THE BEHAVIOUR OF PLANT NUTRIENTS IN COLLIERY SPOIL OF CENTRAL SCOTLAND

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CONTENTS

Page No.

Summary

CHAPTER 1. COLLIERY SPOIL RECLAMATION

Page No.

CHAPTER 2. TRACE ELEMENT CONTENT OF PLANTS GROWING ON RECLAIMED COLLIERY SPOIL

Page No.

Page No.

CHAPTER 3. SPOIL PHOSPHORUS STATUS AND FACTORS AFFECTING PHOSPHATE ADSORPTION BY SPOIL

Page No.

CHAPTER 4. CATION EXCHANGE REACTIONS IN COLLIERY SPOIL

Page No.

CHAPTER 5. THE USE OF A SOIL COVER IN COLLIERY SPOIL RECLAMATION

Page No.

Summary

The work of this thesis is concerned with the chemical analysis of coal waste of the Central Scottish Coalfield, treating this coal waste as a plant growth medium with the aim of rehabilitating it for agricultural use. There are numerous chemical and physical factors acting against plant growth and Chapters 2-4 highlight three of the main problems viz. high levels of trace elements, low phosphorus content and low levels of exchangeable cations. The physical properties of coal waste are outwith the scope of this thesis although they are discussed in Chapter 1.

Chapter 1 describes the two methods of coal extraction, open-cast and deep mining, the latter being the most commonly used in the United Kingdom, producing some 17.5 million tonnes of coal waste annually (at the present coal production rate) adding to that which has accumulated in the past, estimated in 1982 at 3,000 million tonnes covering 13,000 hectares of land. One of the main problems encountered in spoil reclamation is the heterogeneous nature of the material and the considerable differences in chemical and physical properties found between sites, each site requiring careful study at each stage of operation. Coupled with this are the changes which occur in the spoil on exposure, particularly the oxidation of iron pyrites (FeS_2), producing sulphuric acid which threatens plant establishment and also attacks the spoil matrix.

Chapter 2 examines the problem of high levels of available trace elements found in colliery spoil and their uptake by plants, five elements being chosen for study, namely Fe, Aℓ, Mn, Cu and Zn, although there are many others which are limiting to plant growth. A comparison was made of the trace element content of perennial ryegrass (Lolium perenne) grown on a reclaimed spoil and an arable soil to determine the sites of requirement or deposition within the plant. A field trial was set up to determine the effects of addition of lime and organic amendments to spoil on the trace element content of the sward, results being presented for the first harvest taken after eleven weeks growth. Due to the short growing time no conclusions can be drawn from the organic amendment trial although the effects of trace element content on plant yield are discussed.

After nitrogen, phosphorus is the element most restrictive to plant growth in colliery spoil. Chapter 3 discusses the problem of low levels of available phosphorus and the capacity of the spoil to fix phosphorus in an unavailable form through adsorption on hydrous oxides of iron and aluminium. A growth experiment under controlled conditions in which varying levels of lime and phosphorus fertilizer were added to three spoils showed that both spoil pH and phosphorus deficiency are major factors limiting plant growth.

Chapter 4 discusses the cation exchange capacity and exchangeable base content of colliery spoil i.e. the ability of the material to hold cations in a form which may be released to the spoil solution for plant uptake and the levels of exchangeable Ca^{2+}, Mg^{2+}, K^+ and Na^+ found in a range of spoil samples. Results show that the amorphous iron and aluminium oxides in the spoil are adsorbed on exchange sites, while the amorphous manganese fraction, itself being negatively charged, may hold nutrient cations in an available form.

Colliery spoil being a poor growth medium requiring the addition of high rates of fertilizers for plant growth may be reclaimed more successfully, if more expensively, by the use of a soil cover. Chapter 5 reviews the advantages of the use of soil in spoil reclamation based on evidence collected from 13 reclaimed sites in Central Scotland, these being described fully in Appendix II. This study shows that these advantages, however, may be greater if the nature of the underlying spoil was first considered, as the soil layer does not completely remove the plant from the effects of the spoil.

CHAPTER 1

COLLIERY SPOIL RECLAMATION

1.1 INTRODUCTION

Britain's coal industry successfully launched and sustained the first industrial revolution and today coal remains Britain's biggest energy resource, generating 81% of our needs. 87.8 million tonnes of coal were produced in 1985/86 from the 169 pits operated by the National Coal Board, with smaller contributions totalling about 15 million tonnes from opencast mining and from licensed mines ("Britain's Coal Industry", National Coal Board Publication 1986).

The extraction of any underground mineral deposit such as coal, metal ores, sand, gravel, clay, limestone etc. causes disruption of the land surface, the environmental impact of which is dramatic both aesthetically and physically, with the effect on the surrounding land of the waste heaps, or bings, being felt over a wide area and for a long period if left unchecked.

It is the aim of the work described in this thesis to identify some of the factors militating against plant growth on colliery spoil, to improve it as a growth medium and establish a vegetation cover, thereby reducing the effects of spoil on the environment.

1.2 Methods of Coal Mining

The extraction of coal may be carried out using two methods, opencast mining and deep mining, the latter accounting for approximately 82% of total coal production in Britain.

1.2.1 Opencast mining

Strip, surface, or opencast mining involves exposure of the coal seam by removal of the overlying soil and rocks, and the removal of the coal from above. This method was introduced in Britain in 1941, primarily as a quick method of coal extraction to supply a much needed fuel during the Second World War and its use has continued to the present.

Until the mid 1960s surface mining of coal was not considered feasible when the overburden:seam thickness ratio was 10:1 or less. However, since 1965 this ratio has been increasing due to developments in mining technology and, depending on the type of overburden, deposits with ratios of up to 30:1 may now be economically removed (U.S. Congress Office of Technology 1979). In 1985/86, 55 opencast sites produced 15.6 million tonnes of coal, including more than half of Britain's anthracite (non-bituminous coal).

Surface mining has the advantage that the excavation may be expanded more rapidly than deep mining, it imposes lower capital and labour costs per tonne of coal removed, it is less hazardous to miners' health and lives, and recovers a higher proportion of the coal reserve. However, the environmental impact of surface mining may be much more drastic, involving relocation of farms, diversion of streams and rivers, adjustment in water table levels or removal of forests, some of these changes being irreversible. Restored strip-mined land may never regain its productivity or natural beauty completely, although areas of poor land may be improved greatly and made productive, e.g. marshland restored as arable land or amenity areas.

At the introduction of opencast mining little regard was given to restoration of the mined land. However, as this method continued to be used and the area of unreclaimed land increased, it became necessary to introduce legislation to place the responsibility of reclamation on the producer, a cost which in fact represents only 5% of the total cost of mining operations (Arguile 1975). Restoration of the land is now an integral part of the opencast mining program, and operators have a responsibility to restore it to a suitable standard, usually to its original state, and retain responsibility for rehabilitated land for a minimum period, in Britain and the United States this being five years.

The sequence of operations involved in opencast mining is:

1. Remove topsoil and subsoil and store each separately.
2. Remove overburden and expose the coal seam.
3. Remove coal.
4. Backfill the excavation with overburden, placing the material in stratigraphic order if more than one type of material is removed in order to maintain groundwater and drainage regimes, while creating the required landform and topography.
5. Replace subsoil and topsoil.
6. Plant vegetation.

Replacement of overburden should be carried out continuously, removed material being reinstated in the previous strip and the soil cover replaced as soon as possible to avoid storage which may cause changes in soil properties such as breakdown of soil structure. In this way the land is affected for as short a time as possible and there is little physical evidence left on the surface after coal extraction.

1.2.2 Deep mining

In 1985/86, 169 collieries under the control of the National Coal Board produced 87.8 million tonnes of coal using deep mining methods. Underground mining is considerably more complex than surface mining, involving the construction of tunnels and railways, and the transportation of men and coal at depths often exceeding 1000 metres (the deepest coalface in Britain is at Wolstanton Colliery, Staffordshire, at 1102 metres). After removal of the coal seam the tunnel roof may be supported in two ways, either by constructing supports behind the cutting machinery as it travels along the seam, or by leaving pillars of coal in place as a natural roof support. In the latter case, the deeper the mine the larger the pillars which must be left relative to the amount of coal extracted. This method was used widely in deep mines in the United States where it is estimated that the average amount of coal left behind as roof supports in old mines is 42% (Bryenton and Rose 1976). An alternative form of deep mining is "longwall" mining which allows the roof of the tunnel to collapse after the cutter has removed a cross-section of the seam and moved forward. This results in the extraction of practically the whole coal seam but results in deformation of the overlying strata and subsidence of the surface in shallow mines. By the end of 1985 some 47,000 claims were outstanding against the National Coal Board regarding land subsidence, 18,500 claims being received in that year, while 29,000 cases were resolved at a cost to the N.C.B. of £99 million (National Coal Board Annual Report 1984/85).

Deep mining is marked by the deposition on the surface of waste, consisting mainly of shale, sandstone and other rock fragments removed from strata overlying the coal seam. It is estimated that for every five tonnes of saleable coal extracted by deep mining operations, one tonne of waste material is brought to the surface and dumped in heaps, or bings, close to the pit head (Richardson 1976), thus leaving a blot on the land which affects the surrounding area, not only from an aesthetic point of view, but through dust comtamination and seapage of contaminated water into nearby fields and water courses (see section 1.5). By this estimation, approximately 17.5 million tonnes of waste will be deposited on the surface from deep coal mining in the United Kingdom each year, at the present rate of coal production, adding to that which has been produced in the past, estimated at approximately 3,000 million tonnes by 1974 (Gutt et al. 1974). Reclamation of the land taken up by this waste may be accomplished either by dealing directly with the spoil, establishing plants by improving the condition of the material itself, or by using a cover of soil to remove the plants to some degree from the effects of the spoil. The effects that spoil has on plant growth are dealt with in Chapters 2-4 while the use of a soil cover is reviewed in Chapter 5.

1.3 Coal Production Throughout the World

In 1913 Great Britain, Germany, France and the United States together produced more than 90% of the world's coal, this production accounting for virtually all the world's energy supply. However, over the following years there was a gradual shift from the use of coal to oil and gas as energy sources, and by the beginning of this decade coal accounted for only about 30% of the energy need. Despite this relative decline in use, coal still produces 40% of the world's electricity and is virtually the only energy source for the iron and steel industry.

Table 1.1 shows the distribution of world coal deposits and the amount of technically recoverable coal based on present extraction techniques (Wilson 1980). However, it may be assumed that future developments in mining technology will increase the percentage of the geological reserve which will be economically recoverable.

1.3.1 Coal production in Great Britain

Total coal production in Britain in 1985/86 was 102.9 million tonnes - 87.8m.tonnes from deep mines, 13.6m.tonnes from opencast mines and 1.5m.tonnes from licensed mines.

Of the total amount of coal produced in Britain approximately 75% is used by power stations to produce 80% of our electricity, the remainder being used for industrial and domestic burning ("Britain's Coal Industry", National Coal Board Publication 1986). The National Coal Board at present works 169 collieries compared to 1,000 which were taken over at nationalisation of the industry in 1947.

Coal production in Scotland has been declining over recent years, the production figures for the past six years being:

1980/81	7,689,227 tonnes
1981/82	7,162,253 "
1982/83	6,569,300 "
1983/84	5,216,924 "
* 1984/85	280,332 "
1985/86	4,180,992 "

*Production drastically reduced due to miners' dispute.

Figures from N.C.B. personal communication.

These figures are considerably lower than the 20-30 million tonnes produced annually in the 1940-50 period.

Figure 1.1 shows the main coal producing areas of Scotland.

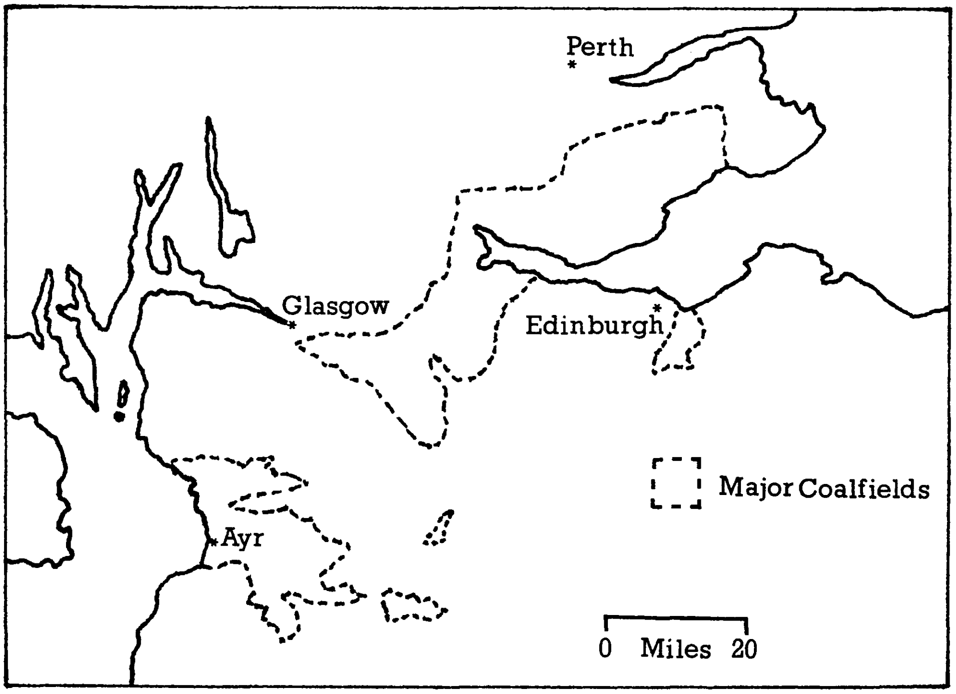


Figure 1.1. Major coalfields of Scotland.

Table 1.1 Distribution of world coal reserves (Wilson 1980)

	Geological Reserves (Million Tonnes)	Technically & Economically Recoverable Reserve (Million Tonnes)	Coal Production (1977) (Million Tonnes)	$\frac{\text{Reserve}}{\text{Production (1977)}}$ *
USA	2,570,000	178,000	623	286
USSR	4,860,000	110,000	516	213
China	1,438,000	99,000	500	198
Poland	126,000	21,000	197	107
United Kingdom	164,000	45,000	123	366
West Germany	247,000	34,000	121	281
India	57,000	34,000	100	340
Australia	262,000	27,000	82	329
South Africa	58,000	27,000	81	333
Canada	115,000	9,000	27	333
Other Countries	228,000	52,000	405	128
TOTAL	10,125,000	636,000	2,775	229

* $\frac{\text{Reserve}}{\text{Production}}$ (1977) Predicts the number of years coal reserves would last based on 1977 production

1.4 Coal Waste Disposal

Dumping of coal mine waste in the past was indiscriminate and done in haste, the prime objective of the mine operators being to extract the coal as quickly as possible, and little thought or planning went into the methods of waste disposal from the point of view of the effects of the bings on the environment. As a result the bulk of the material was piled high on nearby land from a conveyor line, with waste dumped to the front, forming a progressively higher cone (rising in cases up to 50m) with a long sloping tail up which the conveyor tracks ran (Plate 1). Other bings formed by dumping waste from more than one line may have a number of peaks or a plateau top, again having steep slopes.
Bings formed in these ways have steep, unstable slopes which tend to slip and dislodge the roots of any plants which are able to establish themselves naturally (Plate 2). However, weathering and slippage may result in the accumulation at the base of the slopes of material dislodged from above, resulting in some plant establishment at the base with the upper slope remaining bare.

Underground mining uses large quantities of water, both during the removal process for dust suppression and the cooling of machinery, and for the washing of coal on the surface. Waste water containing suspended mineral matter is pumped into lagoons and the water removed by evaporation and seepage, leaving large areas of "washery" waste containing fine coal, silt and clay. Such areas may remain as a slurry for many years due to water being held in the matrix of fine particles or if they dry naturally will lack suitable structure to support plant growth.

PLATE 1 – Typical bing form.

PLATE 2 – Evidence of surface "creep".

In order to reduce the amount of spoil dumped on the surface, other means of disposal have been sought, with some success, although the proportion of the total amount of spoil produced which is utilized by these methods is small. Since only 1/6th of the volume of material extracted is left as waste there is ample space in the worked out mine shafts to accommodate it all. However, although some stowage in mine workings is carried out in the Ruhr coalfield in West Germany, to do so in Britain would involve modification of the existing extraction machinery at great expense to replace all of the waste underground.

A relatively small amount of spoil is used in back-filling of quarry workings, roadworks and other civil engineering activities due to the fact that good compaction can be achieved giving a firm base for construction, and considerable research has been carried out into the use of spoil as an aggregate in concrete production (Bryenton and Rose 1976) and in brick production (Gutt et al. 1974, Lawson and Nixon 1978). Bryenton and Rose (1976) outline 26 "coal-crete" mixes and recommend its use for the construction of roof supports during coal extraction, thereby giving greater coal production from each mine by eliminating the need to leave coal pillars (see section 1.2.2).

It was estimated in 1971/72 that about 50 million tonnes of spoil were deposited on inland sites in Britain in that year and a further 6 million tonnes were dumped at sea, with about 7 million tonnes of the land-dumped material being utilized; 80-90% for backfilling etc. and 10-20% for concrete aggregate and brick manufacture, mainly in Scotland (Gutt et al. 1974).

James (1982) estimated that existing tips in Britain owned by the National Coal Board contained 2,500 million tonnes of spoil and covered 11,000 hectares with a further 500 million tonnes in private ownership. It is unlikely that such a stockpile of material will be significantly reduced by such methods as those outlined above, but with current developments it may be possible to utilize a greater part of the present waste production, thereby reducing the rate at which land is taken up by dumping.

1.5 The Effect of Coal Bings on the Environment

Despite the fact that a bing may only cover a relatively small area, considering the mass of material contained therein, it exerts an effect on the surrounding land over a wide area.

