calcium (minus Ca)
 - (7) All nutrients minus the trace elements boron, copper, molybdenum, zinc, manganese (Bulked - minus Trace)
 - (8) All nutrients (All)
 - (9) No nutrients (None)

Nutrients were applied in three split applications at 0, 6 and 12 weeks after planting.

Total amount of elements applied were as follows :

Nitrogen as ammonium nitrate at 100 kg N ha⁻¹
 Phosphorus as sodium dihydrogen phosphate at 50 kg P ha⁻¹
 Potassium as potassium chloride at 75 kg K ha⁻¹
 Sulphur as sodium sulphate at 80 kg S ha⁻¹
 Calcium as calcium chloride at 75 kg Ca ha⁻¹
 Magnesium as magnesium chloride at 50 kg Mg ha⁻¹
 Boron as sodium tetraborate at 0.5 kg Bo ha⁻¹
 Copper as copper sulphate at 5 kg Cu ha⁻¹
 Molybdenum as sodium molybdate at 1 kg Mo ha⁻¹
 Manganese as manganous chloride at 25 kg Mn ha⁻¹
 Zinc as zinc sulphate at 5 kg Zn ha⁻¹

The experiment commenced (seedlings planted into pots and fertilizer applied) on February 12th, 1985 and seedlings were harvested after 126 days (18 weeks).

4.4.1.5 Glasshouse Experiment 5 - Effect of Omitting Essential Nutrients - Hunter Valley No. 1 Colliery

Design and Treatments

Materials and methods for this experiment were similar to those for glasshouse experiment 4 with the exception that only *E. maculata* seedlings were used. There were 2 replicates. The experiment ran for 126 days.

4.4.1.6 Glasshouse Experiment 6 - Effect of Nitrogen, Phosphorus and Substrate on Early Seedling Growth

A single replicate 2 x 3 x 4² factorial experiment incorporating:

- (i) 2 species :- *E. maculata* and *C. glauca*
- (ii) 3 substrates :- Topdressing (13), Sandstone (14), Shale (15)
- (iii) Fertilizer at the following rates :-

Nitrogen as ammonium nitrate at 0 (NO), 50 (N50), 100 (N100) and 200 (N200) kg N ha⁻¹. Phosphorus as sodium dihydrogen phosphate at 0 (PO), 25 (P25), 50 (P50) and 100 (P100) kg P ha⁻¹

The experiment comprised a total of 96 pots.

Seedlings were planted into pots on February 24th, 1985. Final measurements were taken after 120 days, at which stage all seedlings were harvested.

4.4.1.7 Glasshouse Experiment 7 - Effect of Two Pre-emergent Herbicides on Germination and Early Growth

A 3 x 5 factorial experiment replicated four times as follows:-

- (i) 3 species :- *E. maculata*, *C. glauca* and *A. saligna*
- (ii) 5 herbicide treatments :-
 - Diphenamid[®] (NN-dimethyl-2,2-diphenylacetamide) at 7 kg and 14 kg active ingredient (a.i.) ha⁻¹ (D7, D14 respectively).
 - Surflan[®] (3,5 - dinitro - N4N4 dipropyl sulphanilamide) at 8 l and 16 l ha⁻¹ (S8 and S16 respectively)
 - Control

Surflan (oryzalin) is a member of the dinitroaniline group of herbicides and inhibits both root and shoot growth of many species when absorbed by the roots. The herbicide acts by restricting cell division. The inhibition of root growth is a direct effect and the most observable symptom is the inhibition of lateral root formation. The inhibition of

shoot growth following root absorption is probably a secondary effect caused by limited root growth. Surflan has been effectively used in the control of grasses but does not control broadleaved species.

Surflan is active through the soil but needs to be applied before seed germination.

Persistence in the soil can vary up to twelve months but can vary depending on whether conditions are anaerobic or aerobic (Ashton and Crafts, 1973).

Diphenamid (amide) is a member of the amide group of chemicals. It is used as a pre-emergent herbicide and when applied to the soil inhibits seed germination and early seedling growth, mainly by inhibiting root elongation. It has been shown to have an inhibiting effect on RNA synthesis. If the susceptible seedlings do emerge from the soil they are either greatly stunted and/or malformed. These effects are probably due to an interference with cell division and/or cell enlargement (Ashton and Crafts, 1973). Like Surflan, Diphenamid has been traditionally used in the control of grass species. It has little effect on broad leaved species.

The seeds of each species were sown in Topdressing (16) which was obtained from the topdressed field site at Ravensworth No. 2 Mine. The pots were watered and herbicide was then applied in solution in 40 ml of water per pot. The experiment commenced on February 20th, 1986 and was harvested 169 days later.

Data for variables root weight, shoot weight, total weight and germination energy were subject to a $\log_{10} (x + 1)$ transformation for the purpose of analysis. Data for variables germination per cent and survival per cent were arc sin transformed before analysis.

Foliar N and P concentrations and the N:P ratio for *E. maculata*, (Nil and D7) and *A. saligna* (Nil and D7) were undertaken.

4.4.2 Field Experiments

4.4.2.1 Field Experiment 1 - Site Preparation / Mulch Experiment

A three replicate $3^3 \times 5$ split, split plot design including:

- (i) 3 substrates :- Topdressing (1), Sandstone (2), Shale (3)
- (ii) 3 site preparation treatments (main plots) :-
 - ripping
 - cultivation
 - control
- (iii) 3 mulch treatments (sub plots) :-
 - chitter (coal washery reject material) at 60 t ha^{-1}
 - baled oaten straw at 3 t ha^{-1}
 - control
- (iv) 5 tree species (*E. maculata*, *E. punctata*, *E. cladocalyx*, *A. salicina*, *C. glauca*)

Ripping consisted of two riplines along the length of each plot approximately 1.2 m apart and to a depth of 0.7 m. Cultivation was undertaken using a specially developed agro-plough to a depth of 0.15 m. Plates 4.1 and 4.2 show site preparation treatments.

Seed was sown on May 9th, 1985. Mulches were spread evenly by hand. Site preparation treatments were randomly located within each replicate. Mulch treatments were randomly located within each site preparation block. Plates 4.3 and 4.4 show mulch treatments. Screen, surface and below ground temperatures were taken at regular intervals over a 3 hour period for Sandstone (2) on November 20th, 1985.



Plate 4.1 Deep ripping treatment in field experiment 1 on Sandstone (2) at Hunter Valley No. 1 Mine.



Plate 4.2 Cultivation treatment in field experiment 1 on Sandstone (2) at Hunter Valley No. 1 Mine.



Plate 4.3 Straw mulch treatment in field experiment 1 on Sandstone (2) at Hunter Valley No. 1 Mine.



Plate 4.4 Chitter mulch treatment in field experiment 1 on Sandstone (2) at Hunter Valley No. 1 Mine.

4.4.2.2 Field Experiment 2 - Soil Amendment Experiment

A three replicate 3 x 4 x 5 factorial design including :

- (i) 3 substrates :- As for field experiment 1
- (ii) 4 soil amendments :- gypsum at 3 t ha⁻¹
 - Alginure at 0.75 t ha⁻¹
 - Terrasorb at 1 t ha⁻¹
 - Control
- (iii) 5 tree species (*E. maculata*, *E. punctata*, *E. cladocalyx*,
A. salicina, *C. glauca*)

Each soil amendment was thoroughly mixed (dry) with seed, fertilizer and bran prior to application on May 16th, 1985. Soil amendment treatments were randomly located within each replication.