1. As mentioned in Section 1.4, bings usually have steep-sloping sides and often dominate the landscape, as they are visible for a considerable distance. The aesthetic effect on the surrounding land is therefore great, and for this reason alone reclamation is justified and desirable, to regrade and cover the spoil with vegetation in order to blend it into the surrounding landscape. In an effort to reduce the visual impact of mining waste on the environment, a system of progressive reclamation has been adopted in some areas, notably in the Ruhr area of the Federal Republic of Germany. Waste is deposited in layers, with succeeding layers rising in a step formation and the whole tip having a gentler slope than bings formed by overhead tipping.

As each layer is formed the previous layer is treated and planted with trees, thus as dumping continues, the lower slopes of the bing are reclaimed and screened from view. Such problems as these are minimised or perhaps absent if the mine spoil is dumped in remote areas as is often the case in the United States and Canada where coal reserves have been extracted in mountain areas and the pressure on the operators to reclaim the spoil is reduced, in which case a low maintenance cover is required and the reclaimed land is left as a "wilderness" area.

2. Unprotected spoil heaps, i.e. those lacking a vegetation cover, are sources of dust which may be carried a considerable distance, contaminating surrounding soil, streams and vegetation, discolouring nearby buildings and aggravating human respiratory disorders. Bings therefore affect the environment long after the mines cease to work, and continue to do so until steps are taken to stabilize the surface. The most effective means of reducing the amount of wind-borne pollution is to establish a cover of vegetation, which protects the surface from the direct effects of wind by the wind-breaking action of the plants and also stabilizes the surface of the spoil through the anchorage of plant roots.

3. Dumped material inevitably contains a certain amount of coal, especially older bings which were formed when methods of extraction were less efficient. Air trapped within the spoil and contact of the exposed material with the atmosphere cause oxidation of coal material, with a subsequent liberation of heat and the release of noxious fumes

to the atmosphere. Exposure causing combustion leads to the emission of carbon dioxide, sulphur dioxide, carbon monoxide and nitrous oxides, with the oxygen deficient conditions under which combustion within the tip occurs causing the release of hydrogen sulphide and ammonia. All gaseous releases will be carried away by wind or dispersed in the atmosphere. However, when prevailing weather conditions are unfavourable, the unpleasant odour of hydrogen sulphide and sulphur dioxide blow over the surrounding area.

4. Sheet and gully erosion by rain falling on steep, bare slopes carries large quantities of fine material into streams and rivers (Richardson 1976). Establishment of a vegetation cover greatly reduces the loss of fine material in this way, by increasing the amount of ground litter, and through the action of plant roots in holding spoil particles in the root zone.

5. Acid produced by the oxidation of iron pyrites (see 1.6.4) leaches into streams, lowering the pH and affecting aquatic life downstream of the bing. Acid drainage may also carry with it large amounts of metal ions in solution which also affect water quality. Pulford et al. (1983) found a dramatic increase in the levels of Fe, Al, Mn, Cu, Zn, Co, Ni and K in a stream in E. Scotland in contact with a coal bing, together with a marked decrease in pH, these effects persisting for a considerable distance downstream. Similar results have been reported by Blessing et al. (1975) for stream pollution from abandoned mines in Australia, and by Benza and Lyon (1975), who estimated that 3,000 of

the 50,000 miles of streams in Pennsylvania were polluted by acid mine drainage.

1.6 The Nature and Properties of Colliery Spoil

Waste brought to the surface by deep mining operations consists mainly of shale, sandstone and other fragments of rock which have not been exposed to weathering and the slow processes of soil formation, and which will therefore undergo considerable change, both chemically and physically, on exposure. This material when first exposed may have a pH of 6-7, suitable for plant growth, and if it has suitable texture and the slope is not too steep, natural colonization by grasses and herbaceous species can occur. Waste of low pyrite content can support a sparse grass and shrub cover after a few years, although many inter-related factors play a part in determining the extent of colonization and spread of pioneer species. Natural colonization will occur to varying degrees on most sites, but in many cases the vegetation will be short-lived due to changes in the physical and chemical properties of the spoil which take place on weathering (Costigan et al. 1981).

The use of pioneer species as a bio-indicator to predict the conditions that will probably eventually prevail in a spoil is used by Donovan et al (1976), who recommend the use of such species in the reclamation program as initial surface stabilizers, before planting deeper rooted trees and shrubs. Survival of naturally colonizing plants depends on their tolerance to the spoil conditions, e.g. high levels of trace metals, those without such adaptations quickly dying out.

The problem of high levels of trace metals in spoil is common on acid sites and this aspect of plant adaptation has received much attention. Tolerance to one metal is not accompanied by tolerance to any other, except in the case of Ni and Zn (Gregory and Bradshaw 1965), with individual species having a wide range of tolerances. The number of surviving species is found to be inversely related to the concentration of available metals in the spoil (Walley et al. 1974, Wu et al. 1975) and the exclusion of some species from mine areas is due to their inability to develop metal tolerance (Gartside and McNeilly 1974).

Most bings, if left without regrading or treatment, would eventually attain a degree of cover through natural invasion of grasses and herbaceous species. Bradshaw and Chadwick (1980) listed 105 plant species occurring naturally on 22 untreated colliery spoil bings in Yorkshire and found that the number of species on a site increased as spoil pH increased, while site age had no influence. This is in contrast to the situation on disused agricultural land where succession leads to an increase in the number of species with time, site age being the main determining factor in the early years of colonization. Naturally revegetated sites, however, will be sparsely covered, as spoil conditions remain variable and nutrient levels will not be adequate to sustain growth in many areas, although some nutrients will be added through decaying plant roots and litter, and some nitrogen will be fixed by naturally invading legumes. A controlled program of fertilization and spoil amendment gives some assurance of establishing a vegetation cover and initiating improvements through the cycling of plant nutrients, e.g. nitrogen fixation by planting a suitable seed mixture (see 1.7), and in this way growth can be achieved which, although requiring monitoring and after-care, will provide an effective means of reducing the effect of the spoil on the environment.

1.6.1 Spoil variability

Variations in spoil properties between sites makes it difficult to approach one site based on the experiences of another; even when adjacent bings composed of material of similar age and extracted from the same strata are compared, it may be found that spoil properties vary greatly. Considerable variation in spoil properties is found not only between sites, but also within each site, due in part to the fact that mining operations may have continued at one site over a number of decades, with the result that some material will have undergone weathering and chemical change, while other material on the same site will be fresh, unweathered spoil. The situation is further complicated by the fact that material of different physical and chemical properties will be dumped together and in some cases material from nearby mines will be taken for disposal at other workings, a practice which has gained popularity in the United States and West Germany in recent years, where spoil from a number of sites is disposed of in a common dumping area (Petsch 1975).

Variations in properties within a bing therefore add to the problems encountered when reclaiming a spoil. Whereas a uniform arable soil can be sampled adequately by taking regularly spaced samples which may be bulked together and sub-sampled, such a procedure is inadequate when sampling colliery waste, since significant areas of the site may be missed and "problem" areas will be insufficiently treated. Examples of such areas include areas with a particularly low pH, high pyrite content and potential acidity, poor structure such as washery waste, or low water holding capacity, and therefore the site must first be split into recognizable areas on a broad basis and each area sampled separately, with samples taken randomly within each section. Initial sampling before earthmoving may in some cases pin-point such areas and these may be dealt with at an early stage and buried during regrading. However, during earthmoving, similar material may be brought to the surface and therefore an intensive sampling scheme should be carried out after earthworks, the initial sampling serving only as a guideline for regrading.

Despite intensive sampling of a regraded site it is not guaranteed that the area, once revegetated, will not regress and require further treatment due to the changes which occur in spoil, as it is impossible to amend all spoil sufficiently in a single operation.

1.6.2 Spoil as a soil parent material

Spoil fertility cannot be judged simply by the chemical composition of the material, since its physical properties have a significant influence on its suitability for plant growth. Productive soil, with adequate nutrient reserves and favourable structure, can be downgraded in just a few years through poor soil management which does not give adequate consideration to soil texture. This may result in structural breakdown leading to impeded drainage, compaction and waterlogging, giving a reduction in crop yield. Since colliery spoil in many cases lacks suitable structure, such considerations are of greater importance.

There are 5 major factors which influence the type of soil that develops from any parent material:

1. Climate - particularly temperature and precipitation.
2. Nature of parent material - texture, structure, chemical and mineralogical composition.
3. Living organisms - especially native vegetation, microfauna and microflora.
4. Topography.
5. Time period over which the parent material has been subjected to the above factors.

Colliery spoil can be regarded as a parent material from which a soil will eventually develop, and in a situation where a spoil heap is being reclaimed, a sixth soil forming factor must be included, which is the influence of man, due to his ability to alter the spoil environment and affect many of the above five factors; he can alter the physical and chemical character of the spoil, influence the flora and fauna populations, alter the topography, and regulate the water regime by drainage and irrigation. The main factor to which spoil has not been subjected is time, and little can be done to accelerate the weathering process. For this reason, sites which have been reclaimed by regrading and the improvement of physical and chemical properties, may deteriorate through weathering of the spoil, causing changes which may destroy the vegetation cover. Periodic sampling of the spoil and visual inspection of the vegetation growth will ensure early diagnoses of changes, and further treatments can be carried out to maintain growth and prevent further regression.

1.6.3 Physical factors acting against plant growth

1.6.3.1 Slope and surface instability

Most bings have steeply sloping sides formed by the dumping of material from the top, giving a natural angle of repose of unregraded spoil slopes of 32-36° (Down 1975(a)). Wind and water erosion on such slopes is severe, with fine material being removed constantly, disrupting the surface and preventing plant roots gaining a hold. Coarse surface material often has a "platey" structure, forming an unstable surface which tends to slip, preventing plant rooting. Down (1975(a)) studied 9 tips of ages covering the range 0-178 years, each the product of mining in similar horizons in the Somerset coalfield, and found that over this time scale the surface remained unstable, exceeding that found on a natural soil slope, although the instability decreased markedly within 21 years.

Regrading and establishing a vegetation cover stabilizes the surface and reduces the rate of erosion (Dennington and Chadwick 1978). However, regrading should be minimized with spoils having a high percentage of clay-sized particles due to problems of compaction which could lead to impeded drainage and reduced infiltration rates (Richardson 1976, Down and Stocks 1977). All regraded land should have a minimum slope of 1-2% to prevent water impoundment with land which is to be used for agriculture being graded to a slope of <25% and land for recreation having a slope of <35% (Donovan *et al.* 1976).

1.6.3.2 Texture and water holding capacity

Colliery spoil texture varies greatly with the parent material and with the age of the spoil. Coarse textured spoils have a low water holding capacity and suffer through lack of moisture in dry weather, while heavy textured spoils are easily waterlogged. Down and Stocks (1977) found a rapid decrease in the proportion of coarse material (> 9530 µm) and a consequent increase in the proportion of material in the range 1,000-2,000 µm in the first 40 years. Weathered clay particles, however, may be washed down the profile leaving a largely unaltered surface layer with an accumulation of finer material at depth. Surface drought may still occur, but if plants can be established during periods of sufficient rainfall and have an adequate root system, they may be sustained by the store of water below.

It is therefore important at the planning stage to not only consider the type of material to be reclaimed, but also the rooting habits of the species in the seed mixture.

1.6.3.3 Bulk density

Spoils generally have higher bulk densities than natural undisturbed soils for two main reasons; firstly, spoil contains a higher percentage of rock fragments (shale and sandstone), and secondly, the rock fragments in spoil tend to be less weathered than those of a soil

(Smith et al. 1971). Bulk densities of both soil and spoil can be increased markedly by the passage of heavy machinery (Blackwell et al. 1985), spoil being particularly at risk due to the absence of an organic fraction and its stabilizing effect on spoil structure.

As a result of the high bulk density of a spoil, root penetration is hindered, resulting in poor plant survival. Plant roots improve spoil structure by reducing bulk density and allowing a freer movement of air and water through the matrix, while adding an organic manure or mulch also improves structure and encourages plant rooting.

1.6.3.4 Surface temperature

Colliery spoil, being dark in colour, absorbs more heat than lighter surfaces. Surface temperature differences of 10°C are not uncommon on adjacent south-facing soil and spoil slopes (Richardson 1976, Down and Stocks 1977, Jaynes et al. 1983) and surface temperatures in excess of 50°C have been reported (Fitter and Bradshaw 1974). Slopes with a vegetation cover absorb less heat than bare slopes due to the heat dissipating abilities of plants, by reducing the amount of sunlight directly hitting the surface and through evaporation from plant leaves, creating a cooler microclimate just above the spoil surface. Thus if a vegetation cover can be established, spoil conditions will be improved further, by reducing the temperature range experienced on bare spoil.

1.6.3.5 Drainage

Bare spoil slopes are often cut by deep gullies formed by rain running into channels and removing fine particles of material to the bottom of the slope, either to be deposited there, or carried into drains where problems of silting-up may occur. In order to reduce run-off, it is necessary to increase the permeability of the surface, and also to "hold" the water on the surface for a longer time to allow infiltration. Retention time can be increased by reducing the slope (not too much or water accumulation will occur), or by establishing a plant cover, which will also add organic matter to the spoil and increase permeability (Dennington and Chadwick 1978, Bradshaw and McNeilly 1981).

1.6.4 Chemical factors acting against plant growth

The two main chemical factors acting against plant establishment are the production of acid due to pyrite oxidation and the lack of essential nutrients in the spoil.

1.6.4.1 Problems due to pyrite oxidation

Oxidation of iron pyrites (FeS_2) is the principal chemical change which takes place in spoil, most other changes occurring as a result of acid production through oxidation on contact with air and water (Chadwick 1973, Carrucio 1975, Pulford and Duncan 1975, Costigan et al. 1981).

In the coalfields of Central Scotland, coal waste brought to the surface may contain up to 5% of pyrite which on oxidation produces sulphuric acid and large amounts of soluble salts (Pulford 1976). The chemical oxidation of iron pyrites can be represented in a simple form by the following equations:

(a) Oxidation by oxygen

$$2FeS_2 + 2H_2O + 7O_2 \longrightarrow 2Fe^{2+} + 4H^+ + 4SO_4^{2-}$$

$$4Fe^{2+} + 10H_2O + O_2 \longrightarrow 4Fe(OH)_3 + 8H^+$$

(b) Oxidation by Fe^{3+}

$$FeS_2 + 14Fe^{3+} + 8H_2O \longrightarrow 15Fe^{2+} + 2SO_4^{2-} + 16H^+$$

Bacterial oxidation of pyrite also takes place, reduced sulphur compounds being used as an energy source by Thiobacillus thio-oxidans and oxidation of iron being carried out by Thiobacillus ferro-oxidans, the latter being able to continue this process down to pH 4.0.

Apart from the direct effect of acid on plant roots, a number of spoil reactions which affect plant growth occur, i.e. secondary effects of acidity:

(I) The presence of high hydrogen ion concentrations results in the replacement by H^+ of the bases on the exchange sites of the spoil, leaving these free in solution and readily leached from the root zone (See Chapter 4).

(II) The breakdown of clay lattices by the action of acid reduces the spoil's cation exchange capacity, and thereby limits its ability to hold nutrients for plant uptake.

(III) High levels of trace elements, particularly Fe (from pyrite oxidation), Al (from clay lattice breakdown), Mn, Zn, Cu and other trace elements from mineral breakdown, are brought in to solution as the pH falls, resulting in possibly toxic levels of these elements being available to plants, Pulford et al. (1983). This may result in high trace element uptake, causing plant injury (See Chapter 2) and can also affect the uptake of other plant nutrients e.g. high Al uptake by roots causes a reduction in phosphate uptake (Clarkson 1966, 1967).

(IV) The large amounts of soluble salts produced, both from pyrite oxidation and through acid breakdown of spoil minerals, may cause plant injury, by increasing the osmotic pressure of the soil solution, resulting in water being drawn from the plant roots (Donovan et al. (1976), Bradshaw and Chadwick (1980)). Struthers (1964) exposed a number of strip mine spoils to natural weathering conditions, collecting the leachates for analysis. He found, for the five spoils in his survey which were unsuitable for plant growth, an average yield of 125 tonnes of salts per hectare in 11.5 months, with one sample producing 270 tonnes, in comparison to only 145 Kg of salt leached from an acid silt loam soil under similar conditions.

(V) Fe and Al brought into solution at low pH, and their oxide and hydrous oxide films adsorb or complex phosphate, inducing phosphate deficiency (Pulford and Duncan 1975, Costigan et al. 1982).

Spoil pH may drop rapidly on exposure and can fall to pH 2-3 after a few years, thus discouraging plant growth and destroying any early colonizing plant species. Under these conditions of very low pH spoil material is further attacked, exposing fresh surfaces which may be oxidised, resulting in further acid production. Weathering processes will eventually exhaust the acid potential due to pyrite and the high salt and trace metal levels will be leached from the upper layers, the pH will rise again and a less hostile but nutrient deficient medium will result.

1.6.4.2 Nutrient availability

There are 17 elements at present regarded as essential for plant growth (Brady 1974). These can be divided into elements required in relatively large amounts (macronutrients) and those needed in trace amounts (micronutrients or trace elements) - Table 1.2.

Table 1.2. List of essential macronutrients and micronutrients*
(Brady 1974)

Macronutrients		Micronutrients	
Carbon	Phosphorus	Iron	Copper
Hydrogen	Potassium	Manganese	Zinc
Oxygen	Calcium	Boron	Chlorine
Nitrogen	Magnesium	Molybdenum	Cobalt
	Sulphur		

---

*Other minor elements such as sodium, fluorine, iodine, barium, strontium, silicon, vanadium and beryllium, have not been shown to be essential, but some may increase plant growth and may be shown to have a role in biochemical reactions.

Table 1.3 shows the content in coal of the elements discussed in the following sections.

A. Macronutrient availability in unamended spoil (N,P,K,Ca,Mg,S)

Of the 9 macronutrients listed in Table 1.2 carbon, hydrogen and oxygen are obtained from atmospheric carbon dioxide and water, while the remaining six elements must be taken up by plant roots from the spoil solution.

Nitrogen

Nitrogen may be regarded as the element most restrictive to plant growth in colliery spoil (Williams 1975, Bloomfield 1982).

The importance of nitrogen as a plant nutrient is evident from the fact that it is a constituent of the protein and nucleic acids of the plant, thus being essential for normal plant growth. Approximately 98% of nitrogen in soil is combined with the organic fraction as proteins, amino sugars and nucleic acids, and can only be used by plants after release through microbial action (mineralisation). Since the level of mineralisable organic matter in spoil is very low compared to a soil (Williams and Cooper 1976), nitrogen comes almost entirely from the inorganic fraction. The total nitrogen content of colliery spoil is 1,000-8,000 µg/g (Pulford 1976), similar figures being reported by Chadwick (1981), who found the following nitrogen forms in spoil:

Total nitrogen	2790-5900 μg N/g
Fossil organic nitrogen	2090-4010 μg N/g
Fixed ammonium nitrogen	330-1480 μg N/g
Organic nitrogen	50-410 μg N/g
Available nitrogen	20-30 μg N/g

Coal material in the spoil, although containing fossil nitrogen, will break down to release only a very small amount for plant use, thus it is evident that there is a very limited pool of available or mineralizable nitrogen in spoil, and fertilization is needed to encourage plant growth.

The following are the three main methods used to raise spoil nitrogen levels:

(a) Addition of an organic manure such as animal manures or plant residues, which release nitrogen slowly as they are broken down. Mineralization of organic matter releases ammonium and nitrate ions, which are removed from the soil through plant uptake, assimilation into the bodies of soil microorganisms(and thereby brought back into the organic reservoir), lost by denitrification (the reduction of nitrate to gaseous nitrous oxide and free nitrogen gas) and leaching from the spoil (Dennington and Chadwick 1978). Denitrification is encouraged under conditions of low oxygen supply in the presence of a readily available source of nitrate nitrogen. Poor aeration caused by waterlogging or heavy spoil texture encourage the loss of applied nitrogen fertilizer, particularly if the spoil has been limed, since it is encouraged at higher pH. Incorporation of an organic material has the added advantage of improving spoil structure (reducing losses through denitrification) and building up the microbial population of the spoil.

(b) Addition of an inorganic fertilizer, which supplies a readily available source of nitrogen, either as ammonium or nitrate ions. A single dose of a compound fertilizer containing N,P and K applied at seeding, together with any necessary spoil amendments such as lime will initiate plant growth. However, losses due to denitrification and leaching will occur and re-application at a later stage may be required. Appendix II shows the rate of compound fertilizer used on 13 sites at reclamation, generally N.P.K. fertilizer with a ratio of 20:10:10 at a rate of 350-510kg/ha. Gemmell (1973) recommends application of a 20:10:10 fertilizer at 250kg/ha at seeding, with annual application of a 15:15:15 fertilizer at the same rate, although on many sites this was found to be a minimal treatment, simply maintaining plant growth, with no long-term spoil improvement through humus and organic matter build up.

Reapplication as a top dressing to growing crops, either of compound fertilizer or of nitrogen alone if the vegetation shows deficiency symptoms, is essential if growth is to be maintained, a careful monitoring of the condition of plant growth will prevent regression due to deficiency resulting from changes in spoil conditions.

(c) The inclusion of a leguminous species in the seed mixture, thus reducing the dependency of the crop on nitrogen fertilizer additions. Fixation of atmospheric nitrogen by root-colonizing bacteria supplies nitrogen directly to the spoil for immediate uptake, thus reducing losses through leaching which will occur in (a) or (b). Bradshaw _et_ _al_. (1973) found that legumes inoculated with N-fixing bacteria fixed 52-112 kg N/ha in a year, while Palmer and Iverson (1983) found white clover (_Trifolium_ repens) to add 146-167 kg N/ha/year to spoil, nitrogen fixation being higher with high phosphate treatments (Hue and Adams 1984). Sowing of a mixed grass/clover sward reduces the need for nitrogen fertilizer addition, and in fact if sufficient N is added to establish grass growth, the legume component of the sward may be suppressed and can die out (Chadwick 1981). Leguminous nitrogen fixation is inhibited by an abundance of available nitrogen, and legume growth is not increased by supplying nitrogen to spoil, it simply reduces the amount fixed, thus reducing the beneficial effects of sowing a mixed sward (Child 1980).

The proportion of legumes used in the seed mixture on Scottish spoil varies from 7-15% (See Appendix II), almost entirely red clover (Trifolium pratense) and white clover (Trifolium repens), far lower than the level used in wilderness strip-mine spoil in Canada, where up to 60% of the seed mixture may be leguminous (Watkins, personal communication). Bradshaw and Chadwick (1980) found 11 leguminous species occurring naturally in a study of 22 naturally vegetated sites in Yorkshire, the most commonly occurring species being birdsfoot trefoil (Lotus corniculatus) - See Table 1.4.

Spoil conditions, especially pH and phosphorus availability, will govern the rate of natural colonization by legumes and will determine whether or not a particular species will survive. With liming and fertilizer addition many species will therefore survive on reclaimed spoil, however, the most frequently sown species, red clover (Trifolium pratense) and white clover (Trifolium repens), are those used on arable land, since those such as lupins (Lupinus sp.) or birdsfoot trefoil (Lotus corniculatus), although they add nitrogen and organic matter to the spoil, are of little use in an arable situation.

Table 1.3. Chemical composition of coals of the United States (U.S. National Research Council 1980)

Element (%)	Anthracite	Bituminous	Sub-Bituminous	Lignite	Average	Estimated Worldwide Average
Nitrogen (N)	-*	-	-	-	-	-
Phosphorus (P)	-	-	0.10	0.007	-	0.05
Potassium (K)	0.24	0.21	0.06	0.20	0.18	0.01
Calcium (Ca)	0.07	0.33	0.78	1.20	0.54	1.00
Magnesium (Mg)	0.00	0.08	0.18	0.31	0.12	0.02
Sulphur(S), Total	0.80	2.70	0.70	1.70	2.00	2.00
Sulphur(S), Pyritic	0.35	1.70	0.36	0.68	1.19	-
Iron (Fe)	0.44	2.20	0.52	2.00	1.60	1.00
Aluminium (Al)	2.00	1.40	1.00	1.60	1.40	1.00
Manganese (Mn)	0.002	0.010	0.006	0.015	0.010	0.005
Copper (Cu)	0.003	0.002	0.001	0.002	0.002	0.002
Zinc (Zn)	0.002	0.005	0.002	0.003	0.004	0.005

*Dashes indicate no data included in source

Table 1.4. Occurrence of legume species on 22 naturally vegetated sites (expressed as % of sites) - Bradshaw and Chadwick (1980)

		% of sites
Birdsfoot trefoil	(*Lotus corniculatus*)	27
Red clover	(*Trifolium pratense*)	14
White clover	(*Trifolium repens*)	14
Common vetch	(*Vicia sativa*)	5
Lupins	(*Lupinus* sp.)	5
Black medick	(*Medicage lupulina*)	5
Gorse	(*Ulex europaeus*)	5
Scotch broom	(*Cytisus scoparius*)	5
Yellow trefoil	(*Trifolium dubrium*)	5
Bitter vetch	(*Lathyrus montanus*)	5
Kidneyvetch	(*Anthyllis vulneraria*)	5

Phosphorus

Phosphorus deficiency is a major factor inhibiting plant growth in colliery spoil and is dealt with in depth in Chapter 3.

Levels of available phosphorus are low due to the low solubility of most inorganic phosphorus compounds, giving a low concentration in solution, and the situation is compounded by the fact that reactions occur in the spoil which "fix" soluble phosphorus and render it unavailable to plants, thus reducing the effectiveness of added soluble phosphorus fertilizers. The mechanicms by which phosphorus availability is reduced are:

(a) Precipitation by complex formation, mainly with Fe, Al and Ca. As spoil pH falls below pH 5.5, soluble Fe and Al concentrations increase greatly, causing precipitation as colloidal Fe- and Al-phosphates. With time these colloidal forms slowly convert to the crystalline variscite $AlPO_4.2H_2O$ and strengite $FePO_4.2H_2O$, availability decreasing with increasing crystallinity. Liming spoil, although reducing the soluble Fe and Al levels, results in precipitation with soluble Ca, Ca-phosphates forming above pH 6.0.

(b) Adsorption on the surface of hydrous oxides of Fe and Al forming insoluble phosphates. Oxides and hydroxides of Fe and Al on clay surfaces are positively charged and react with soluble phosphate ions $H_2PO_4^-$ and HPO_4^{2-}. Phosphate ions fixed in this way form stable complexes and lose exchangeability, with up to 75% of added phosphate being lost to plants by this process in the first year after application (Williams 1950).

(c) Isomorphous replacement in crystal lattices by which phosphate ions may substitute for silicate or hydroxyl ions.

By these processes fertilizer phosphorus is removed from the available pool and it is often necessary to re-apply it to make good the loss and maintain a vegetation cover (Roberts et al. 1981, Bloomfield et al 1982).

Potassium

Potassium is the most common nutrient cation in plants, being involved in cell structure and essential for photosynthesis. Its availability in some spoils may be limiting to plant growth, although generally not such a limiting factor as nitrogen or phosphorus (Barnhisel and Massey 1969, Bradshaw and Chadwick 1980). Bloomfield et al. (1982) found no response to added potassium fertilizer in growth experiments with 8 spoils, to which adequate nitrogen and phosphorus were added, due to the acid breakdown of montmorillonite and illite clays supplying sufficient available potassium to maintain plant growth. Although fixation of potassium occurs in soils the reaction is favoured under alkaline conditions (Russell 1973), thus acid spoil conditions reduce the potassium fixing capacity and maintain potassium supply through clay mineral attack.

Calcium, Magnesium and Sulphur

Calcium, magnesium and sulphur are components of plant tissue and are essential for healthy plant growth.

Calcium is found in plant cell walls and is also involved in enzymic reactions within the plant. Calcium levels in coal waste vary considerably between sites, the main source being limestone strata associated with coal deposits. Kimber (1982) found calcium levels ranging from 0-35,700 μg/g, extractable by 0.5M acetic acid in Central Scottish coal wastes, with no overall significant correlation between pH and the level of extractable calcium. Spoils of the Scottish coalfields are almost invariably acid and require the addition of lime at reclamation, thus raising the level of available calcium sufficiently to satisfy plant requirements, although unamended spoil may exhibit calcium deficiency.

Magnesium is essential to all plants as it is a constituent of the chlorophyll molecule and is involved in enzymic reactions related to phosphate transport. Magnesium levels vary similarly to calcium due to their association in calcium-bearing minerals. Kimber (1982) found levels of 0.5M acetic acid extractable magnesium ranging from 0-3070 μg/g with very acid spoil being potentially deficient due to leaching under acid conditions, although there is no significant correlation between pH and levels in the spoil (Pulford 1976).

Sulphur is essential as a constituent of many plant proteins, legumes having a higher requirement than grass species. It is present in many rocks and minerals, the main source in colliery spoil being iron pyrites (FeS_2) which, on weathering, releases sulphate (SO_4^{2-}), the form in which sulphur is taken up by plants. Availability of sulphur in pyrite-bearing spoil (a characteristic of spoils of the West of Scotland) will therefore not be a factor limiting plant growth although non-pyritic spoil (such as those of Eastern Scotland) may show sulphur deficiency, particularly under waterlogged conditions which cause reduction of sulphate to sulphides, in which form it is toxic to root growth.

B. Micronutrient availability in colliery spoil (Fe,Al,Mn,Cu,Zn)

Levels of available trace elements in spoil affect plant growth in two ways; firstly by having a direct effect on plant growth, essential elements promoting growth at low levels and being potentially toxic at high concentrations (Hausenbuiller 1972), and secondly, by affecting the uptake of other nutrients required by the plant, e.g. high levels of available aluminium reducing phosphate uptake (Russell 1973, Brady 1974).

Table 1.5 lists the levels of iron, manganese, copper and zinc considered to be required by plants for normal growth and the concentration ranges which produce deficiency and toxicity symptoms, together with the levels found in herbage growing on colliery spoil (Chapter 2). The figures shown in columns 2-4 of Table 1.5 are broad generalisations and cannot be taken as absolute limits, particularly since variations between plant species and within each species in their ability to take up nutrients are not considered, nor is the fact that spoil factors such as the ratio of available levels of both essential and non-essential elements affects the plant's ability to absorb nutrients. Table 1.5 lists only those micronutrients chosen for further study in Chapter 2 in relation to plant uptake from spoil and these elements are discussed briefly in this section.

Table 1.5. Levels of iron, manganese, copper and zinc in plants grown on colliery spoil, compared to deficient, normal and toxic levels ($\mu g/g$ dry weight)

Element	Deficiency*	Normal*	Toxicity*	Reclaimed spoil**
Fe	10-80	30-150	None	217-2179
Mn	5-20	15-100	>1000	98-306
Cu	3-5	5-15	> 20	12-62
Zn	15	10-50	200-500	41-351

* Data summarised by Hausenbuiller (1972)

** See Chapter 2.

Iron

Iron occurs in igneous rocks as iron ores, micas and other ferromagnesian minerals. In colliery spoil it occurs mainly as iron pyrites (FeS_2) and is also found in clay minerals as a replacement ion for aluminium. Iron is essential for plant growth, being required for respiration and oxygen transport and for nitrogen fixation in legumes. Deficiency of iron causes chlorosis of the leaf and occurs mainly on calcareous soils in which iron may be present, but in an unavailable form.

Solubility of iron oxides is very low in the oxidised state and movement of iron is more common in the reduced Fe^{2+} state, the form in which most plant uptake occurs. Since iron occurs in more than one valence state, its availability is affected by the oxidation-reduction state of the soil.

Amorphous iron oxides, which are a product of pyrite oxidation in the spoil, affect plant growth indirectly by adsorption of phosphate, thus reducing its availability to plants (Pulford and Duncan 1975). Mitchell (1964) reported a normal range of 10-100 μg/g for acetic extractable iron in Scottish topsoil, while much higher levels are found in colliery spoil, Devlin (unpublished) finding levels of 5-742 μg/g and Kimber (1982) reporting levels up to 4,420 μg/g.

As Table 1.5 shows, the concentration of iron in plant tissues from the spoil field trial varies from 217-2179 μg/g, much higher than the "normal" plant content.

Aluminium

Aluminium is the most abundant metal in the earth's crust, occurring as bauxite and gibbsite. It is a structural component of clay minerals and also occurs as oxide and hydrous oxide coatings on clay mineral surfaces, these having great influence on spoil properties.

Aluminium is not essential for plant growth, but high aluminium ion level is one of the main causes of plant failure in acid soils; aluminium in solution reacting with soluble phosphate thus reducing the amount of phosphate available to the plants. In acid soils the concentration of aluminium is much greater than the phosphate ion concentration, therefore the reaction favours the formation of the aluminium phosphate (Messing 1971, Russell 1973), while phosphate levels are further reduced through adsorption onto hydrous oxide surfaces. Once taken up by the plant, aluminium in the plant cells interferes with the phosphate metabolism. Berg and Vogel (1969) reported the toxicity of aluminium in plants grown on colliery spoil.

Aluminium released by weathering of spoil is mainly precipitated as hydrous oxides, Kimber (1982) finding far more aluminium in oxalate extracts of Scottish spoils than in acetic acid extracts. Devlin (unpublished) found levels of acetic acid extractable aluminium ranging from 6-427 μg/g, while the plant tissue content varied from 120-3026 μg/g (See Chapter 2).

Manganese

The average manganese content of the earth's crust is 1,000 μg/g(Knezek and Ellis 1980). Soils contain a number of minerals which contain manganese in combination with O_2, SiO_2 and CO_2, and it is also found as a substitute ion in ferromagnesium minerals. Plants require manganese as an enzyme activator, however, high concentrations are toxic, causing leaf chlorosis and stunted growth.

Manganese ions occur in the soil in two forms - Mn^{2+}, held mainly on cation exchange sites on clay and organic matter and Mn^{4+} as amorphous oxides (Kimber 1982). Availability to crops depends on the pH and oxidation-reduction potential of the soil; low pH and reducing conditions increasing the Mn^{2+} ion content of the soil solution, the form in which plant uptake occurs.

Kimber (1982) found a substantial amorphous manganese oxide fraction in colliery spoil as well as a high level of acetic acid-extractable manganese (up to 536 μg/g) which could lead to manganese toxicity, this also being reported by Cummins et al. (1965).

Mitchell (1964) reported acetic acid-extractable manganese levels in Scottish arable soils ranging from 5-100 μg/g, a similar range being found by Devlin (unpublished) in colliery spoil at 18-118 μg/g.

Table 1.5 shows that the levels of manganese in plants growing on colliery spoil are higher than the "normal" level, however, they do not exceed the level considered to be toxic to plants.

Copper

The average copper content of the earth's crust is 55 μg/g with the average in British surface soils being 20 μg/g (Swaine and Mitchell 1960). Copper is essential for plant growth, being required for enzyme complexes involved in photosynthesis. It occurs in minerals in combination with S, O, CO_3 and SiO_4 and is found in association with ferromagnesian minerals. Copper is strongly affected by chelation, > 98% of copper in solution being in an organic complex form, bound by carboxyl groups as $[Cu(OH)]^+$ and phenolic groups as Cu^{2+} (Hodgson et al. 1966, Russell 1973). Two inorganic ionic forms exist in solution, Cu^+ and Cu^{2+}, the reduced state being more soluble at the pH values commonly found in soils.