4.4.2.3 Field Experiment 3 - Fertilizer Rate/Pellet Experiment

A three replicate 2 x 3² x 5 factorial design comprising :

- (i) 2 methods of fertilizer application : - loose
 - pelletized
- (ii) 3 substrates :- As for field experiment 1.
- (iii) 3 combinations of N and P each at - 0 kg ha⁻¹ (NO P0)
 - 10 kg ha⁻¹ (N10 P10)
 - 20 kg ha⁻¹ (N20 P20)
- (iv) 5 tree species (*E. maculata*, *E. punctata*, *E. cladocalyx*,
A. salicina, *C. glauca*)

The seed was pelletized by slowly mixing methyl cellulose (gum arabic) dissolved in water with the seed to form an even coating on the seed. The gum arabic was prepared as a 1.5% gel. Appropriate quantities of ammonium nitrate and sodium di-hydrogen phosphate were pre-mixed and applied slowly to the adhesive coated seed until the required rates of N and P have been achieved. Prior to mixing, both fertilizer compounds were ground to a fine consistency using a mortar and pestle. Non-pelletized seed was coated with gum arabic and allowed to dry. Non-pelletized seed was mixed and broadcast with loosely applied fertilizer. The experiment commenced at the same time as field experiment 2.

4.4.2.4 Field Experiment 4 - Grass Competition / Herbicide Experiment

The experiment consisted of a split plot 2 x 4 x 5 x 6 factorial design in randomized blocks replicated three times as follows:-

- (i) 2 pasture age classes (newly sown (0) and 10 months)
- (ii) 4 herbicide treatments (with and without pre-emergent herbicide; with and without knockdown herbicide)
- (iii) 5 tree species (*E. maculata*, *E. punctata*, *E. cladocalyx*, *A. salicina*, *C. glauca*)
- (iv) 6 pasture swards

The experiment was established on the topdressed area at Hunter Valley No. 1 Mine in two stages. The first stage was established in early June, 1985, and consisted of pegging out and sowing the six pasture mixtures onto half the plots, the intention being to establish pasture swards on those areas by March, 1986. At this time, the second stage of the experiment was established. The second stage consisted of sowing the remaining plots with the same pasture seed combinations which was in turn followed by sowing the standard tree seed mix into all the plots.

Starter 15 was broadcast onto all plots immediately after pasture sowing in both 1985 and 1986 at the rate of 200 kg ha⁻¹.

Starter 15 analysis gives 15% N as ammonia, 13.1% Total P made up of 12.6% water soluble, 0.4% citrate soluble, 0.1% citrate insoluble, 0.2% Ca as superphosphates, and 10.3% S as sulphates

All plots were chisel ploughed prior to pegging out in 1985. Prior to sowing in March, 1986 plots not sown in 1985 were chipped free of weeds and lightly scarified by hand with a McLeod tool (rake-hoe).

Immediately prior to sowing in 1986, half the plots established with pasture in 1985 were treated with the knockdown herbicide Roundup at the recommended rate of 30 ml per litre of water for 9 m². At the same time, half the plots sown in 1986 were treated with the pre-emergent herbicide Diphenamid at 7 kg a.i. ha⁻¹ immediately after sowing.

The six pasture swards established were :

Mix	Pasture Species	Rate kg ha ⁻¹
1. (Standard Soil Conservation Service Mix)	Wimmera rye grass (<i>Lolium rigidum</i>)	6
	Phalaris (<i>Sirocco</i>)	8
	Jemmalong Medic (<i>Medicago trunculata</i>)	4
	Northam sub clover (<i>Trifolium subterraneum</i>)	2
	Nungaria sub clover (<i>Trifolium subterraneum</i>)	2
	Wooenlup sub clover (<i>Trifolium subterraneum</i>)	2
	Seaton Park sub clover (<i>Trifolium subterraneum</i>)	2
	Lucerne (<i>Medicago sativa</i>)	4
	Rhodes grass (<i>Chloris gayana</i>)	10
	Couch grass (<i>Cynodon dactylon</i>)	<u>6</u>
	TOTAL	<u>46</u>
2. (Tree Mix)	Wimmera rye grass	10
	Rhodes grass	5
	Couch grass	4
	Lucerne	4
	Jemmalong medic	<u>4</u>
		<u>27</u>

Mix	Pasture Species	Rate kg ha ⁻¹
3. (Annual)	Oats (<i>Avena stiva</i>)	20
4.	Phalaris	10
5.	Jemmalong medic	12
6.	Nil	0

Per cent ground cover was assessed at the time of final measure on 50% of each plot (randomly located).

Gravimetric water content and surface and sub-surface temperatures were taken weekly and averaged over the 12 months period following sowing.

Due to dry conditions following sowing, insufficient germination occurred to allow analysis of results. However, some trends are discussed.

4.4.2.5 Field Experiment 5 - Sowing Time Experiment

The experiment consisted of a 3 x 5 x 6 factorial design with three replicates as follows :-

- (i) 3 substrates :- Topdressing (1), Sandstone (2), Shale (3)
- (ii) 5 tree species :- (*E. maculata*, *E. punctata*, *E. cladocalyx*, *C. glauca*, *A. salicina*)
- (iii) 6 sowing times at monthly intervals.

The first plots were sown on April, 4th 1986. Monthly sowing plots were randomly located within each replicate. Germination and survival were measured six months after each sowing event, i.e. April (1986) sowing was measured in October (1986). Height and basal area were measured in April 1987 regardless of sowing time.

All sites were lightly scarified one week prior to sowing. Germination and losses were measured monthly for the first six months after each sowing event. Gravimetric water % was measured weekly for all three substrates and averaged over each month.

4.4.2.6 Field Experiment 6 - Pre-emergent Herbicide Experiment

The experiment consisted of a 3 x 4 x 5 factorial design replicated three times as follows :-

3 substrates :- Topdressing (16), Sandstone (17),
Topdressed and grassed (18)

4 species :- (*E. punctata*, *E. maculata*, *C. glauca*, *A. saligna*)

5 herbicide treatments :- Diphenamid at 7 kg a.i. ha⁻¹
Diphenamid at 14 kg a.i. ha⁻¹
Surflan at 8 l a.i. ha⁻¹
Surflan at 16 l a.i. ha⁻¹
Control

The experiment was conducted on three substrates on the eastern side of workings at Ravensworth No. 2 Colliery. Topdressing (16) represented a level topdressed site. Sandstone (17) was on a sandstone like material on level ground. The third site, Topdressed and grassed (18) was a level topdressed site with established pasture predominated by Rhodes grass.

All three sites were deep ripped at three metre spacings. After deep ripping the Topdressed and grassed (18) site was sprayed with Roundup at 10 l ha⁻¹ in one metre wide strips along each proposed planting row. Roundup was again applied in a similar manner one month later (one month prior to planting).

Each pre-emergent herbicide treatment within each replication comprised 20 seedlings planted in a row. Each row contained five trees of each species. Species were randomly located in each row. The trees were planted on a 3 m x 3 m grid. A 6 m buffer was placed between each replication. Immediately after planting, 30 cm high x 20 cm diameter wire cages were placed around all trees to prevent hare damage. The trees were planted in mid April, 1985 and dead trees were replaced 3 weeks prior to herbicide application in mid June, 1985. All trees had two 21 g Agriform slow release fertilizer tablets placed 10 cm to each side of the base of each root ball.