Mitchell (1964) reported the acetic acid-extractable copper level in Scottish arable soils ranging from < 0.05-1.0 µg/g, while Devlin (unpublished) found colliery spoil to contain 1-42 µg/g.

Chapter 2 shows that in most cases, plants grown on colliery spoil contained copper levels in excess of the "normal" value, and in most instances exceeds the level regarded as being toxic to growth (12-62 µg/g).

Zinc

The average zinc content of the earth's cryst is 80 µg/g and that of soils ranging from 10-300 µg/g (Swaine and Mitchell 1960). Zinc is present in the soil as sulphides, oxides and silicates, and exists in the soil solution as Zn^{2+} and $[Zn(OH)]^+$, held on the clay and hydrous oxide surfaces. It is taken up by plants as Zn^{2+} ion, being required for growth hormones and the promotion of protein synthesis.

Hodgson et al. (1966) found that 60% of the zinc in arable soils existed in an organic complex form, similar results being reported by Russell (1973) who found that EDTA extracted more zinc than acetic acid.

Mitchell (1964) found levels of acetic acid-extractable zinc in Scottish arable soils ranging from < 2-30 µg/g, while Devlin (unpublished) found levels of 1-16 µg/g in reclaimed colliery spoil.

Table 1.5 shows that the levels of zinc found in plants growing on spoil ranged from 41-351 µg/g, which is greater than the "normal" plant content but below the level considered to be toxic to plant growth.

From these results it would appear that the levels of zinc in the spoil and vegetation are unlikely to seriously affect plant growth.

1.6.4.3 Summary

The nitrogen budget of colliery spoil is low due to lack of mineralisable organic matter, although legumes may naturally colonise the spoil and some fixation will occur (Bradshaw and Chadwick 1980), likewise phosphorus levels require to be raised by fertilization (Pulford 1976) although losses are substantial due to fixation by spoil. Potassium is not commonly a limiting factor due to release from clay mineral breakdown. Calcium and magnesium levels vary widely (Kimber 1982) in unamended spoil, but liming will ensure adequate supplies to plants, while sulphur is abundant due to release of sulphate on oxidation of iron pyrites.

Levels of trace elements, both essential (iron, manganese, copper and zinc discussed here and highlighted in Chapter 2) and non-essential (aluminium) are also limiting to plant growth, but in contrast to the macronutrients the problems encountered are of toxicity due to high levels occurring in spoil. Trace element availability and their uptake by herbage are discussed in Chapter 2.

1.7 Colliery Spoil Reclamation Techniques

The practical aspects of bing re-profiling are outwith the scope of this thesis, although the importance of considering the nature of the spoil during earthmoving is discussed. Reclamation is normally conducted on the basis of improving the chemical status of the spoil, with a soil cover being utilized as a means of improving growth conditions only on a limited scale, due to lack of availability. The situation regarding the use of a soil cover, and the advantages gained, are discussed in Chapter 5 based on the study of reclaimed sites under both schemes.

The normal sequence of operations involved in spoil reclamation without the use of soil are:

(a) Regrade the bing
(b) Apply lime
(c) Apply fertilizer
(d) Seed the amended spoil

1.7.1 Regrading

Regrading of coal bings involves the movement of thousands of tonnes of material in order to create a new land-form. Fig. 1.2 shows two cross-sections of Enterkine bing (See Appendix II) with the original and reclaimed surface shapes. Through experience gained by many visits to reclamation schemes during the course of this study, and by discussion with engineers and others involved in the earthmoving stages of spoil reclamation, it was found that most regrading projects involve the removal of "mounds into hollows" as shown in Fig. 1.2(a).

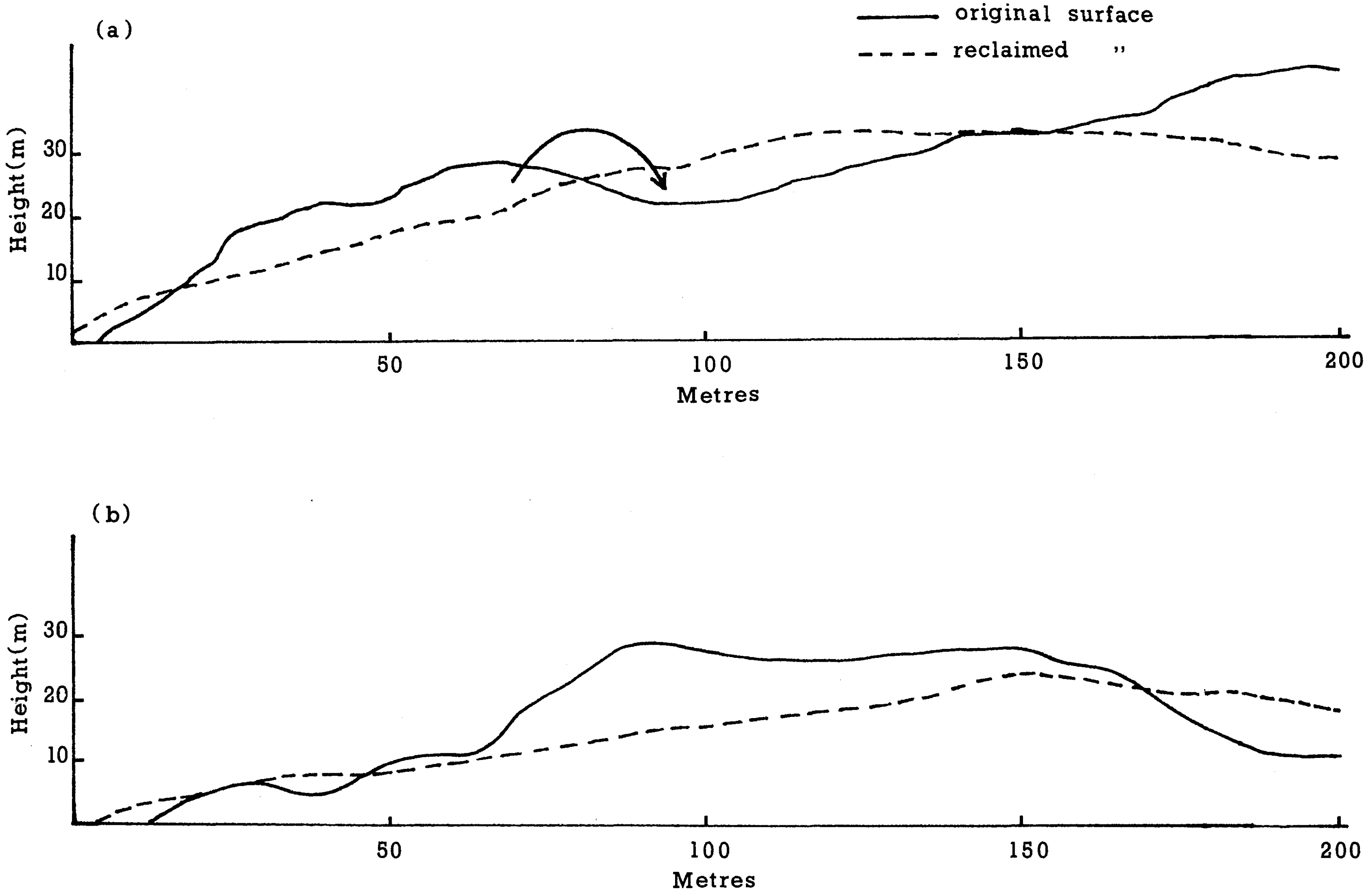


Figure 1.2. Cross-sections of Enterkine bing showing the original landform and reclaimed topography.

In most cases no consideration is given to the physical or chemical properties of the material at the regrading stage and sampling is only carried out after earthmoving, to determine the ameliorative treatments required before a vegetation cover may be planted. However, by following this procedure there are many drawbacks. Weathered surface spoil which has undergone chemical and physical changes, and which may even support a natural vegetation cover, will be buried, exposing unweathered spoil which, although it may permit plant growth initially, will break down and be altered, principally through the oxidation of iron pyrites resulting in plant failure (Doubleday 1973). The importance of extensive sampling after regrading has been stressed by many workers (Kimber et al. 1978, Kent 1982). However, a preliminary sampling program before earthmoving would ensure that unsuitable material would be identified and buried at regrading, although it would still be necessary to sample the new surface before planting. In this way plant survival would be increased and the need for re-cultivation due to spoil regression would be reduced.

"Clayey" spoils are not easily regraded and tend to become compacted by heavy vehicles, leaving a poor structure with high bulk density and low porosity which may waterlog and give high run-off rates during heavy rainfall. On drying, this type of material forms a massive structure which allows little gaseous exchange through the spoil, thus discouraging root growth, and preventing the breakthrough of young shoots due to the formation of a hard cap. Donovan et al. (1976) recommends avoiding the movement of material on the surface which has a clay content > 15% due to the risk of compaction. Therefore identifying spoil which is unsuitable for surface spreading before regrading is carried out would allow for such material to be buried on site, thus giving a greater chance of plant survival.

1.7.2 Liming

Since most Scottish spoils are acidic, or become acidic on weathering, liming is essential for plant establishment. Lime requirement is calculated by equilibration of spoil with calcium hydroxide solutions (Chapter 2), to find the amount of liming material required to raise the spoil pH to 6.2 (the optimum for pasture growth). Incorporation of lime to a depth of approximately 30 cm. using a disc cultivator provides a suitable seed bed and retains added fertilizer in an available form. Liming encourages plant root growth particularly in leguminous species, which do not thrive under acid conditions. Deeper rooting improves spoil structure through the addition or organic matter, encouraging granulation of loose-textured spoils and breaking up heavy spoils. The chemical effects of liming include raising spoil pH, reducing the levels of trace metals available for plant uptake, increasing the availability of added phosphate and increasing the amount of nitrogen fixed by legumes.

In addition to the quantity of lime required to raise spoil pH, account must be taken of the potential acidity of the material due to pyrite oxidation, and assuming complete oxidation of the pyrite, a further 40 tonnes of $CaCO_3$/ha should be added to neutralise each 1% of FeS_2 in the spoil (Costigan et al. 1981). Following this procedure liming rates of up to 50 tonnes/ha are not uncommon in pyritic spoil (Bradshaw and Chadwick 1980).

Addition of lime with phosphate fertilizer to acid spoil increases plant yield, although higher liming rates reduce the effectiveness of the fertilizer (Chapter 3). Therefore, the addition of extra lime actually reduces the potential phosphate supply of the spoil. Inadequate liming at planting may result in acid regeneration, causing die-back of the plant cover, and it has been found that surface dressings of lime to re-establish spoil pH at depth are ineffective due to the slow rate of percolation (Brown et al. 1956, Bloomfield et al. 1982), the most effective method being to plough in any vegetation cover with the incorporation of lime, thereby gaining the additional effect of adding organic matter. Due to the heterogeneous nature of spoil it is not uncommon to find areas on reclaimed sites which require re-liming, since liming rates must be calculated based on the average condition of the spoil at the time of sampling, therefore areas of highly pyritic material may be insufficiently limed and such areas will require recultivation.

1.7.3 Fertilizer addition

Fertilizer addition is usually as a single dose of a compound inorganic fertilizer, in most cases 20:10:10 or 20:15:15, N:P:K ratio, with in some instances, additional phosphate being applied as superphosphate or basic slag (see Appendix II).

Addition of sufficient fertilizer at planting to promote growth is not in itself a solution to the problem of low nutrient status of spoil. Bradshaw et al. (1973) found that the addition of 20:15:15 at a rate of 626 kg/ha was sufficient to establish a plant cover on a number of sites, but in order to maintain growth, additions of a further 376 kg/ha in the second year and even the third year were necessary, similar findings being reported by Bloomfield (1982). Nitrogen loss occurs mainly through leaching, and phosphorus is made unavailable by fixation through reaction with the spoil (see previous sections), thus, it is necessary to carry out a continuous system of monitoring of the nutrient status of the spoil in order to replace losses.

Due to the increasing cost of inorganic fertilizers in recent years, alternative forms of fertilizer have been used in order to reduce the cost of spoil reclamation. Two cheap organic fertilizers which are readily available, and which have the added advantage over inorganic forms of improving spoil structure due to their organic content, are poultry manure and sewage sludge.

(I) Poultry manure

Broiler litter is richer in nitrogen than most other bulky organic manures and for this reason is particularly useful as a colliery spoil amendment. It is usually applied mixed with bedding litter such as straw or wood shavings, giving a bulky, friable product which is relatively dry and can be spread easily with a dung spreader. This supplies available nitrogen immediately as well as releasing more nitrogen on breakdown. Bradshaw et al. (1973) found that the addition of poultry manure at a rate of 12.5 tonnes/ha to colliery spoil in S. Wales was an effective means of reducing the cost of fertilizer additions, a typical analysis being 2.3% N, 0.9% P, 1.6% K, 68% organic matter (Bradshaw and Chadwick 1980).

(II) Sewage sludge

Quality and analysis of digested sewage sludge cake vary greatly depending on the source, e.g. sludge from industrial areas may have high levels of trace metals. Most soluble nutrients are lost during drying giving a slow-release nitrogen and phosphorus fertilizer with a dry solid content of about 25%, containing 3% nitrogen and 3.5% phosphorus (W.R.C. Publication). Stucky et al. (1980) found that digested cake, when used on acid spoil, improved the soil pH, provided a source of nutrients and improved the physical condition of the spoil.

1.7.4 Seeding

Seeding is usually done using conventional broadcast methods, with hydroseeding only being used on inaccessible slopes. Seeding rate varies according to the species and the site, the rates used on the sites described in Appendix II being 49-110 kg/ha, and the mixture varying from region to region.

As discussed in previous sections, the inclusion of one or more leguminous species in the seed mixture is desirable as a means of spoil improvement and as a method of maintaining a nitrogen supply for the main grass component of the sward, thus reducing the requirement for nitrogen fertilizer additions. It is therefore necessary to use legume seed which has been inoculated with the nitrogen-fixing bacteria required by them to carry out their function.

The grasses and legumes most commonly used in seed mixtures are shown in Table 1.6 (Kent 1982), although many other species not listed may be included.

Table 1.6. Grass and legume species most commonly used in spoil reclamation (Kent 1982)

English Name	Latin Name
Common bent	*Agrostis tenuis*
Crown vetch	*Coronilla varia*
Cocksfoot	*Dactylis glomerata*
Meadow fescue	*Festuca pratensis*
Creeping red fescue	*Festuca rubra*
Chewings fescue	*Festuca rubra commutata*
Italian ryegrass	*Lolium multiflorum*
Perennial ryegrass	*Lolium perenne*
Small-leaved timothy	*Phleum bertolonii*
Timothy	*Phleum pratense*
Smooth-stalked meadow grass	*Poa pratensis*
Black medock	*Medicago lupulina*
Birdsfoot trefoil	*Lotus corniculatus*
Alsike clover	*Trifolium hybridum*
Red clover	*Trifolium pratense*
White clover	*Trifolium repens*

Appendix II lists the seed mixtures used on the 13 sites surveyed in Chapter 5, representing reclamation schemes carried out over the period 1965-1978 in Central Scotland, and this shows that, while varying the rates of application of lime and fertilizer depending on spoil properties, each regional body maintains more or less the same seed mixture on all of its sites.

The species used on these sites are listed below.

Perennial ryegrass (*Lolium perenne*)

This grass is often the main constituent of the seed mixture and was used on all of the sites studied, accounting for 11-45% of the mix. It is a hardy perennial species commonly found on wasteland and is used widely with clover for pastures, having quick emergence and giving a leafy growth providing grazing over a long season.

Red fescue (*Festuca rubra*)

This is a perennial grass which is widespread and grows abundantly on soils of low fertility, e.g. sand dunes, salt marshes. It spreads by means of creeping rhizomes which makes it particularly useful on spoil as a method of stabilizing the surface, especially on slopes.

Timothy (*Phleum pratense*)

A very hardy perennial grass used extensively for grazing and hay production, commonly found naturally on waste sites. It has a shallow rooting system, grows well on a moist, heavy soil and can tolerate low temperatures.

Meadow fescue (*Festuca pratensis*)

A tufted perennial grass found abundantly in meadows and which grows well on heavy, moist soils.

Brown top (*Agrostis stolonifera*)

A perennial grass commonly found on waste ground, which grows on a wide range of soil types from sandy to clayey, and survives well on poor, dry, acid soils. It spreads by means of rhizomes, and although of relatively low agricultural value it can be used to add organic matter and bind spoil surfaces.

Common bent (*Agrostis tenuis*)

A perennial grass which grows abundantly on a wide range of soil types. It spreads by means of short rhizomes, which bind and stabilize the surface material.

Rough-stalked meadow grass (*Poa trivialis*)

This is a perennial grass which spreads by creeping, leafy stolons (prostrate or creeping stems with roots growing at the nodes, giving rise to vegetative shoots). It thrives on rich, moist soil, has a vigorous growth and provides good ground cover.

Smooth-stalked meadow grass (*Poa pratensis*)

A perennial grass with creeping rhizomes, often found on wasteland. It is an important pasture species which grows well on well-drained, sandy loam soils and is used widely as a surface stabilizer on banks and roadsides.

Cocksfoot (*Dactylis glomerata*)

This is a densely tufted, coarse, perennial grass which grows well in rough grassland and meadows. It is deep rooted and can withstand drought conditions.

Crested dogtail (*Cynosurus cristatus*)

A perennial grass which survives on a wide range of soils, light or heavy texture, acid or alkaline reaction. It is a low growing grass with a leafy base, suitable for sheep grazing, can withstand drought and cold conditions, and is often used in mixtures for upland pasture.

Red clover (*Trifolium pratense*)

A perennial legume used extensively for pasture, hay production and green manure. It is found naturally on a range of soils and is abundant in meadows and forests. Although it grows best on well-drained loam soils it adapts to wetter conditions and is most productive at pH 6.6-7.6.