Agriform analysis :

- 20% Total N present as urea formaldehyde made up of
 - 7% water soluble N
 - 13% water insoluble N
- 4.3% Total P present as citrate soluble from calcium phosphate
- 4.1% Total K present as sulphate of potash
- 2.6% Ca present as calcium phosphate and calcium sulphate
- 1.6% S present as potassium sulphate, ferrous sulphate, ammonium sulphate and calcium sulphate
- 0.35% Fe present as ferrous sulphate
- 0.6% Biuret (Maximum)

The trees were well watered at one and four weeks after planting.

Immediately after planting the following pasture seed was broadcast over the ungrassed Topdressing (16) and Sandstone (17) sites.

<u>Species</u>	<u>Rate (kg ha⁻¹)</u>
Wimmera Rye grass	10
Rhodes grass	5
Couch grass	4
Lucerne	4

Jemmalong medic	<u>4</u>
TOTAL	<u>27</u>

Starter 15 was applied with the pasture seed at 200 kg ha⁻¹.

Immediately after sowing pre-emergent herbicide was applied evenly on all three sites in a one metre wide band centred on each planted row.

The height of all seedlings was measured at 0, 6 and 12 months. The basal area of all trees was measured at planting and after 12 months. Basal area was measured at 5 cm above ground level. Per cent ground cover was assessed after 12 months by randomly dropping a one metre square 15 times along each planting line. Ground cover per cent and species composition was assessed for each drop for four categories i.e. Pasture alive and dead, Weeds (non-pasture) alive and dead.

The experiment was analysed as a split-plot design, with the main plots being herbicide treatments and the subplots, species.

4.4.2.7 Field Experiment 7 - Japanese Millet Competition Experiment

The experiment consisted of 2 x 5² factorial design replicated three times, as follows :-

- (i) 2 substrates :- Topdressing (19), Overburden (20)
- (ii) 5 sowing rates of Japanese Millet (*Echinochloa utilis*) -
0, 2.5, 5, 10 and 20 kg ha⁻¹
- (iii) 5 native tree species (*E. maculata*, *E. punctata*, *A. costata*, *C. glauca*, and *A. longifolia*)

Both sites were level and were deep ripped at three metre spacings to a depth of 0.5 m, three weeks prior to sowing. Immediately prior to sowing Starter 15 was broadcast at a rate of 300 kg ha⁻¹. The

sites were tine cultivated to a depth of 15 cm after which Japanese millet was immediately sown. The different sowing rates were randomly located within each replication. Each plot was then broadcast sown with a standard mixture of native tree seed in the third week of April, 1985.

Per cent ground cover was assessed at four and twelve months. Results were adjusted using per cent crop cover as a co-variant. After twelve months, native tree seedlings were measured for height and basal area. The gravimetric water content for each sowing rate on Topdressing (19) was measured between May 4th, 1987 and June 12th, 1987. On each day three samples were randomly taken from each plot.

4.5 Broadacre Field Trials

A wide range of broadacre field trials (predominantly sowing trials) was established over the three year study period. Two of these sites have been selected as being representative of results obtained.

4.5.1 Broadacre Field Trial 1. Bloomfield Colliery (Lower Hunter Valley).

This trial comprised two parts.

(1) A broadacre sowing trial was designed to examine germination and growth on two substrates (Topdressing (19) and Overburden (20)). The trial was established at the same time and adjacent to field experiment 7. The sown site was evenly divided between the two substrates on a 0.6 ha site. Seed was sown at the same rates and in the same manner as field experiment 7. Starter 15 was applied after ripping at 300 kg ha⁻¹ and prior to cultivation. Both sites were deep ripped with a single tine at 3 m spacing to a depth of 50 cm. Deep ripping occurred in early April, 1986, followed immediately by cultivation and sowing.

(2) Seedlings of the same species as sown above were planted adjacent to the broadacre sowing trial. The experiment was designed to compare the growth of sown and planted seedlings.

Twenty-five trees of each species were planted on each of the two overburden materials. Seedlings were six months old, between 15 and 30 cm in height, and had been grown under standard conditions at the Forestry Commission Nursery at Muswellbrook. The trees were planted in the centre of rip lines on a 6 m x 4 m grid at the same time as sowing occurred on the adjacent broadacre trial. The site received the same site preparation and fertilizer treatments as the broadacre sowing site. The height of a 2% sample (by area) of seedlings on the sown site were compared with those for all planted seedlings after 12 months. Where applicable, results were converted to a per hectare basis to facilitate comparison.

4.5.2 Broadacre Field Trial 2. C.S.R. Lemington Colliery (Upper Hunter Valley)

A broadacre sowing trial was designed to compare germination and growth on two substrates. The two substrates were located approximately 500 m apart on level ground. A 0.1 ha area on each site was pegged out, deep ripped and sown with seed as shown in Table 4.2. Prior to ripping, Starter 15 was broadcast evenly over each site at the rate of 300 kg ha⁻¹. Both sites were deep ripped with a single tine at 3 m spacing to a depth of 70 cm. Deep ripping occurred in the first week of June, 1986 and the seed mix was sown in the second week in June, 1986. Germination and height variables were measured for all surviving trees on both plots after 12 months. Where applicable, results were converted to a per hectare basis to facilitate comparisons.

In both field trials 1 and 2 variance components were not homogeneous so individual t-tests were done instead of standard analysis of variance.

4.6 Physical and Chemical Properties of Substrates

All substrate material was collected from within 10 cm of the surface of re-spread material. Materials had been exposed to the atmosphere for between 6 and 24 months. Material passing through a 5 cm sieve was collected on site and analysed, as follows. *Topdressing material, containing a mixture of A and B horizons, had been stockpiled for approximately 18 months.*

4.6.1 Chemical Analysis

Nutrient Analysis, pH and Salinity

Substrate samples were air dried, crushed to pass through a 2 mm sieve and chemically analysed for a range of plant nutrients, using the following methods :-

- (a) pH measured in (i) a 1:1 soil/distilled water suspension after shaking for ten minutes, as described by Black (1965), (ii) a 1:1 soil/1 M KCl solution (*methodology of N.S.W Forestry Comm.*).
- (b) E.C. measured in a 1:1 soil/distilled water suspension after shaking for ten minutes, as described by Tucker and Beatty (1974).
- (c) Total N determined by the manual salicylate-hypochloride procedure for determining ammonium-N in Kjeldahl digests, as described by Fukumoto and Chang (1982).
- (d) Organic material based on Walkley-Black chromic acid method as described by Jackson (1958).

- (e) Total P using concentrated hydrochloric acid as described by Murphy and Riley (1962).
- (f) Exchangeable Ca, Mg, K and Na - extracted with 1 M ammonium acetate at pH 7 (Chapman 1965a and b) and determined by atomic absorption spectrophotometry.
- (g) H₂O equilibrium P as described by Murphy and Riley (1962). All the above analyses, with the exception of EMC and pH were carried out at the chemistry section of the Wood Technology and Forest Research Division of the N.S.W. Forestry Commission. EMC's and pH were determined at Muswellbrook Forestry Commission Nursery.

Exchangeable Sodium Percentage

Concentrations of exchangeable cations Ca, Mg, K and Na, determined using the technique described in 4.6.1 (f) were used to calculate an approximation of exchangeable sodium per cent (ESP) as follows:

$$\text{ESP} = \frac{(\text{Na}^+)}{(\text{Ca}^{++}) + (\text{Mg}^{++}) + (\text{K}^+) + (\text{Na}^+)}$$

where () indicates the concentration of cations in meq l^{-1} . The above calculation assumes that Cation Exchange Capacity (CEC) equals the sum of the exchangeable bases. A valid assumption if $\text{pH} > 6.5$.

4.6.2 Physical Analyses

Water Holding Characteristics

Gravimetric Water Content

The gravimetric water content of substrates was calculated using the technique outlined by Corbett (1969) and described previously in Section 4.3.2.8.

Percentage Soil Moisture at Field Capacity and Wilting Point

Percentage soil moisture at field capacity (pF 2.0) and wilting point (pF 4.2) was determined using a pressure plate apparatus and technique as described by Black (1965).

Soil Temperature

Soil temperature, both at the surface and at a depth of 2 cm was calculated for particular field experiments and for general substrate types. The technique has been generally described in Section 4.3.2.8.

For general substrate characterization, two methods were employed at Hunter Valley No. 1 Mine. In method 1 maximum and minimum temperatures for each substrate were measured at ground level under a well ventilated 40 cm high plastic waste-paper basket at weekly intervals over a 12 month period. Weekly temperatures were meaned for each month. Individual readings were taken between 10 a.m. and 11 a.m. The second method measured short term temperature fluctuations for each substrate over a 5 hour period (8 a.m. - 1 p.m.) on January 30th, 1987. Temperatures were measured at the surface under both shaded (under ventilated waste-paper basket as described in method (1)) and unshaded (full sun) conditions and also at a depth of 2 cm (unshaded). For each substrate a representative site was selected within each substrate boundary and measurements were consistently taken at these sites.

Particle Size Distribution

Substrate samples were ground to pass through a 2 mm sieve. Measurement was undertaken using the hydrometer method described by Black (1965).

4.7 Foliar Analysis

Analysis of foliar nutrients for glasshouse experiments 1 and 4 were undertaken using the technique described by Lambert (1982). Analysis of foliage from glasshouse experiment 7 was undertaken using the Technicon Block Digestor (Tech. Pub. TA4-0323-10) and a de-automated version of the Technicon Autoanalyser Technique (Technicon Industrial Systems 1977).

CHAPTER 5

PHYSICAL AND CHEMICAL PROPERTIES

OF SUBSTRATES

5.1 Introduction

A large number of overburden and interburden strata are normally removed during mining. During extraction, stockpiling and subsequent re-shaping mixing of layers occurs and the identity of individual strata is lost. Consequently, even if the nature of specific strata is known, it is difficult to predict if this material will appear near the surface, and to what extent. To some degree pre-stripping, stockpiling and re-spreading topdressing material on the surface reduces the variability of surface material. However, even in this case, mixing of horizons occurs and variability is the norm.

In order to have any ability to extrapolate results or to identify limiting conditions, the characteristics of each batch of spoil must be identified. This is quite feasible for glasshouse experiments where discreet, manageable volumes are used. However, for field experiments this is more difficult and even extensive sampling may not indicate all the substrate conditions plants are likely to encounter in the field.

This chapter characterizes the various substrates used in individual experiments. Due to the inclusion of a variety of collieries the array of characteristics is even more varied than if only one mine was used. The use of a variety of collieries was intentional, and was designed to broaden the results of the research and to help assess the range of physical and chemical characteristics likely to be encountered. This, in turn, would indicate broad limits to tree establishment, assuming the tolerance of native tree species could be quantified.

Original classification of substrates as shale, sandstone etc. was based purely on visual appearance. Despite attempts to obtain materials for glasshouse experiments from the same sites, it became quickly apparent from analyses that considerable variation still occurred in the characteristics of apparently similar substrates. Consequently, similar classifications, e.g. shale, imply no similarity unless shown as such in the tables of physical and chemical characteristics.

5.2 Results and Discussion

5.2.1 Chemical Analyses

The results of chemical analyses of substrates are shown in Table 5.1.

For both Hunter Valley No. 1 and C.S.R. Lemington mines the shale material generally had the highest total N, while the sandstone material consistently had the lowest total N. The high total N level for the shale material probably relates to the high organic content in the form of carbonaceous material and unoxidized coal. Nitrogen bound in this form is considered unavailable to plants in the short term. With the exception of Topdressing (7) from Hunter Valley No. 1 used in glasshouse experiment 5, all materials from these two collieries were considered deficient in N and plants should respond to nitrogenous fertilizer. Topdressed and grassed (18) from Ravensworth Colliery had an adequate N level but this site had been previously fertilized during pasture establishment.

The H₂O soluble P test gave an indication of how much P is being supplied by the soil in soluble form. The range of total P values is small. All P levels were considered extremely low and plants should respond to phosphate fertilizer on all substrates.

Table 5.1 Chemical characteristics of substrates used in glasshouse and field experiments and broadacre field trials.

Location	Experiment	Substrate Type & No.	pH (1:1 H ₂ O)	E.C. (1:1H ₂ O) μ S cm ⁻¹	N Tot %	Organic %	Total P ppm	H ₂ O Equil P ppm	Exchangeable Cations meq / 100 g				ESP
									Ca	Mg	K	Na	
HV No.1 Colliery	Glass.Exp.1,2	Topdr 1	5.91	561	0.13	2.62	253	0.07	3.21	1.18	0.38	1.79	27.3
Colliery	Field Exp.1, 2,3,4, & 5	Sandst.2	8.09	742	0.11	3.54	329	0.15	22.08	2.82	0.56	2.76	9.8
		Shale 3	7.99	982	0.26	9.23	334	0.13	8.31	3.48	0.56	5.70	31.6
HV No.1 Colliery	Glass.Exp. 3	Topdr. 4	5.56	89	0.22	2.71	252	0.28	3.89	6.01	0.40	0.83	7.5
		Sandst.5	8.10	405	0.05	0.76	472	0.10	9.68	13.06	0.71	1.60	6.4
		Shale 6	7.53	85	0.30	9.86	349	1.02	5.73	25.09	0.47	7.51	19.4
HV No. 1 Colliery	Glass.Exp. 5	Topdr. 7	6.03	74	0.54	3.08	295	0.26	4.86	5.81	0.57	0.68	5.7
		Sandst.8	8.24	299	0.06	0.78	396	0.11	10.65	13.57	0.67	1.76	6.6
		Shale 9	8.03	271	0.26	7.37	414	0.18	8.25	20.59	0.51	4.89	14.3
C.S.R. Lemington Colliery	Glass.Exp. 4	Topdr .10	5.42	249	0.09	3.35	177	0.82	2.93	4.24	0.40	0.67	8.1
		Sandst.11	8.20	378	0.05	2.20	238	0.10	4.46	4.45	0.24	0.76	7.7
		Shale 12	8.34	1830	0.16	2.85	305	0.79	4.70	7.23	0.33	2.86	18.9
C.S.R. Lemington Colliery	Glass.Exp. 6	Topdr. 13	5.10	218	0.07	1.86	119	0.37	1.52	2.04	0.29	0.42	9.4
		Broadacre Fld Sandst.14	8.64	231	0.06	1.25	240	0.28	5.07	5.87	0.31	0.76	6.3
		Trial 2 Shale 15	8.44	396	0.16	2.69	292	0.15	4.39	5.43	0.32	0.77	7.1
Ravensworth	Field Exp. 6	Topdr. 16	5.88	220	0.08	2.02	142	0.00	3.80	4.18	0.37	0.71	7.8
	Glass.Exp. 7	Sandst.17	7.93	890	0.20	2.36	206	0.00	5.99	9.64	0.51	1.75	9.8
No.2 Colliery	(Topdress only)	Topdr. 18 & grassed	5.47	310	0.54	2.37	169	0.53	2.66	3.60	0.33	0.35	5.0
Bloomfield Colliery	Field Exp. 7	Topdr. 19	4.11	274	0.04	0.48	79	0.00	0.17	2.14	0.32	1.10	29.5
Colliery	Broadacre Fld.	Over-											
	Trial 1.	burden.20	4.41	589	0.07	1.17	130	0.11	0.15	3.44	0.38	0.75	15.9
Suggested Critical Values			(5.5-8.5)***	1580**	0.4**			20*		(0.4-1.2)**	(0.2-0.8)**	(0.3-0.3)**	6+