White clover (*Trifolium repens*)

A long-lived perennial legume which is shallow rooted and spreads by stolons which root at the nodes. It tolerates poor conditions better than most clovers and will grow on slight- to medium-acid soil, although it grows best at pH 6-7 with optimal nitrogen fixation at pH 6.5.

1.8 The Use of a Soil Cover in Spoil Reclamation

Spoil consists of unweathered rock and coal fragments which, given time (the length of which depends on the properties of the material) will weather and support a natural vegetation cover (Richardson 1976). However, as discussed in previous sections, the establishment of a plant cover has many factors acting against it, e.g. low pH and the problem of potential acidity, low nutrient status, high levels of trace metals, high surface temperatures, high bulk density, low water holding capacity, and these problems must be overcome, or at least corrected to an acceptable level before plants will grow successfully.

An alternative means of establishing a ground cover, rather than treating the spoil as a low grade soil requiring amendment, is to spread a layer of soil on the surface and thus reduce the influence of the spoil material on the plants. A soil layer provides a better seed bed and removes the seed from the spoil, thereby increasing plant survival at the early stage, although problems may be encountered subsequently and regression may still occur - the question concerning the use of a soil cover is fully discussed in Chapter 5.

Generally, if soil is available it is used as a cover, spread as a 10-15 cm layer on top of compacted spoil, the most common source of soil being from fields around the bing which are used to accommodate the spoil at regrading, as importing soil on to a site can only be done at reasonable cost over short distances. Topsoil and subsoil are removed, spoil is spread on the stripped land and the soil replaced. However, the nature of the underlying material is not taken into account nor is the downgrading of possibly good quality topsoil as it is mixed with subsoil layers.

Power et al. (1981) found in field trials with sodic mine spoil that herbage yield increased as the thickness of the soil layer increased, maximum yield being attained with 20 cm of topsoil over 70 cm of subsoil, this soil thickness removing the influence of the spoil from the vegetation cover, a similar soil layer thickness being recommended by Donovan et al. (1976).

Arnold (1981), using a maximum topsoil thickness of 25 cm on colliery spoil, also found yield to increase with soil depth, but concluded that the quality of the soil was of far greater importance than the quantity used.

Improved growth using a soil cover may be attributed to a number of factors including a greater capacity of the soil to retain moisture, ease of penetration of roots, and the soil used generally has a higher pH than the spoil, all of these factors improving conditions for plant growth. The common practice of utilizing soil at a depth of 10-15 cm does not provide a permanent solution to the problem, as spoil influences will still occur at depth. However, this is an acceptable depth, both from an economic point of view, and also is adequate in aiding plant establishment (Doubleday 1973).

Reclamation of colliery spoil for agricultural use involves high inputs of fertilizer to maximise yield and the discussions and recommendations in the present work pertain to this end. However, there are situations which do not demand such intensive reclamation. In some cases it is sufficient simply to regrade and "green-over" a site to remove the dominating presence of a bing from the landscape while some sites are reclaimed as parkland in urban green belt areas. In these situations a cover is sought which requires low maintenance and low inputs, with a predominance of slower growing, hardier species such as fine-leaved sheeps fescue (*Festuca tenuifolia*), hard fescue (*Festuca longifolia*), brown top (*Agrostis stolonifera*), velvet bent (*Agrostis canina*) and common bent (*Agrostis tenuis*) which form low growing swards requiring relatively little cutting.

1.9 Aims of the Thesis

As described in previous sections, coal waste disposal is a world-wide problem, requiring the expertise of soil analysts, among others, to integrate it back into the environment. Many of the physical and chemical problems found in poor soils are brought together in coal waste giving a hostile plant environment which can, however, be reclaimed successfully provided that enough information is obtained from the material before reclamation. Regrading, addition of lime and fertilizers, and planting a mixture of grasses and legumes does not guarantee success, as many factors in the spoil make it unsuitable for plant growth.

The two nutrients most commonly lacking in colliery spoil are nitrogen and phosphorus, which must be added at higher rates than are required in a soil for plants to grow. Nitrogen addition can be increased by the inclusion of inoculated legumes in the seed mixture. However, no such biological *in situ* addition is available for phosphorus. Both the low solubility of inorganic phosphorus fertilizers, and the ability of spoil to fix soluble phosphates result in low levels of phosphorus being available to plants, and this aspect of spoil chemistry has been studied by many workers.

The work described in this thesis was carried out to assess aspects of spoil reclamation from the point of view of its chemical suitability for plant growth. The problem of low phosphorus supply and phosphate fixing capacity, the cation exchange capacity of the spoil and therefore its capacity to hold plant nutrients, and the problem of high levels of available trace metals in reclaimed spoil are examined, together with an assessment through field studies of the successes achieved using a soil cover.

The results of these studies emphasise the need to assess each spoil site in some detail, both before and after regrading, and to continually monitor the progress of vegetation growth, as it is impossible to draw up a plan of work which can be used on all sites, the main characteristic of coal bings being that they are heterogeneous mixtures of materials, differing one from another both physically and chemically.

CHAPTER 2

TRACE ELEMENT CONTENT OF PLANTS GROWING ON RECLAIMED COLLIERY SPOIL

2.1 INTRODUCTION

Plants probably contain traces of all the elements present in the environment in which they grow, however, the mere fact of uptake does not imply that an element is essential for the plants' development, simply that it is present in the root zone in a form which the roots have no mechanism to exclude. Soil analysis to determine the trace element content, and the form in which each element occurs, will indicate the liklihood of deficiency or toxicity in crops grown on that soil, but the analysis of the sward will give no clue to the levels or forms of these elements in the soil (Cornwell and Stone 1973, Dick et al. 1985). Uptake of a particular element by a plant depends not only on the level of the active form of that element in the soil but also on the availability of many other elements, both essential and non-essential for growth (Carter et al. 1969, Jones et al. 1973, Bjerre and Schierup 1985, Culvenor 1985).

2.2 Effect of Soil Conditions on Trace Element Availability

I. Soil acidity

One of the main effects of soil acidity on plant growth is an increase in the availability of trace elements and a deficiency of Ca, Mg, Mo and available P (Hallsworth et al. 1957, Kimber 1982). A decrease in soil pH may bring about either or both of these changes, as well as having a direct effect on the growth of the plant roots and soil micro-organisms. Murphy et al. (1984) found that infection of root hairs by nitrogen-fixing bacteria was susceptible to high aluminium concentration in the soil, therefore modulation subsequent to infection of legume roots was reduced.

II. Moisture content

Mitchell et al. (1957) found that in the Scottish soils in their study the most important pedological factor influencing trace element availability was the drainage status of the soil, with higher levels of Co, Ni, V, Cu, Mo, Fe, Ti and Cr extracted from a poorly drained site compared with an adjacent well-drained site. They also found that the differences in levels of extractable Co and Ni in the soil were reflected in the content of these elements in the vegetation, while such a relationship did not exist for Fe, V, Cr or Ti.

III. Organic matter

Due to the association of some trace elements, notably Cu and Zn, with the organic fraction in the soil (Robson and Reuter 1981), the level of organic matter affects their availability. Bjerre and Schierup (1985) found levels of Cu and Zn in organic soils exceeding those of sandy soils, although plant uptake was lower from the organic soil due to the association with the insoluble organic fraction.

IV. Cation exchange capacity

Adsorption of trace elements on the cation exchange complex removes them from the available to the exchangeable pool, and cannot be taken up by plants unless an exchange reaction first occurs, e.g. replacement by H^+ from root exudiates. Hinesly et al. (1982) found that plant uptake of cadmium, added to soil as a soluble salt, was reduced in soils with higher cation exchange capacity.

2.3 Plant Adaptation to Adverse Soil Conditions

Certain plant species are able to tolerate a high level of a particular trace element present in the soil and can accumulate high concentrations of that element in their tissues (Smith 1971). Such accumulator species must be identified to protect grazing animals from trace element toxicity, e.g. seleniferous species (Allaway 1968), while they may be useful for mapping ore deposites (Bollard and Butler 1966, Joyce 1975).

Plant species can adapt to the prevailing soil conditions such as high or low pH, high salinity or contamination by heavy metals (Gregory and Bradshaw 1965). Calcifuge plants (those which grow under acid soil conditions) can tolerate higher levels of available Aℓ than those growing under alkaline conditions (calcicole) which show signs of Aℓ toxicity and Ca deficiency under acid conditions (Russell 1973).

2.4 Variation Between Plant Species

Plant species vary widely in their trace element requirement and also in their trace element content (Allaway and Carey 1964). Total mineral content of a sward is important from the point of view of grazing animals and many studies have been carried out on grasses and legumes to determine the intake by animals, mainly in relation to trace elements which are toxic at high levels (Allaway <u>et</u> <u>al</u>. 1966, Wilkins 1979). Trace element intake is determined primarily by the availability of the soil trace elements and also by the botanical composition of the sward, but is modified significantly by the season and stage of maturity of the plants (Reay and Marsh 1976, Thompson and Warren 1979, Merry and Tiller 1986). In general, for the constituents of a mixed sward, the relative content of whole plant samples remains reasonably constant, e.g. clovers generally have higher N, Ca and trace element contents than grasses (Hemingway 1962, Jarvis 1980). Differences with stage of maturity will affect the level of each mineral, but not enough to change the order between species (De Medeiros and Haridasan 1985).

2.5 Distribution of Trace Elements Within Pasture Plants

Trace element distribution within stems and leaves of pasture plants is uneven and the distribution pattern varies between species (Fleming 1963, Fleming and Murphy 1968, MacLean 1976). Fleming (1963) found a higher concentration of Mg in the leaves of four grasses and one clover species, a higher concentration of Mn in the stems of the grasses and in the leaves of the clover and a concentration of Ni in the seed heads of all species, these latter two observations being confirmed by Davey and Mitchell (1968).

Plant yield on reclaimed spoil is lower, when compared to a soil, due to many factors discussed in Chapter 5, hence the preference to use a soil cover over treatment of the bare spoil. Whole plant analysis shows that high levels of trace elements may occur in plants growing on mine spoil (Gregory and Bradshaw 1965, Stucky *et al*. 1980) and yield is increased by the use of a soil cover (Arnold 1981, Power *et al*. 1981), however this does not completely remove the plant from the influence of the underlying spoil. A comparison of the trace element content of perennial ryegrass growing on a soil and a reclaimed spoil was made to determine the differences in levels of uptake and the distribution within the plant of the elements studied.

2.6 Trace Element Content of Plant Parts

Plate 3 shows two samples of hay at the same stage of maturity and grown on adjacent land under the same management scheme. The "arable" sample was grown on a sandy clay loam soil and the "reclaimed" sample was grown on reclaimed spoil with a cover of 10-15 cm of a similar soil. Both samples were taken from Lochore Meadows Phase IV, Hilton of Beath Farm (see Appendix II).

In order to make a direct comparison of the trace element content of the two hay samples, only the perennial ryegrass (*Lolium perenne*) in each sample was analysed. Each plant was separated into four parts for analysis (Fig. 2.1):

1. Seed head
2. Upper stalk to which the seed is attached
3. Main stalk
4. Leaf

PLATE 3 - Samples of hay grown on "arable" and "reclaimed" land.

Fig. 2.1. Division of perennial ryegrass (*Lolium perenne*) for trace element analysis

2.6.1 Ashing Procedure

All samples were dried at 105°C in a force draught oven and ashed using a nitric acid (HNO_3)/perchloric acid ($HC\ell O_4$) procedure based on research carried out in the Agricultural Chemistry Department, Glasgow University, showing the suitability of this method for herbage analysis (Khalil 1981).

Method

Approximately 1g of sample was weighed into a 500 ml conical flask to which 20 ml of Aristar conc. HNO_3 was added, and the flask placed on a sand bath at 120-140°C with a filter funnel in the neck of the flask to reflux the acid. Dense brown fumes appeared during organic matter oxidation and digestion was continued until fuming ceased (approx. 1 hr). After cooling, 5 ml of Aristar conc. $HC\ell O_4$ was added and the flask returned to the sand bath at 200°C for approx. 2 hrs. On completion of the digestion dense white fumes of $HC\ell O_4$ appeared and the digest was clear with a residue of silica remaining. The digest was cooled and the funnel and inside of the flask were rinsed with de-ionized water, filtered to remove the silica residue and the volume made to 100 ml.

All analyses were carried out in duplicate, together with a blank digestion containing the acid mixture only. All steps were carried out in a fume cupboard.

Five elements which occur in colliery spoil at higher levels than are found in arable soils were chosen for this study, namely Fe, Aℓ, Mn, Cu and Zn, each determined by atomic absorption spectrophotometry using a Perkin Elmer model 370A.

2.7 RESULTS

Table 2.1 shows the levels of iron, aluminium, manganese, copper and zinc found in each plant part. (See also Figs. 2.2-2.6).

Table 2.1. Levels of Fe, Aℓ, Mn, Cu and Zn in "arable" and "reclaimed" samples of perennial ryegrass (*Lolium perenne*)–range and mean.

PLANT PART		Total metal content (μg/g dry matter)				
		Fe	Aℓ	Mn	Cu	Zn
SEED HEAD	A*	66(39) (60-72)	50 (45-55)	39(18) (36-42)	21(5) (20-22)	42(37) (39-45)
	R**	103 (95-111)	110 (100-120)	56 (52-60)	16 (13-19)	37 (34-40)
LEAF	A	113(101) (106-120)	170 (160-180)	68(41) (61-75)	19(5) (18-20)	30(20) (29-31)
	R	141 (131-151)	170 (158-182)	93 (84-102)	21 (19-23)	43 (40-46)
MAIN STALK	A	40(22) (36-44)	370 (350-390)	58(56) (53-63)	27(4) (22-32)	28(13) (26-30)
	R	52 (48-56)	440 (319-361)	58 (51-65)	24 (21-27)	44 (40-48)
UPPER STALK	A	70 (65-75)	70 (64-76)	25 (22-28)	17 (14-20)	28 (25-31)
	R	75 (71-79)	112 (104-120)	30 (27-33)	17 (15-19)	35 (31-39)

* A = "Arable" sample

** R = "Reclaimed" sample

Results in brackets are those of Fleming (1963) for perennial ryegrass grown on a well-drained acid brown earth of medium base status.

ANALYSIS OF VARIANCE SHOWS WHICH FACTOR I.E. MANAGEMENT REGIME OR PLANT PART HAS A SIGNIFICANT EFFECT ON PLANT CONTENT. SEE FIGS 2.2-2.6.

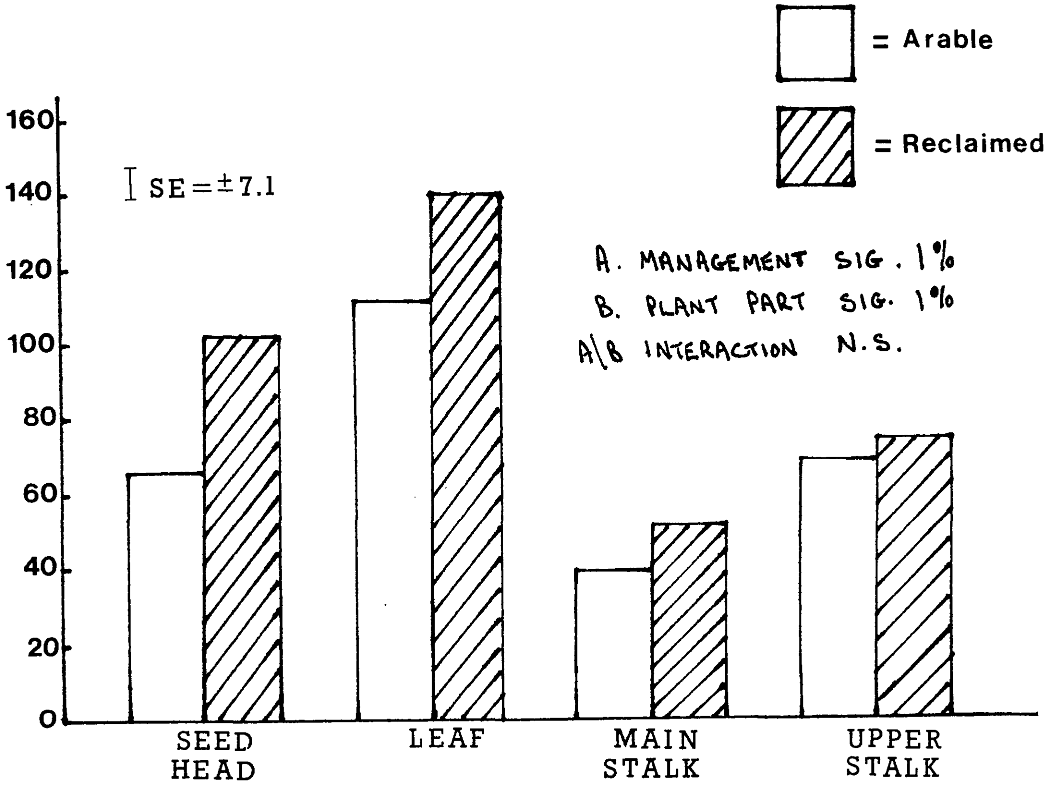


Fig. 2.2. Iron content of perennial ryegrass.