* For nutrients these are levels below which a plant (agricultural crop) response to the application of the element might occur.

** Doll and Lucas (1973)

+ Richards (1954)

** Elliott and Hannan (1980)

* Brady (1974) (> 20 ppm adequate for crops, < 10 extremely low). ** Bevege (1986). *** State Pollution Control Comm.(1983).

The pH (1:1 H₂O) of most of the substrates was within the critical range; the exception being Sandstone (14) from C.S.R. Lemington. Generally, the sandstone substrates had the highest pH (1:1 water) and were moderately to highly alkaline. Nutrient availability, and particularly P availability may be reduced in materials with a pH higher than 8. The topdressing materials had the lowest pH and were all slightly to moderately acidic.

With one exception, the electrical conductivity of all substrates tested was not considered limiting, the exception being Shale (12) from C.S.R. Lemington. Growth of some species could be expected to be affected on this material.

Exchangeable cation values appear adequate for growth. None of the exchangeable cation values were below suggested critical values or ranges for any of the substrates tested. However, there may be an imbalance in the Ca:Mg ratio which was frequently below the critical level of 1.0 (e.g. glasshouse experiment 3) proposed by Emerson and Bakker (1973).

Most of the substrates had ESP values > 6 , the commonly accepted critical value for Australian soils.

Most analyses of chemical (and physical) characteristics available for comparison have been taken from borehole and profile investigations prior to mining. A direct comparison with characteristics in this study is therefore difficult due to the fact that this study examines recontoured materials which have experienced considerable mixing. Generally, however, the results fell within broad ranges established in earlier studies at these or adjacent mines. As an example, the low proportion of soluble P to total P was expected and typical of results for both soil and overburden strata materials in the upper Hunter Valley (Elliott *et al.* 1980. Croft and Associates, 1986). Low N levels were also a characteristic of most materials (Elliott *et al.* 1980). Generally, pH, conductivity, organic % and exchangeable cations agreed with earlier findings. However, some exceptions were noted. For example, at

Ravensworth No. 2 Mine Croft and Associates (1986) noted deficiencies in at least one of the exchangeable cations in a variety of topsoil materials. Similar deficiencies were not apparent for Ravensworth No. 2 in Table 5.1. However, absolute values may not always be an accurate guide as deficiency in, for example Mg, may be induced by high exchangeable K percentages.

5.2.2. Physical Analyses

The results of physical analyses are shown in Table 5.2

Soil-water relationships for substrates from Hunter Valley No. 1 and Bloomfield collieries can be seen in Table 5.2, and Figures 5.1 and 5.2 respectively. Although the moisture characteristics, as determined by the gravimetric water percentage at pF 2.0 and pF 4.2 can be questioned as an accurate measure of plant available water (Gardiner 1971), they do provide a basis for comparing substrates.

The results for Hunter Valley No. 1 (Figure 5.1) indicate that under field conditions Topdressing (1) can hold a greater percentage of water and for a longer period than the other two substrates for equivalent rainfall. Shale (3) would be influenced by the steeper slope of the site and consequent enhanced drainage and reduced water retention.

For Bloomfield (Figure 5.2), the two substrates had a similar range of moisture content between wilting point and field capacity. However, the lower wilting point of the overburden material would effectively result in water being available to plants for a longer period after rainfall.

Generally, the available water capacity of all substrates at both collieries was high. The capacity of the sandstone and shale materials can be expected to further improve with weathering and as soil forming processes progress.

Table 5.2 Physical characteristics of substrates used in glasshouse and field experiments and broadacre field trials.

Location	Experiment	Substrate Type & No.	Particle Size Composition %				Type	Percent Soil *		Avail. H ₂ O capacity
			Clay	Silt	Fine Sand	Coarse Sand		Moisture at pF2.0	pF4.2	
HV No. 1 Colliery	Glass.Exp.1 & 2	Topdress. 1	36	26	26	12	Silty Clay loam	30.5	10.8	19.7
	Field Exps.1, 2 3, 4 & 5	Sandstone 2	27	22	38	13	Clay loam	24.7	10.3	14.4
		Shale 3	30	22	24	24	Clay loam	27.0	12.9	14.1
HV No. 1 Colliery	Glass.Exp. 3.	Topdress. 4	36	26	26	12	Silty clay loam			
		Sandstone 5	27	22	38	13	Clay loam			
		Shale 6	30	22	24	24	Clay loam			
HV No. 1 Colliery	Glass.Exp. 5	Topdress. 7	31	20	33	16	Clay loam			
		Sandstone 8	31	26	31	12	Silty Clay loam			
		Shale 9	23	32	35	20	Silt Loam			
C.S.R. Lemington Colliery	Glass.Exp. 4	Topdress.10	16	9	32	43	Sandy loam			
		Sandstone11	9	8	29	54	Loamy sand			
		Shale 12	20	14	19	47	Loam			
C.S.R. Lemington	Glass.Exp. 6 Broadacre Field Trial 2	Topdress.13	11	6	39	54	Sandy loam			
		Sandstone14	11	5	27	57	Sandy loam			
		Shale 15	15	10	29	46	Sandy loam			
Ravensworth No. 2 Colliery	Field Exp. 6 Glass.Exp. 7 (Topdress.only)	Topdress.16	22	11	31	36	Clay loam			
		Sandstone17	23	10	26	41	Clay loam			
		Topdressed & Grassed 18	21	13	26	40	Loam			
Bloomfield	Field Exp. 7 Broadacre Field Trial 1	Topdress.19	45	12	26	17	Clay	29.1	13.2	15.9
		Overburden 20	39	18	28	15	Clay	26.0	9.0	15.0