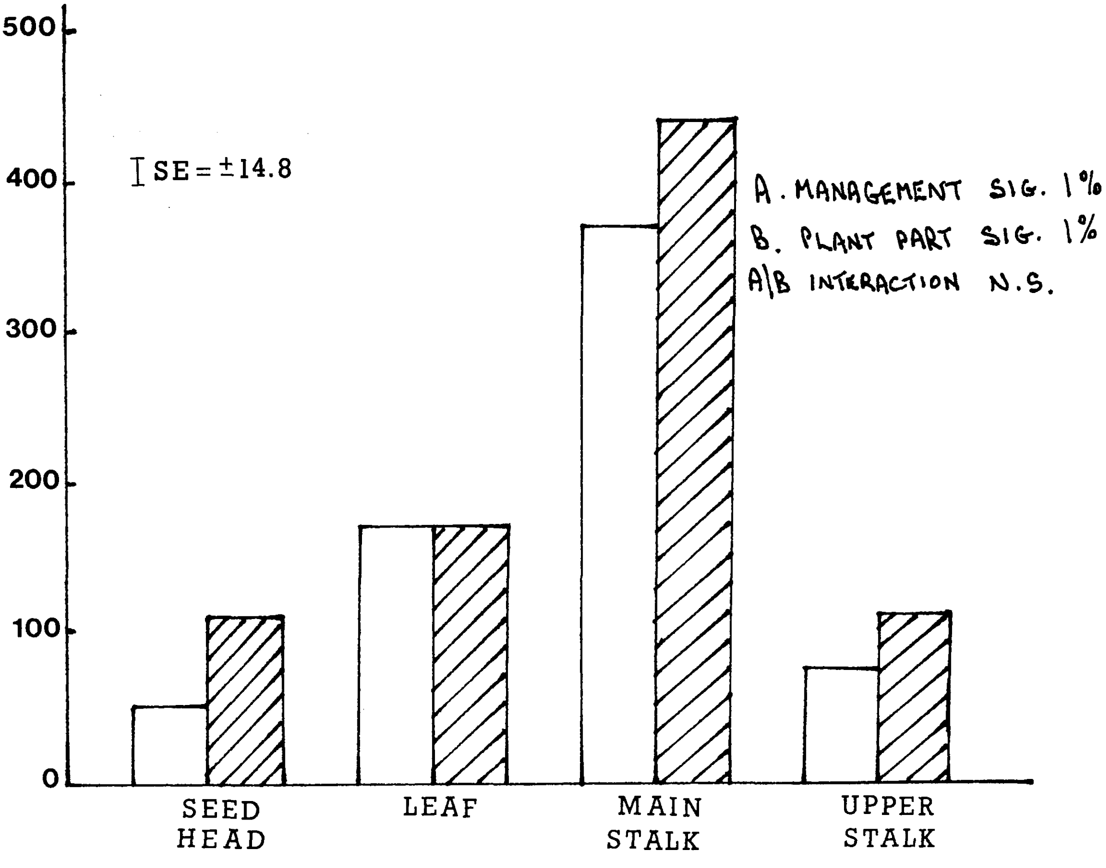


Fig. 2.3. Aluminium content of perennial ryegrass.

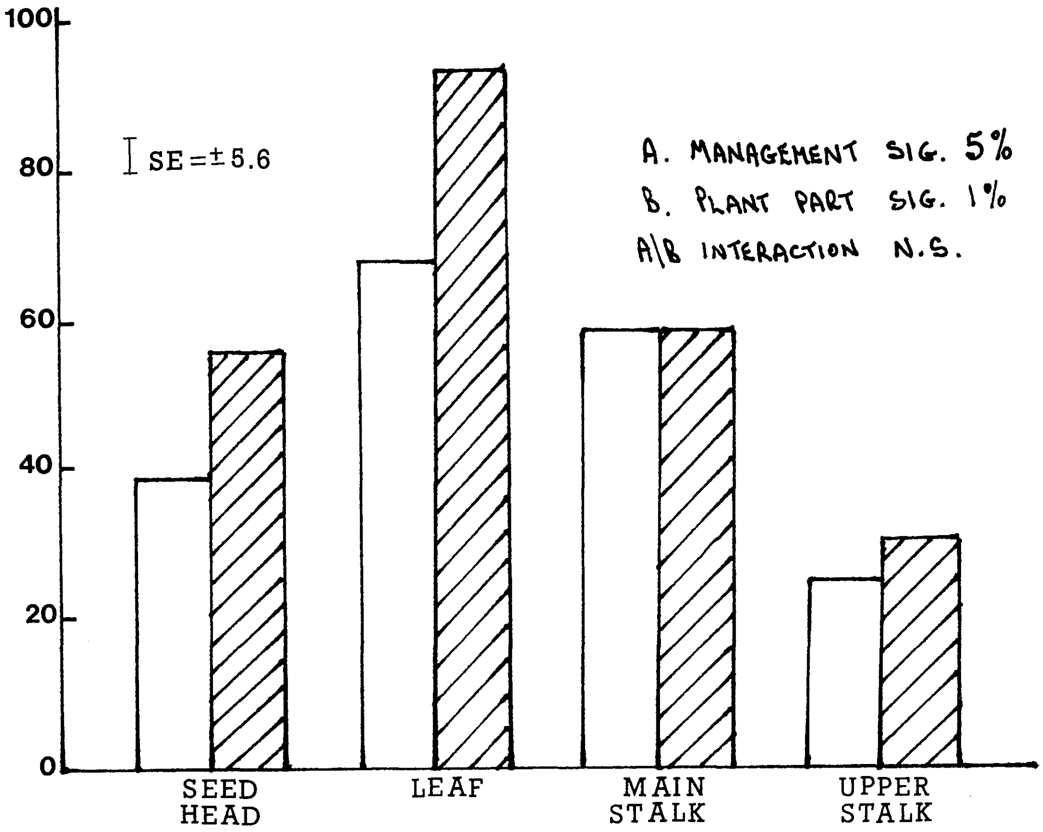


Fig. 2.4. Manganese content of perennial ryegrass.

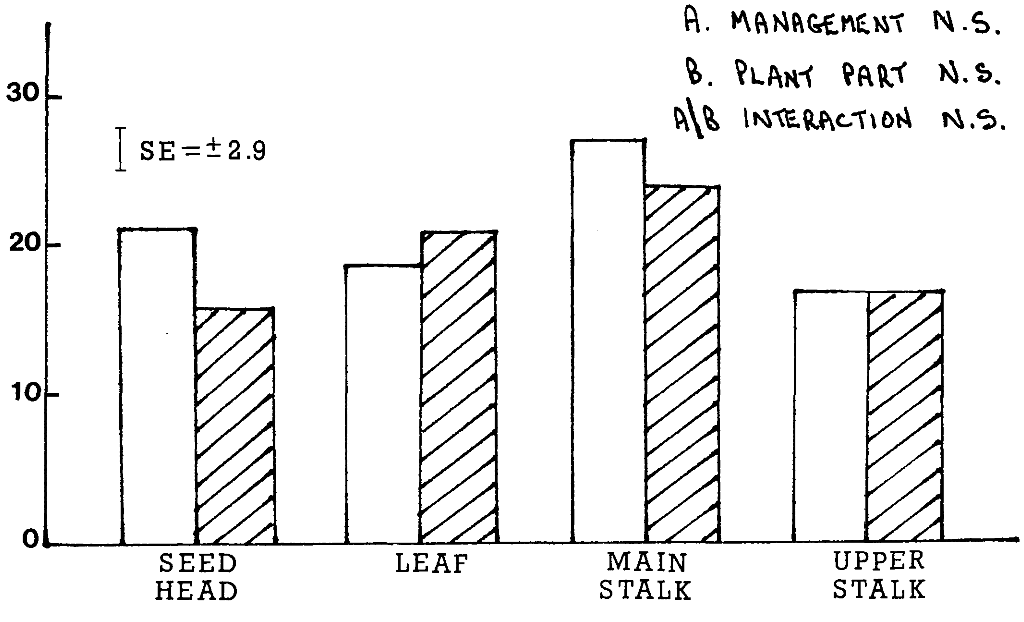


Fig. 2.5. Copper content of perennial ryegrass.

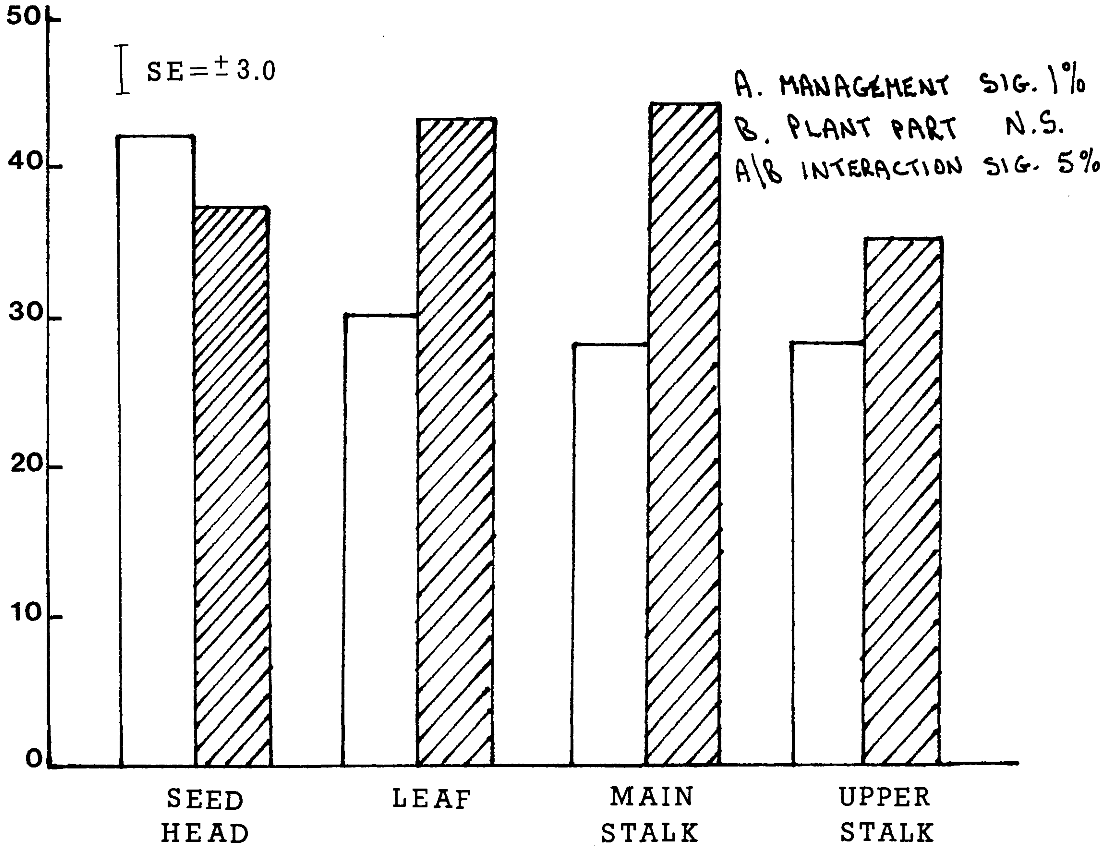


Fig. 2.6. Zinc content of perennial ryegrass.

As Table 2.1 shows, in most cases the level of each element is higher in both hay samples than the results of Fleming (1963), and in most cases, except for Cu, the "reclaimed" sample has a higher content in each plant part than the "arable" sample. The only two instances where the levels agree with Fleming (1963) are the concentrations of Mn in the main stalk and Zn in the seed head. This second observation is of particular note since Fleming (1963) found that for each of the four grass species studied that the level of Zn in the seed head was very similar; perennial ryegrass (Lolium perenne) 37 μg/g, meadow fescue (Festuca pratensis) 37 μg/g, cocksfoot (Dactylis glomerata) 35 μg/g and timothy (Phleum pratense) 36 μg/g, despite the variations in trace element content of other plant parts. Although the leaf and main stalk samples shown in Table 2.1 have higher Zn contents than those found by Fleming (1963), the seed head maintains a reasonably constant value. In a later study Fleming (1963-1965) again draws attention to this and suggests a possible association with tryptophan and auxin synthesis, an association also stated by Nason and McElroy (1963), who also found an accumulation of Mn in the leaf (see Table 2.1), and by Salami and Kenefick (1970). There may therefore be a mechanism in perennial ryegrass which regulates the accumulation of Zn in the seed head and Mn in the main stalk where these elements are required for a metabolic function which would cease if the levels exceeded a threshold value.

Despite variations between the samples in the levels of the five elements studied, the distribution pattern within the plants from both sites is similar in many cases - highest Fe and Mn content in the leaf with Aℓ and Cu accumulation in the main stalk.

2.8 DISCUSSION

The levels of Fe, Aℓ, Mn and Zn in perennial ryegrass (Lolium perenne) from the reclaimed site are higher than those in the arable sample in most plant parts.

When plant roots take up ions from the soil solution, there are mechanisms in the plant which regulate the amount which is transported to each site within the plant. Such a regulatory system serves two functions, firstly, elements which are required at a specific site are transported to that site which will be favoured during periods of short supply, and secondly, elements which disrupt any metabolic function at a particular site when present at a concentration exceeding a critical value can be directed to, and stored at, sites where they have no ill effect on the plant. One or both of these mechanisms may explain the occurrence of similar concentrations in both samples in certain plant parts. The level of Fe and Cu in the upper stalk, Aℓ in the leaf, Mn in the main stalk and Zn in the seed head are similar in both samples despite differences in the other plant parts. It may be that these levels represent the concentration required by the plant at that site, or that these are the maximum tolerable levels, further uptake being directed elsewhere in the plant.

2.9 The Effect of Addition of Lime and Organic Matter to Spoil on the Mineral Content of Herbage (Field Trial)

To investigate the effect of lime and organic matter addition on herbage mineral content, a field trial was set up in June 1980 on a regraded coal bing (Baads, Lothian Region) in conjunction with

Mr. J. Devlin, Agricultural Chemistry Department, Glasgow University.

The material consisted of unburnt shale and coal waste with material $>$ 150 m.m. removed. The average pH over the trial area was pH 2.6 and iron pyrites content 0.51%.

2.10 Materials and Methods

2.10.1 Lime requirement

5g samples of $<$ 2 m.m. spoil were weighed into six 100 ml glass-stoppered flasks. Additions of approximately 0.01N $Ca(OH)_2$ (standardised) and de-ionized water were made as shown in Table 2.2.

Table 2.2. Volume of 0.01N $Ca(OH)_2$ and de-ionized water added to flasks for lime requirement determination

	1	2	3	4	5	6
vol. 0.01N $Ca(OH)_2$ (ml)	0	10	20	30	40	50
vol. de-ionized water (ml)	50	40	30	20	10	0

Each flask was shaken on a wrist shaker for 30 mins, the pH of the suspension measured and a graph of pH against volume of 0.01N $Ca(OH)_2$ added was plotted. From the graph, the volume of $Ca(OH)_2$ required to raise the pH to 6.2 (suitable for a grass/ clover sward) was found and the lime requirement calculated, assuming that 1 milliequivalent of H^+ per 100g spoil requires 1.12 tonnes $CaCO_3$/ha plough layer. The liming material used was ground limestone with a neutralizing value of 46% (compared to 100% for CaO) giving a ground limestone requirement of 25 tonnes/ha.

Based on the assumptions that all of the iron pyrites in the spoil will eventually oxidise, and that it requires 4 tonnes ground limestone/ha to neutralise each 0.1% FeS_2, the lime requirement to include an allowance for this was calculated at approximately 50 tonnes/ha. Two liming rates were therefore applied and compared to unlimed plots.

2.10.2 Organic amendments

To determine the effect of organic matter incorporation in the spoil on herbage mineral content, the following materials were disced to 10 cm. Each amendment was added at three rates and compared to treatments with lime 25 & 50T|ha and fertilizer 300 kg|ha only.

(a) Peat

Peat was added at 5, 10 and 20 tonnes/ha

(b) Chicken manure

Chicken manure mixed with bedding litter (wood shavings) was added at 1, 2 and 4 tonnes/ha

(c) Sewage sludge

Dried sewage sludge cake was added at 5, 10 and 20 tonnes/ha

(d) Alginure

This is a commercial formulation supplied by Comtec (U.K.) Ltd., Kent, consisting of gypsum ($CaSO_4$), calcified seaweed and alginure (a seaweed hydrolysate) in a ratio of 10:1.5:1.5 added at 2.75, 5.5 and 11.0 tonnes/ha.

2.10.3 Fertilizer

The entire trial area received 300 kg/ha of a 15:10:10, N:P:K compound fertilizer.

2.10.4 Seed mixture

The seed mixture used was a grass/clover mix, at a rate of 120 kg/ha:

50% Perennial ryegrass (*Lolium perenne*)
25% Red fescue (*Festuca rubra*)
10% Cocksfoot (*Dactylis glomerata*)
9% Timothy (*Phleum pratense*)
6% White clover (*Trifolium repens*)

2.10.5 Number of plots

The trial consisted of three randomized blocks of 39 treatments. Each plot measured 8m x 3m, in triplicate, thus giving 117 plots.

Fertilizer and lime were incorporated by discing to 20 cm, followed after 7 days by the organic amendments which were lightly disced to 10 cm, with the seed being broadcast after a further 9 days.

2.10.6 Herbage sampling

Eleven weeks after sowing, samples were taken from each plot by placing two half-square metre frames at one metre from opposite corners of the plot. Samples were cut by hand leaving a 5 cm stubble, placed in sealed polythene bags and the yields recorded.

2.10.7 Sample preparation

All samples were washed with running tap water to remove adhering spoil material and rinsed with de-ionized water (Hargrove et al. 1985). A representative sample was taken from each of the duplicate harvests from each plot, dried at 105°C in a force draught oven and ashed following the procedure in Section 2.6.1.

The elements chosen for study were Fe, Aℓ, Mn, Cu, Zn, due to their toxicity to plants at high concentration and the presence of high levels in colliery spoil (MacLean and Dekker 1976, Pulford 1976, Kimber 1982), Ca and Mg to determine the levels of uptake and changes with liming. Each element was determined by atomic absorption spectrophotometry using a Perkin Elmer model 370A.

2.11 RESULTS AND DISCUSSION

Due to the heterogeneous nature of the spoil, many plots showed patchy growth, and only those which gave a yield in both half square metre sections were selected for herbage analysis, thus from a total of 117 treatment plots only 82 gave a recorded yield. Of the 39 treatments in this study, 22 gave a yield in each of the three replicate areas, 7 gave yields in only two areas and 2 treatments were successful in only one area, with the remaining 8 treatments giving no yield. Table 2.3 shows the treatments which gave no recorded yield.

Table 2.3. Treatments giving no recorded yield in any of the three replicate areas

Lime rate	Amendment	Amendment rate (tonnes/ha)
0	-	-
0	Peat	5, 10, 20
0	Chicken manure	1
0	Alginure	2.75
0	Sewage sludge	5, 20

As Table 2.3 shows, all of the unsuccessful treatments were unlimed, indicating that spoil acidity is a major factor liming plant growth (MacLean and Dekker 1976).

In order to determine whether the two samples taken from each plot were representative of the plot as a whole, the correlation between the levels of each element in the duplicate samples was determined (Table 2.4).

Table 2.5 shows the levels of Fe, Aℓ, Mn, Cu, Zn, Ca and Mg in the herbage from each treatment (82 plots) together with herbage yield and spoil pH. The levels of Fe, Aℓ, Mn, Cu and Zn in the spoil (0.5M acetic acid-extractable) are shown in Table 2.6. I am indebted to Mr. J. Devlin for his permission to use the spoil analysis data in this study.

Table 2.4. Correlation coefficient and significance of the relationship between the levels of each element in the duplicate samples from each plot

Element	Correlation coefficient (n = 82)
Fe	0.955^{***}
Aℓ	0.950^{***}
Mn	0.990^{***}
Cu	0.226^{*}
Zn	0.527^{***}
Ca	0.940^{***}
Mg	0.962^{***}

* Significant at 5% level

** Significant at 1% level

*** Significant at 0.1% level

Note. This statistical notation is retained throughout this thesis in relation to correlation analysis.

As the results in Tables 2.5 and 2.6 show spoil pH, trace element content, yield and herbage mineral content vary widely between replicate plots in many cases. This is due to a number of factors, including uneven application of lime and fertilizer and variability of spoil properties across the site. A further factor to consider regarding herbage content is the fact that these results represent an immature sward after only 11 weeks

Table 2.5. The following is a listing of treatments, spoil pH, herbage yield and mineral content of herbage from a field trial on Baads colliery spoil (mean of two samples)

CM = Chicken manure
ALG = Alginure
SS = Sewage sludge

Yield expressed as kg (fresh weight)/ha
Herbage mineral content expressed as µg/g (dry weight)

Treatment	Lime rate (tonne/ ha)	Amendment	Amendment rate (tonnes/ ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (µg/ g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
1	0	CM	2	3.2	15	936	1211	136	16	58	7022	1467
				3.6	258	298	320	112	32	75	3081	1180
2	0	CM	4	3.1	29	367	328	129	38	74	2815	1552
				2.4	32	395	458	125	29	142	3739	1591
				3.1	39	894	1913	98	28	152	2720	1425
3	0	ALG	5.5	2.7	20	713	1054	121	25	62	2566	1054
4	0	ALG	11.0	3.4	14	775	728	154	32	55	4357	1342
				3.7	11	589	589	137	17	63	4504	1258
5	0	SS	10	3.3	21	794	788	214	31	73	4767	1695
				3.0	24	785	681	306	26	351	4739	1733
6	25	0	0	3.9	65	505	381	301	26	68	4963	1584
				5.8	65	338	359	192	20	42	6294	998
				5.9	45	467	929	162	21	57	4772	1298

Table 2.5 (contd.)

Treatment	Lime rate (tonne/ha)	Amendment	Amendment rate (tonnes/ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (µg/g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
7	25	PEAT	5	6.5	29	322	334	153	28	74	5618	1569
				6.2	9	1279	1205	167	37	190	7756	1926
				6.3	58	1566	3026	153	23	53	5724	1299
8	25	PEAT	10	4.5	22	493	478	203	21	59	5637	1096
				6.5	45	312	280	179	14	53	5383	1029
				4.6	20	513	430	141	15	56	4831	1160
9	25	PEAT	20	4.5	27	400	398	226	30	60	5734	1494
				5.1	45	704	886	159	22	63	6573	1474
				6.5	42	649	573	166	18	56	7019	1530
10	25	CM	1	5.9	182	341	278	151	28	48	5549	1193
				6.2	46	704	886	159	22	63	5236	1059
				3.3	134	364	381	138	25	64	4862	1403
11	25	CM	2	6.3	250	354	267	138	37	70	5701	1414
				6.0	75	515	320	159	19	81	5932	1431
				6.2	147	464	379	164	19	57	6216	1556

Table 2.5 (contd.)

Treatment	Lime rate (tonne/ha)	Amendment	Amendment rate (tonnes/ ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (µg/g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
12	25	CM	4	6.0	320	295	170	106	40	71	4615	1425
				6.0	486	253	200	122	23	52	4589	1160
				4.8	450	286	200	180	40	113	4639	1563
13	25	ALG	2.75	5.1	14	375	450	157	30	70	5448	1498
				7.1	75	474	721	194	21	72	6499	1332
				6.5	24	733	689	261	15	78	6336	1327
14	25	ALG	5.5	5.7	9	1060	1100	179	50	89	5548	1067
				6.0	121	675	749	146	22	63	5201	1098
				5.1	85	846	1474	131	18	63	5280	1275
15	25	ALG	11.0	6.1	21	565	618	207	27	63	4918	1177
				5.4	22	843	866	157	21	47	5936	1076
				6.0	18	489	499	224	14	164	4919	1297
16	25	SS	5	5.9	8	464	499	148	22	55	5894	1376
				5.8	64	534	515	164	23	137	7764	1750

Table 2.5 (contd.)

Treatment	Lime rate (tonne/ha)	Amendment	Amendment rate (tonnes/ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (μg/g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
17	25	SS	10	3.2	64	620	537	176	27	58	6667	1353
				4.1	91	676	867	127	19	69	6749	1356
				6.6	113	356	419	145	19	39	5770	1158
18	25	SS	20	6.3	7	579	558	154	27	67	9097	2023
				6.6	320	696	674	143	46	72	7419	1533
				5.7	65	1006	2022	188	35	67	8490	1862
19	50	0	0	6.4	22	592	498	172	34	67	6837	1455
				6.0	27	384	420	195	12	74	4576	1199
				6.7	43	252	200	158	20	42	7087	1298
20	50	PEAT	5	5.6	58	694	840	142	16	62	7430	1360
				5.2	76	379	450	167	14	41	5901	1100
21	50	PEAT	10	6.1	29	481	487	199	33	66	7109	1221
				5.0	19	653	521	227	16	193	6007	1151
				6.0	18	518	571	227	17	68	6639	1352

Table 2.5 (contd.)

Treatment	Lime rate (tonne/ha)	Amendment	Amendment rate (tonnes/ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (μg/g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
22	50	PEAT	20	6.7	41	394	364	179	35	79	5999	1427
				4.8	28	576	680	168	17	62	7530	1560
				6.7	26	571	570	150	32	95	5804	1101
23	50	CM	1	7.0	132	2179	1119	178	19	67	5647	1379
				6.3	83	835	650	132	22	261	5496	1499
24	50	CM	2	6.4	340	217	166	150	34	69	5681	1401
25	50	CM	4	4.7	190	331	309	119	29	67	2874	1477
				4.6	350	559	592	110	62	204	6497	1693
				6.6	570	218	120	139	19	70	4448	1479
26	50	ALG	2.75	6.7	60	348	340	198	30	65	5605	1309
				5.7	73	478	509	214	12	36	5461	998
				6.4	20	626	691	190	25	77	5588	1332
27	50	ALG	5.5	6.2	47	546	698	188	25	48	6131	1097
				6.5	45	655	718	157	22	79	5105	1226
				6.8	58	419	393	185	35	71	5317	1470

Table 2.5 (contd.)

Treatment	Lime rate (tonne/ha)	Amendment	Amendment rate (tonnes/ha)	pH	Yield (fresh weight) (kg/ha)	Herbage content (μg/g D.M.)						
						Fe	Aℓ	Mn	Cu	Zn	Ca	Mg
28	50	ALG	11.0	6.3	29	538	520	175	28	60	5176	1079
				6.9	18	436	520	177	55	72	5097	959
				6.9	50	1065	720	130	28	54	5131	1100
29	50	SS	5	6.1	48	1240	1665	176	33	61	7208	1495
				5.8	35	557	689	237	17	254	4904	999
				6.7	67	388	380	128	30	82	5743	1279
30	50	SS	10	5.0	82	371	389	190	31	55	6101	1358
				3.9	55	555	538	182	18	177	6078	1196
31	50	SS	20	6.3	73	577	349	159	37	73	8076	1424
				6.3	150	310	239	167	30	69	5940	1595
				6.4	61	362	349	198	26	70	6716	1826

Table 2.6. Acetic acid-extractable Fe, Aℓ, Mn, Cu and Zn in spoil from Baads field trial (μg/ g dry weight)

Treatment	Acetic acid-extractable (μg/g)				
	Fe	Aℓ	Mn	Cu	Zn
1	54	227	28	2.1	8.1
	70	236	56	2.2	6.4
2	222	258	38	2.4	5.6
	308	211	34	1.5	9.8
	84	307	67	2.4	10.7
3	272	340	54	4.2	8.3
4	176	359	42	3.0	7.7
	100	254	28	0.9	4.1
5	200	427	40	4.2	8.9
	419	271	83	4.5	5.8
6	347	308	92	1.8	9.2
	222	149	64	3.3	3.9
	59	124	66	1.8	3.9
7	78	152	49	1.5	11.3
	54	227	28	2.1	3.8
	42	64	84	1.5	4.5
8	238	254	23	0.8	3.8
	132	37	85	2.0	2.7
	180	195	47	1.6	3.3
9	123	293	41	1.1	5.3
	51	77	42	0.8	3.9
	22	33	95	0.9	3.6
10	110	184	77	0.8	5.3
	121	90	74	1.3	4.2
	277	258	18	1.3	2.6

Table 2.6 (contd.)

Treatment	Acetic acid-extractable (μg/g)				
	Fe	Aℓ	Mn	Cu	Zn
11	220	49	66	1.0	6.5
	331	81	72	1.8	3.6
	96	112	77	0.6	7.9
12	154	75	71	0.7	2.7
	18	31	87	1.7	15.8
	107	344	93	1.5	9.3
13	71	142	34	2.1	12.7
	9	6	109	1.8	1.3
	71	162	77	1.1	12.9
14	233	170	71	1.2	3.0
	118	119	47	1.1	5.0
	78	135	54	1.7	4.1
15	208	401	51	2.1	6.9
	99	62	66	0.9	3.5
	93	189	82	0.6	6.2
16	118	97	44	0.9	5.6
	99	47	49	0.8	3.4
17	316	140	19	1.3	3.4
	55	67	88	1.6	4.9
	55	85	82	1.7	6.7
18	210	92	52	1.1	4.6
	51	27	92	1.8	6.2
	113	123	62	1.1	7.0
19	103	92	40	1.3	5.4
	152	111	82	1.2	2.6
	27	142	91	1.4	2.5
20	128	83	32	0.8	2.5
	154	131	49	1.3	3.4

Table 2.6 (contd.)

Treatment	Acetic acid-extractable (µg/g)				
	Fe	Aℓ	Mn	Cu	Zn
21	425	48	36	0.5	1.7
	742	140	65	3.5	2.3
	62	74	84	0.7	5.7
22	12	16	73	1.6	1.8
	121	116	31	0.7	3.0
	8	44	93	2.3	4.5
23	73	53	73	2.0	4.8
	35	29	80	1.0	2.5
24	62	84	59	1.4	3.6
25	119	199	42	1.2	5.8
	59	56	31	0.8	5.4
	5	18	113	1.2	11.8
26	126	154	71	1.1	5.2
	181	142	42	1.4	2.7
	38	72	85	1.3	4.9
27	121	40	45	0.7	2.2
	71	76	66	1.4	3.6
	7	17	98	1.5	2.5
28	179	94	54	1.1	4.0
	18	10	118	2.1	1.7
	8	7	94	16.5	1.5
29	166	80	54	0.8	2.9
	427	100	81	4.3	3.5
	13	35	98	2.4	7.3
30	50	66	71	1.9	4.8
	242	140	45	2.3	3.8
31	182	174	53	1.0	3.4
	69	84	69	1.2	4.4
	46	121	75	1.0	6.3

growth, at which stage differences arising as a direct result of treatments may not show up due to variations in spoil properties. For this reason it was felt inappropriate to average the results of replicate plots and therefore the discussion of results is based on 82 separate plots.

2.11.1 Herbage iron content

The level of iron in the herbage (Fe (herbage)) ranges from 217-2179 µg/g, with spoil levels covering the range 5-742 µg/g. Acetic extractable Fe is significantly negatively correlated with spoil pH ($r = -0.413^{***}$) in agreement with soil analyses (Russell 1973, Kunishi 1982), but uptake by the herbage is not related to the level of Fe in the spoil ($r = 0.001$), nor is herbage content affected by spoil pH (Fig. 2.7, $r = 0.005$). However, by taking only those samples with Fe (herbage) < 1000 µg/g ($n = 75$) there exists a negative correlation significant at the 1% level between pH and Fe (herbage) ($r = -0.321$). This result agrees with the findings of Kimber (1982) who found levels of Fe in common bent (*Agrostis tenuis*) growing on spoil with pH < 4 many times greater than plants on spoil with pH > 4. Table 2.7 shows selected data for those samples with Fe (herbage) < 1000 µg/g.

Table 2.7. Correlation of herbage Fe content, spoil pH, yield and acetic extractable Fe in spoil for those samples with Fe (herbage) $< 1000\ \mu g/g$

pH v Fe (herbage)	$r = -0.321^{**}$
pH v Fe (spoil)	$r = -0.402^{***}$
pH v yield	$r = 0.119^{n.s.}$
Fe (spoil) v Fe (herbage)	$r = 0.135^{n.s.}$

n.s. = not significant.

As Table 2.7 shows, low spoil pH leads to an increase in Fe (herbage) which is not, however, significantly related to the increase in Fe (spoil) with increasing acidity, due to the fact that spoil levels exceed plant requirements and will not be limiting to plant growth. It may be that reduced Fe uptake at higher pH is due to interference from high levels of carbonate and bicarbonate with lime addition (Woolhouse 1966).

There is a non-linear relationship between yield reduction and Fe uptake (Fig. 2.8). Fe uptake reduces yield at all levels, most dramatically when Fe (herbage) exceeds 500 μ g/ g. By plotting the logarithm of each parameter a relationship is found which is significant at the 0.1% level (Fig. 2.9, r = -0.392). It is impossible to draw conclusions on the effect of Fe alone on yield as there are many other factors which have as great an effect, as discussed in later sections, although clearly Fe uptake has a strong influence in restricting plant growth.

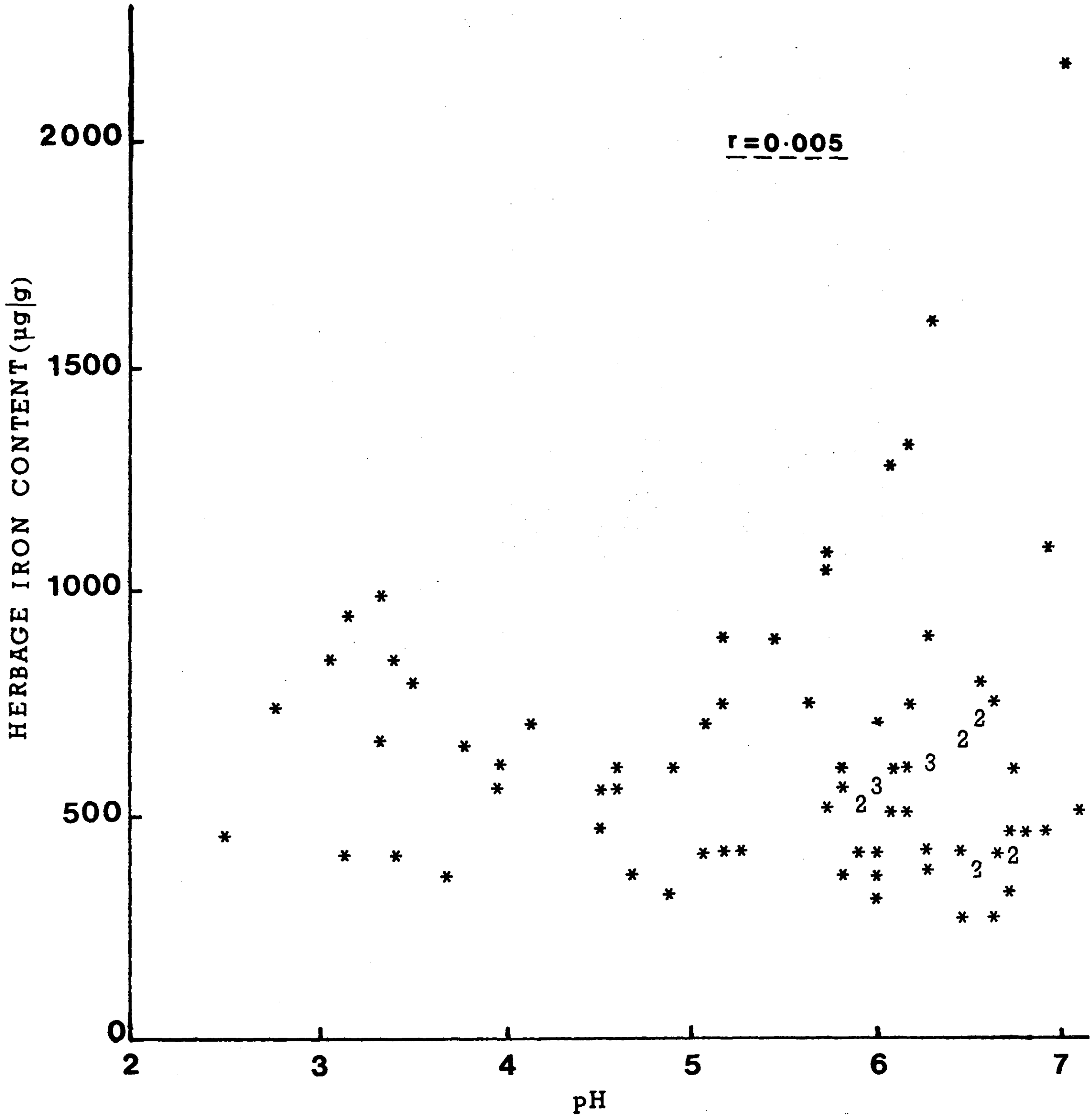


Figure 2.7. Relationship between spoil pH and herbage iron content.