* For relevant field experiments only

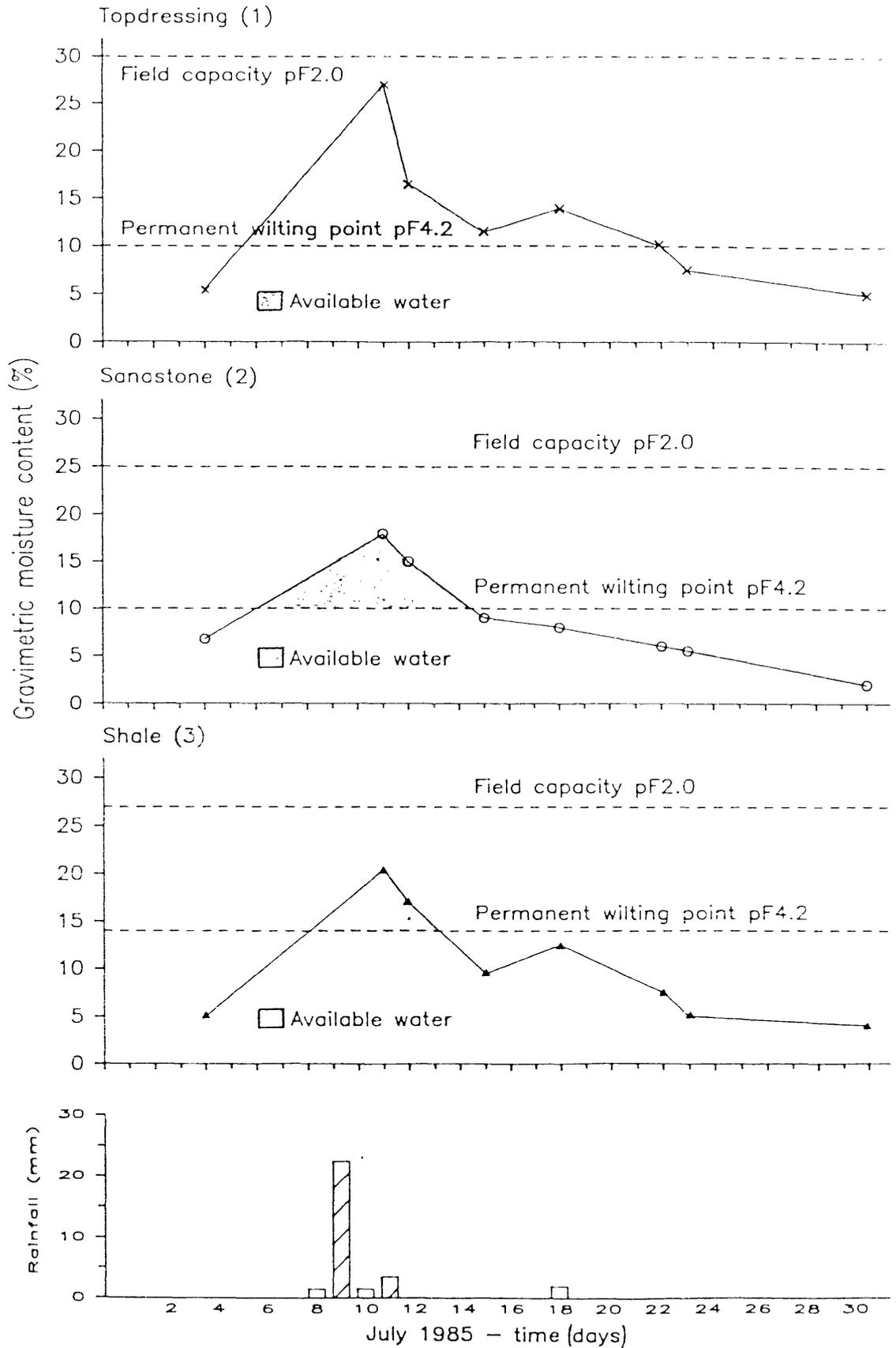


Figure 5.1 Effect of rainfall on gravimetric moisture content for three substrates from Hunter Valley No. 1. Colliery.

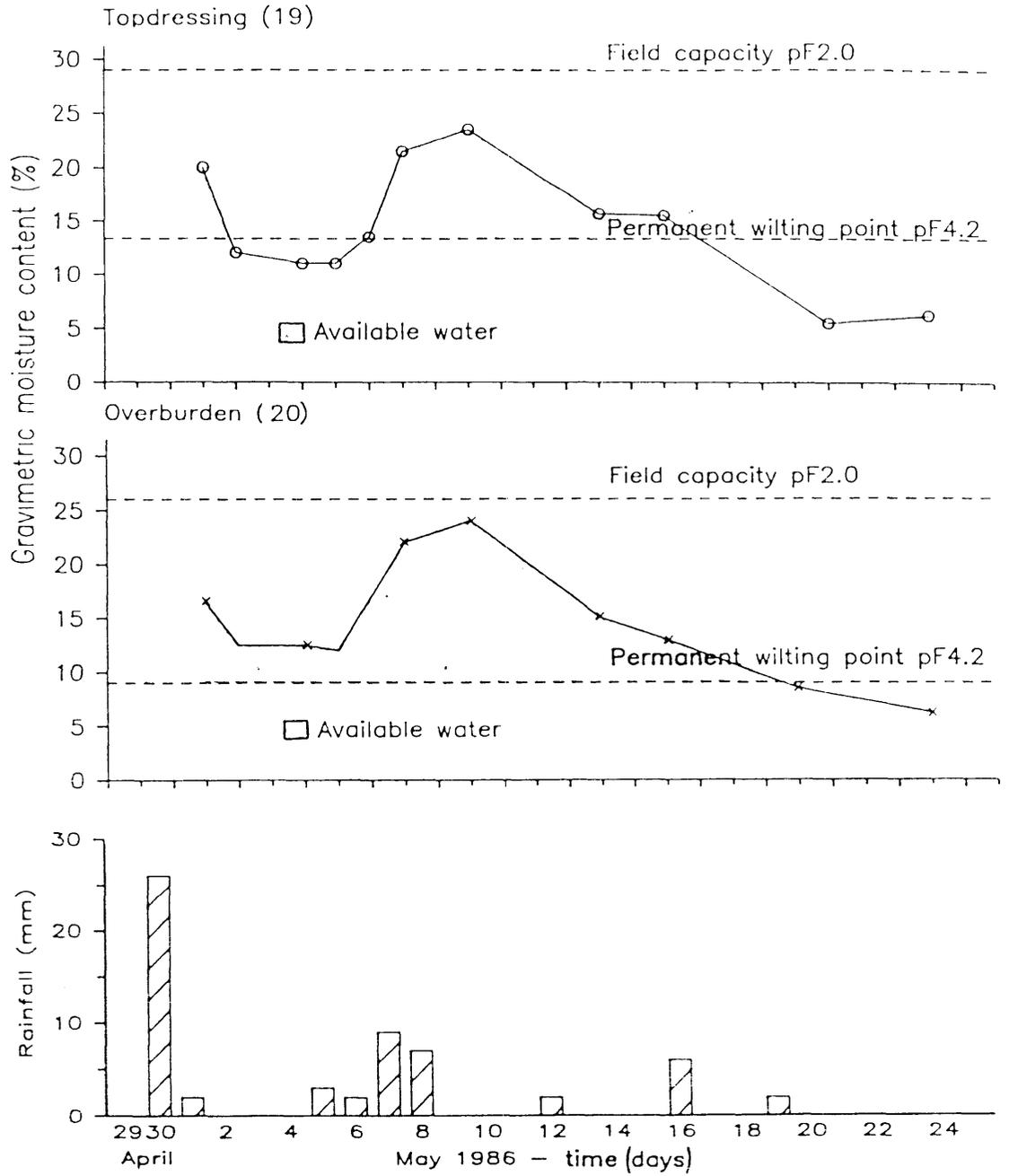


Figure 5.2 Effect of rainfall on gravimetric moisture content for two substrates from Bloomfield Colliery.

Generally, the moisture content of non-topdressing materials was much higher than the moisture content of non-topdressing materials found by Irving (1987) at Tarong in Queensland. However, moisture content levels were similar to those found by Evans (1985) at Gregory Mine in Central Queensland for comparable substrates.

The results of the particle size analysis (PSA) are shown in Table 5.2. The results emphasize the variability that can occur on a relatively small field site for visually similar material. The analysis does not indicate the large rock fragment (over 5 mm) discarded after preliminary sieving of the sandstone and shale materials, nor the 2 mm - 5 mm fragments discarded prior to PSA analysis.

Both textural and structural differences can be expected to affect available water capacity (Cook 1976). In sandstone and shale material, pedal structure is generally absent immediately after mining, while frequent handling of topdressing material also destroys any pedality. Consequently, textural differences become more critical in their role in storing water. Figure 2.1 in Section 2.3.3.3 gives a comparison of the water holding capacities of different texture classes. These results generally agree with the relative water holding capacities of Hunter Valley No. 1 substrates shown in Figure 5.1.

Average monthly maximum and minimum temperatures under shade at Hunter Valley No. 1 during the 1986 are shown for each substrate in Figure 5.3. These results show variation in the substrate temperatures throughout the year. Of particular importance was the slower rate of temperature increase of Sandstone (2) following low mid-winter temperatures. All substrates experienced very hot surface conditions under shade in March and April. Minimum winter temperatures dropped below zero for all substrates with topdressing material having the lowest minimum winter temperature.

If it is considered that optimum germination of most savannah *Eucalyptus* species occurs between 22° and 30°C and that the mean monthly temperatures reflect sustained conditions then the optimum months for germination from a temperature viewpoint is from late March to early May and from mid-August

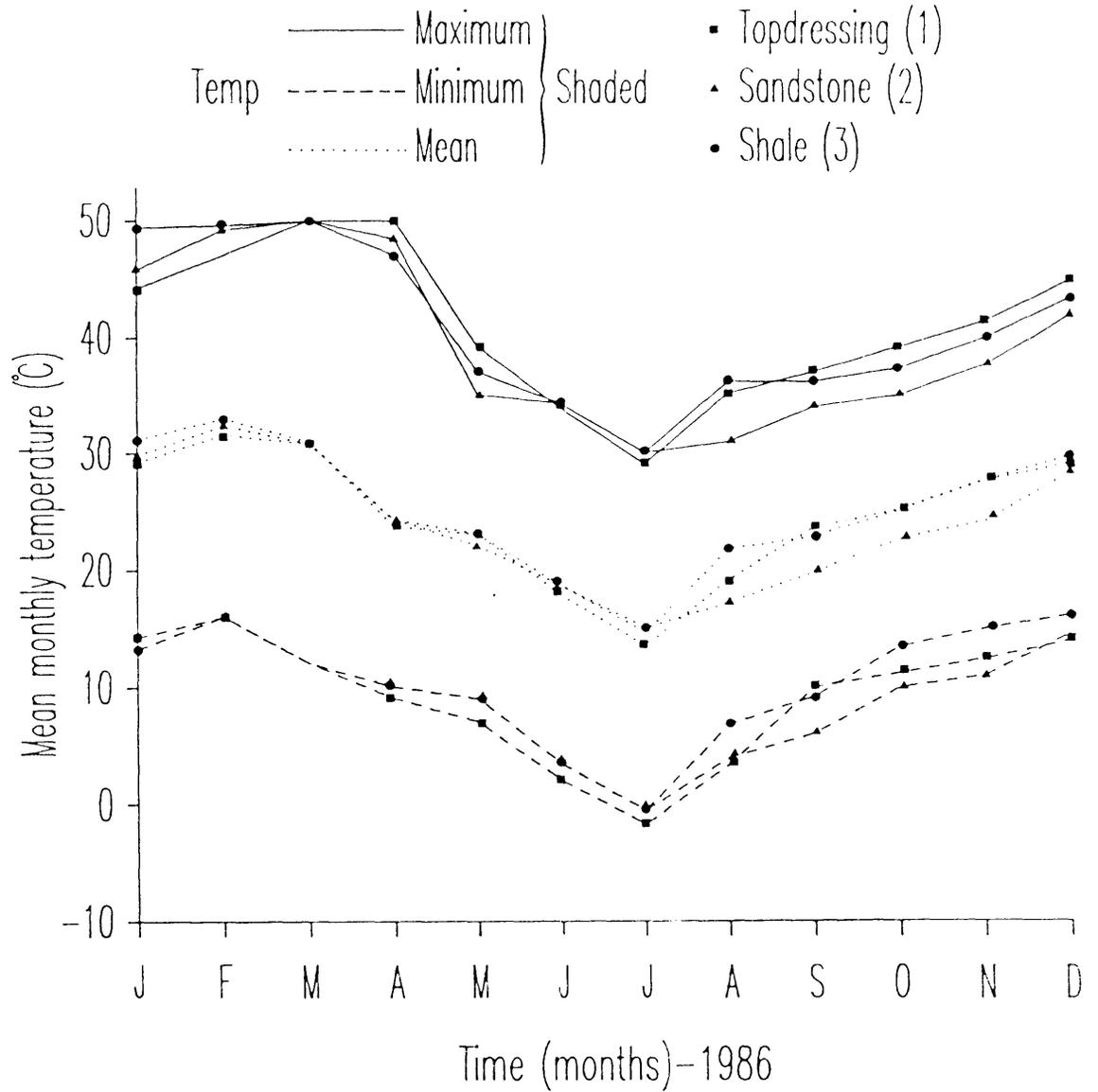


Figure 5.3 Mean monthly maximum, minimum and mean temperatures at the spoil surface under shade for a 12 month period at Hunter Valley No. 1. Colliery.

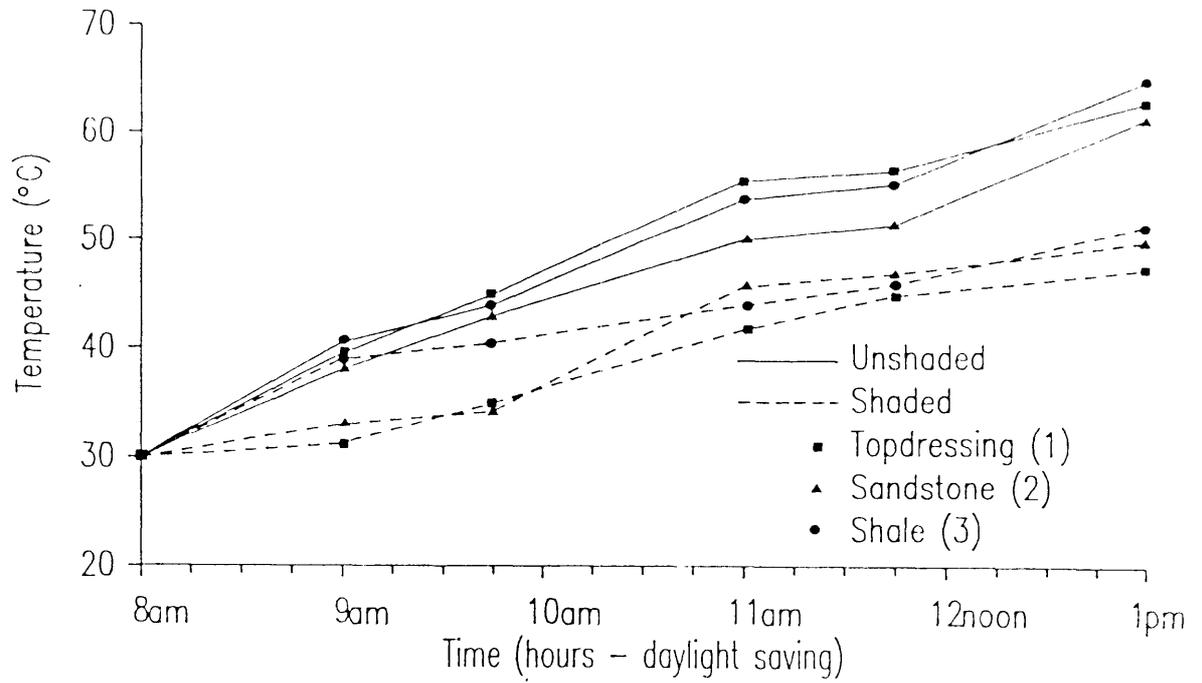
to December (depending on substrate). When these results are superimposed over rainfall and particularly evaporation data for Hunter Valley No. 1 as described in Chapter 3, it appears that May is the most suitable month for sowing. While August also appears suitable, subsequent monthly evaporation rapidly became more severe and the risk of unfavourable conditions increases rapidly.

Temperature variation at the surface and at 2 cm depth over a five hour period in summer (January 30th, 1987) are shown for Hunter Valley No. 1 in Figure 5.4. Temperature readings at the surface were made in both shaded and unshaded conditions. Unshaded temperatures at the surface would be typical of those experienced by newly sown seed. Unshaded temperatures for all substrates were between 30° and 32°C at 8.00 a.m. These uniformly high early morning temperatures suggest that relatively high surface ground temperatures were maintained overnight.

Unshaded temperatures rose to between 60°C and 65°C at 1 p.m. and were still rising, while shaded temperatures rose to between 45° and 52°C at this time. Shaded temperatures would be typical of those experienced by seed under a mulch or in the shade of grass or trees. Both shaded and unshaded temperatures at 8 a.m. were adequate for the germination of all eucalypt species listed in Table 2.2. However, the very high unshaded temperatures experienced after 11 a.m. can be expected to be detrimental to new germinants. Consequently, sowing in summer without a mulch cover would not be advisable, even assuming adequate soil moisture was available.

Differences in substrate temperatures were substantial at certain times throughout the five hour period. In the unshaded situation there was little difference between the temperature of Topdressing (1) and Shale (3) throughout the day. However, Sandstone (2) temperatures were up to 6°C below the other two substrates. This may relate to reduced ground heating as a result of re-radiation from this lighter coloured material. In the shade, Shale (3) temperatures initially rose more rapidly. This probably reflected the greater heat absorbing capability of this material due to its

(a) At surface



(b) At 2cm depth (unshaded)

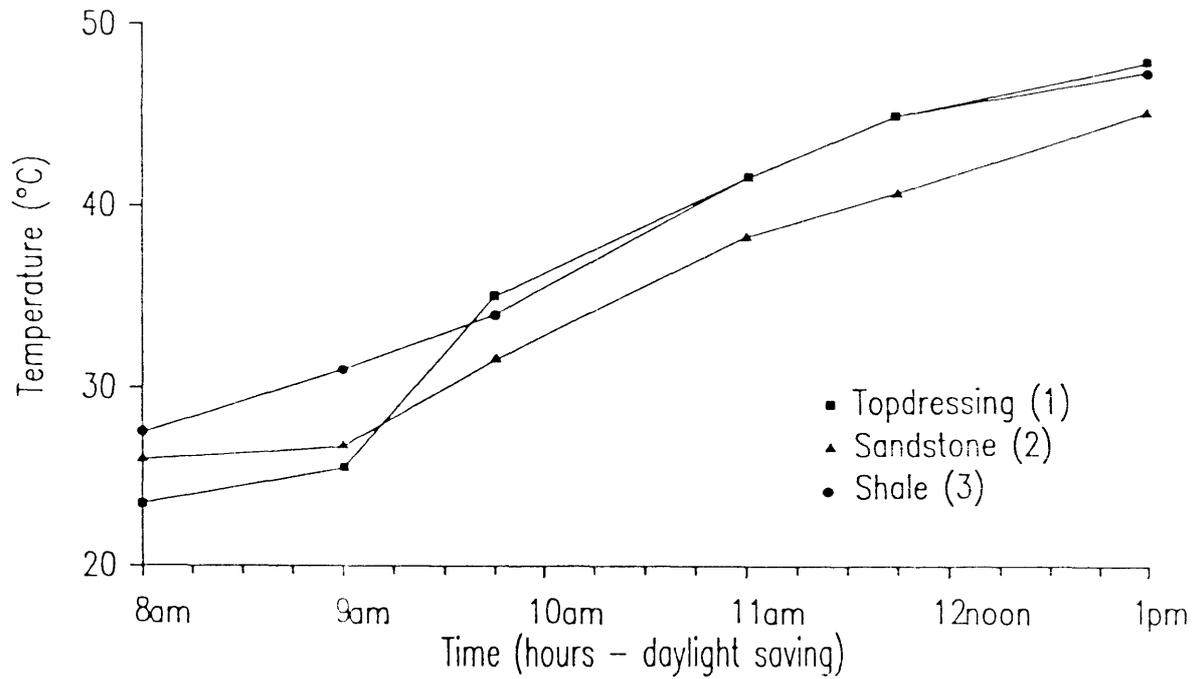


Figure 5.4 Temperature variation over a five hour period at Hunter Valley No. 1. Colliery on the 30th January, 1987.

darker colour. Both the Sandstone (2) and Shale (3) temperature effects could be important in determining the level and/or rate of germination on these substrates in cooler months.

Temperature trends at 2 cm depth were very similar to those at the surface under shade, and may approximate temperatures experienced by the roots of new germinants. Although temperatures at 2 cm depth were up to 7°C lower than those in surface shaded position for equivalent substrates, differences had been reduced to 3 -4°C by 1 p.m. Again Shale (3) initially warmed more rapidly, while Sandstone (2) temperatures were generally lower than the other two substrates

5.2.3. Implications for Rehabilitation

Most of the substrates had ESP values > 6 indicating that sodicity (both physical and chemical effects) could be limiting to plant establishment. The random occurrence of saline materials was experienced (Shale (12) from C.S.P. Lemington) which is in agreement with findings by Croft and Associates (1986) and the SPCC (1983). Nutrient deficiencies were also apparent and would have to be overcome if healthy plant growth were to occur.

Available water capacity did not vary greatly between substrates and was considered high for all substrates. However, substrates with lower permanent wilting points would be able to keep plants alive longer under dry conditions than those with higher permanent wilting points. This could be a critical difference for new germinants.

Substantial monthly and daily temperature differences were apparent. The extent to which these differences affect germination, survival and growth is difficult to predict although daytime temperatures during summer months in particular were extremely high and could be expected to be detrimental to plant establishment. Temperatures at 2 cm depth at these times were also high and root development of new germinants may be affected in addition to the more obvious affect of high surface temperatures on shoot development.

May was the most suitable month for sowing and best met the requirements for successful germination and establishment.