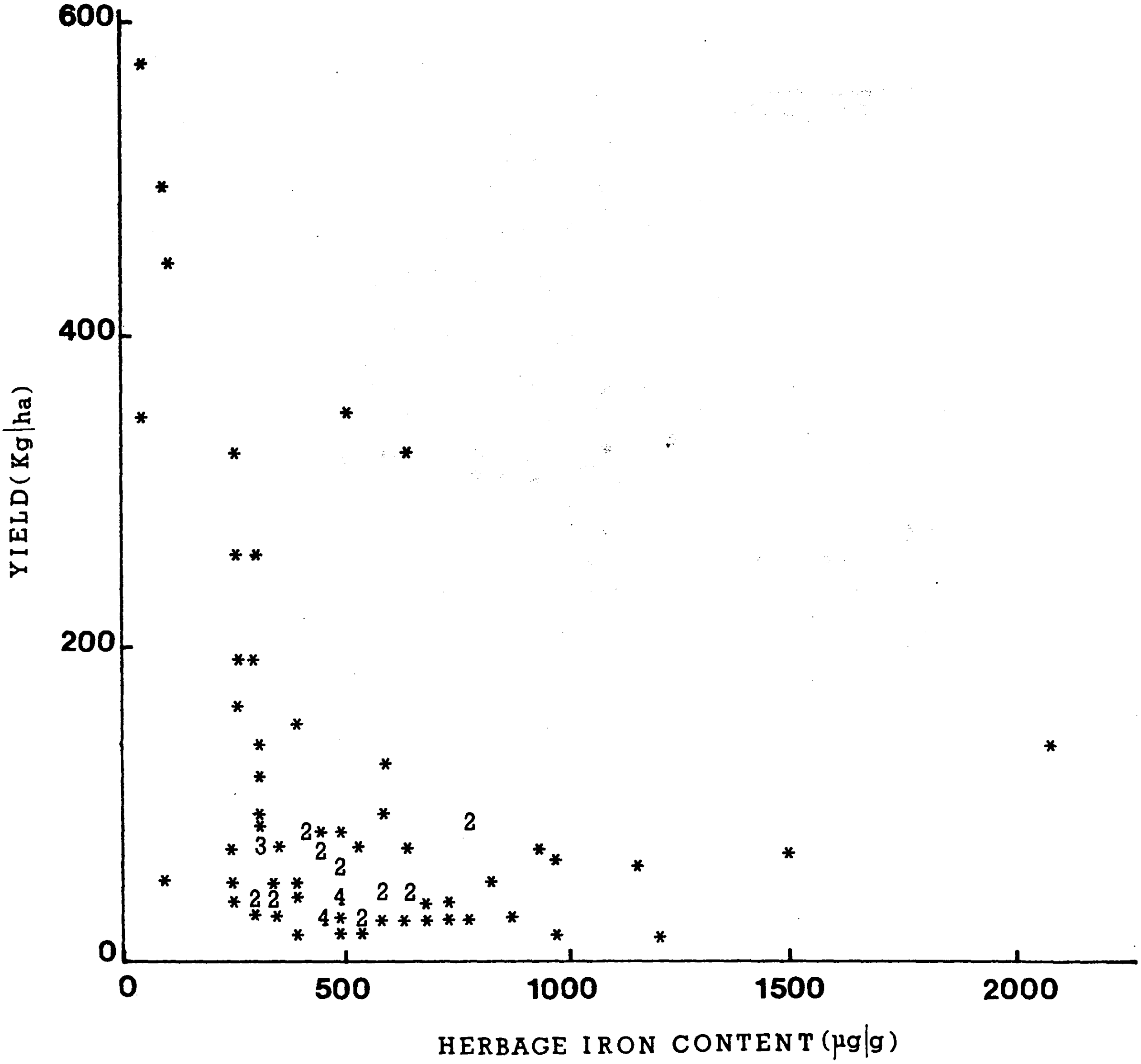


Figure 2.8. Relationship between herbage iron content and yield.

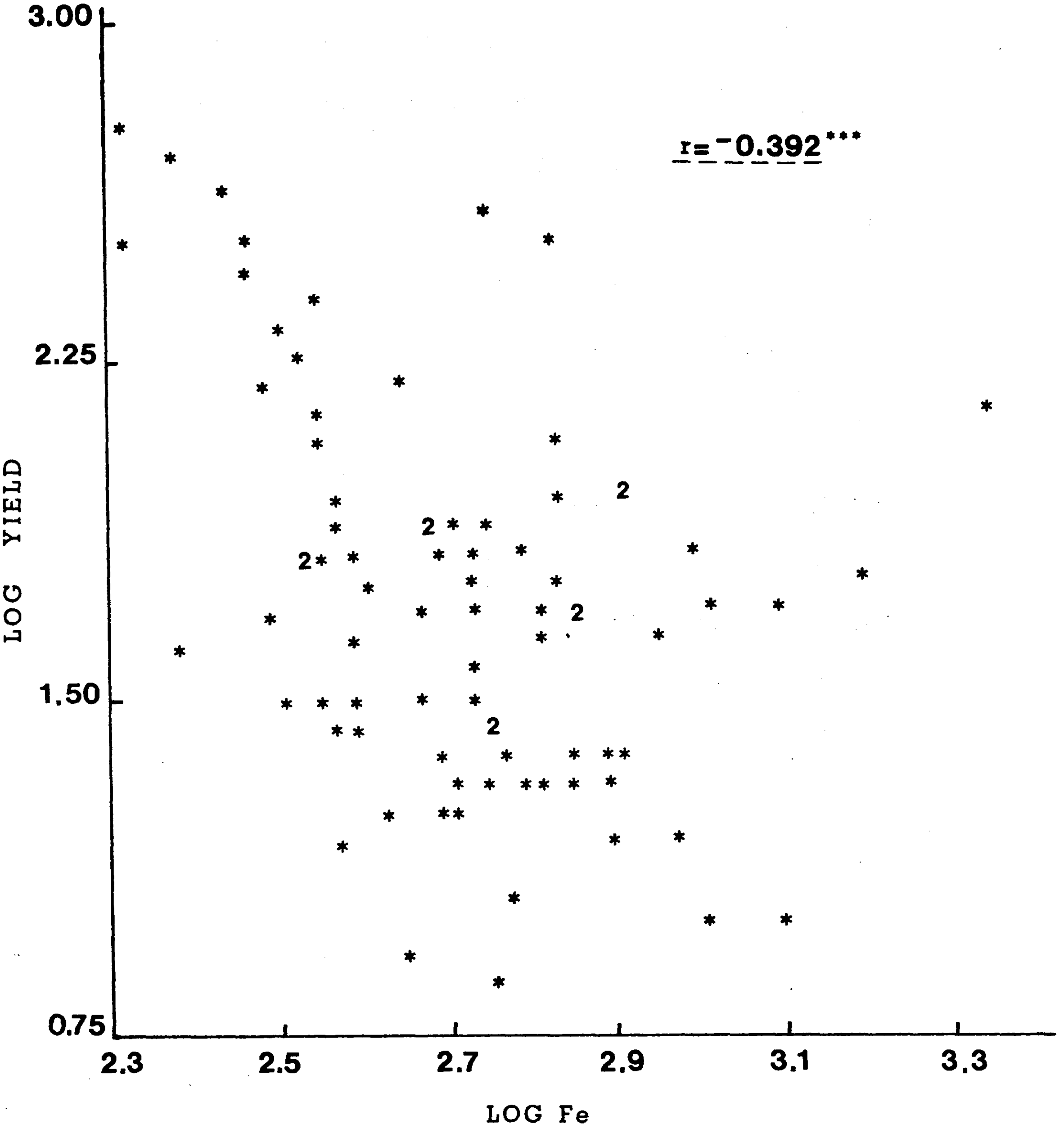


Figure 2.9. Plot of logarithm (Fe (herbage)) against logarithm (yield).

2.11.2 Herbage aluminium content

Although aluminium is not essential for plant growth, failure of crops due to Aℓ toxicity is a major problem on acid soils, causing restricted root growth and reduced phosphorus uptake (Hallsworth et al. 1957, Brady 1974, Kunishi 1982). Herbage Aℓ content ranges from 120-3026 μg/g in the trial, with spoil levels of 6-427 μg/g being found.

As is the case with Fe, the level of acetic extractable Aℓ in the spoil is strongly influenced by spoil pH ($r = -0.713^{***}$) although spoil levels do not influence herbage uptake ($r = 0.025$). The increase in Aℓ (herbage) at lower pH reported by Kimber (1982) was not found in this study (Fig. 2.10, $r = -0.099$). There is a reduction in plant yield due to Aℓ uptake (Fig. 2.11), with yield decreasing markedly when Aℓ (herbage) > 600-700 μg/g. Fig. 2.12 shows log Aℓ (herbage) plotted against log yield ($r = -0.437^{***}$).

2.11.3 Relationship between herbage Fe and Aℓ content

The levels of acetic extractable Fe and Aℓ in the spoil, both strongly influenced by pH, are closely related ($r = 0.358^{***}$), and although neither show any relationship with plant uptake there is a strong correlation which is unaffected by lime and organic amendments between the levels of these elements in the herbage (Fig. 2.13, $r = 0.770^{***}$) - Table 2.8.

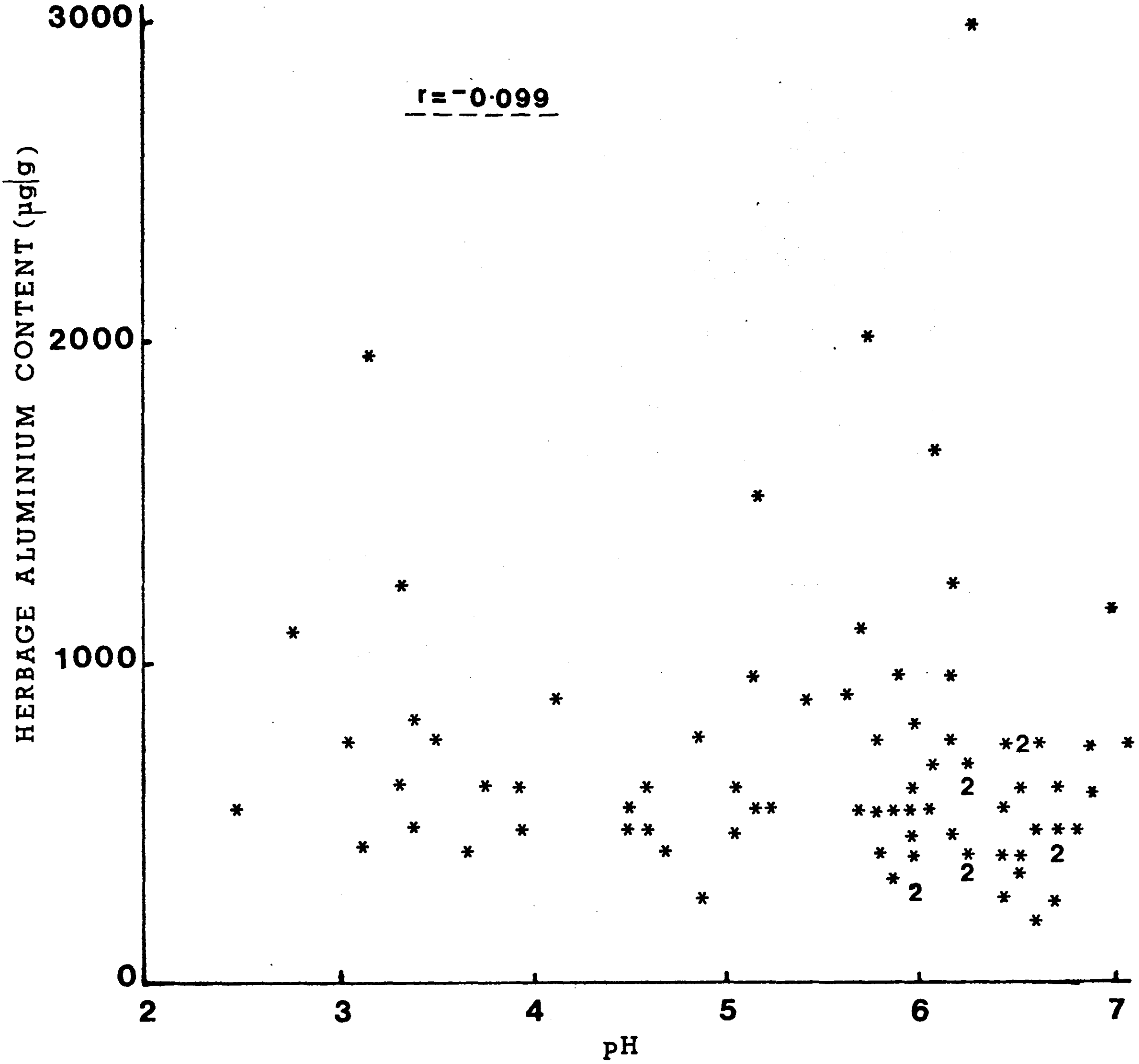


Figure 2.10. Relationship between spoil pH and herbage aluminium content.

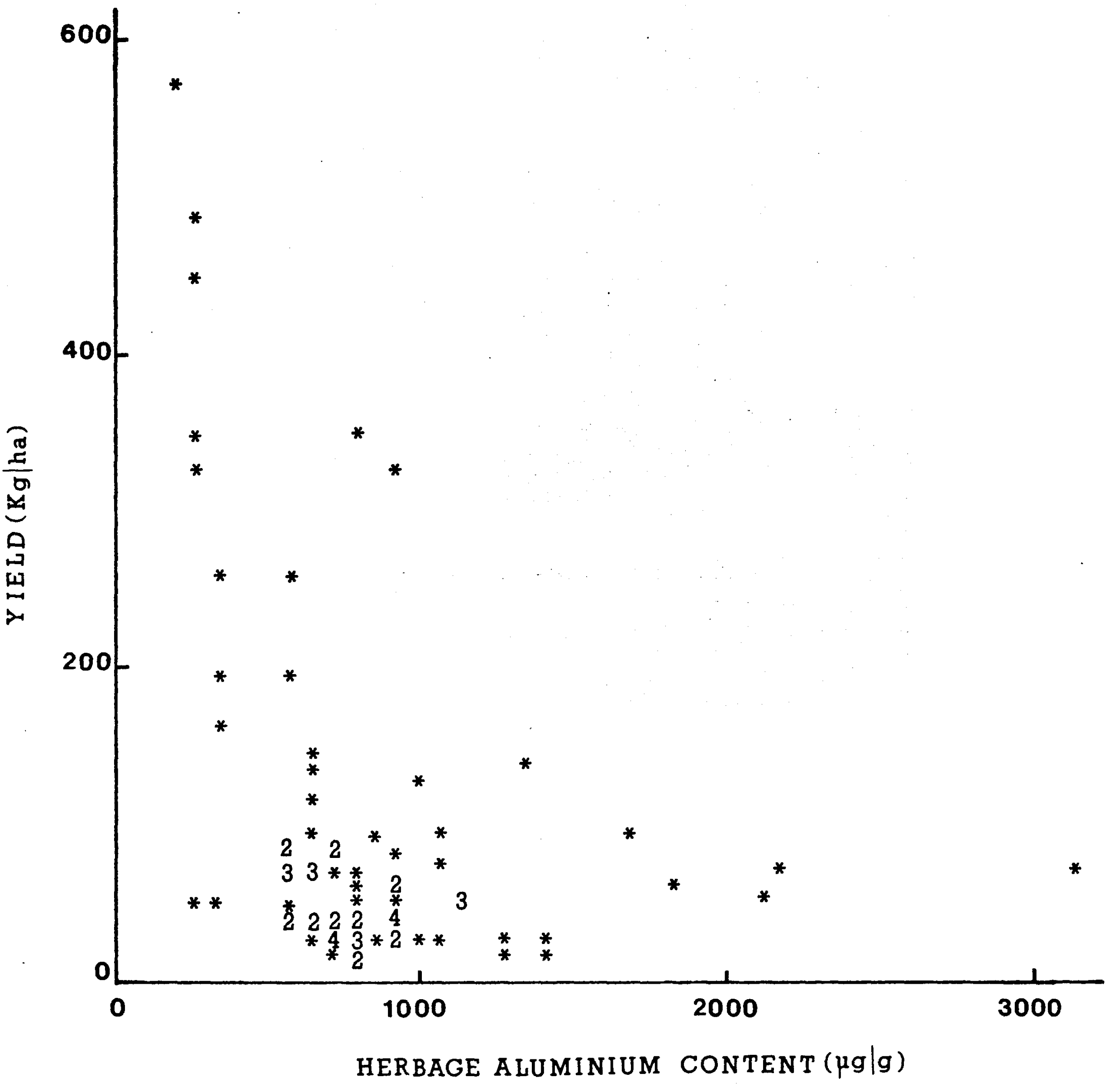


Figure 2.11. Relationship between herbage aluminium content and yield.

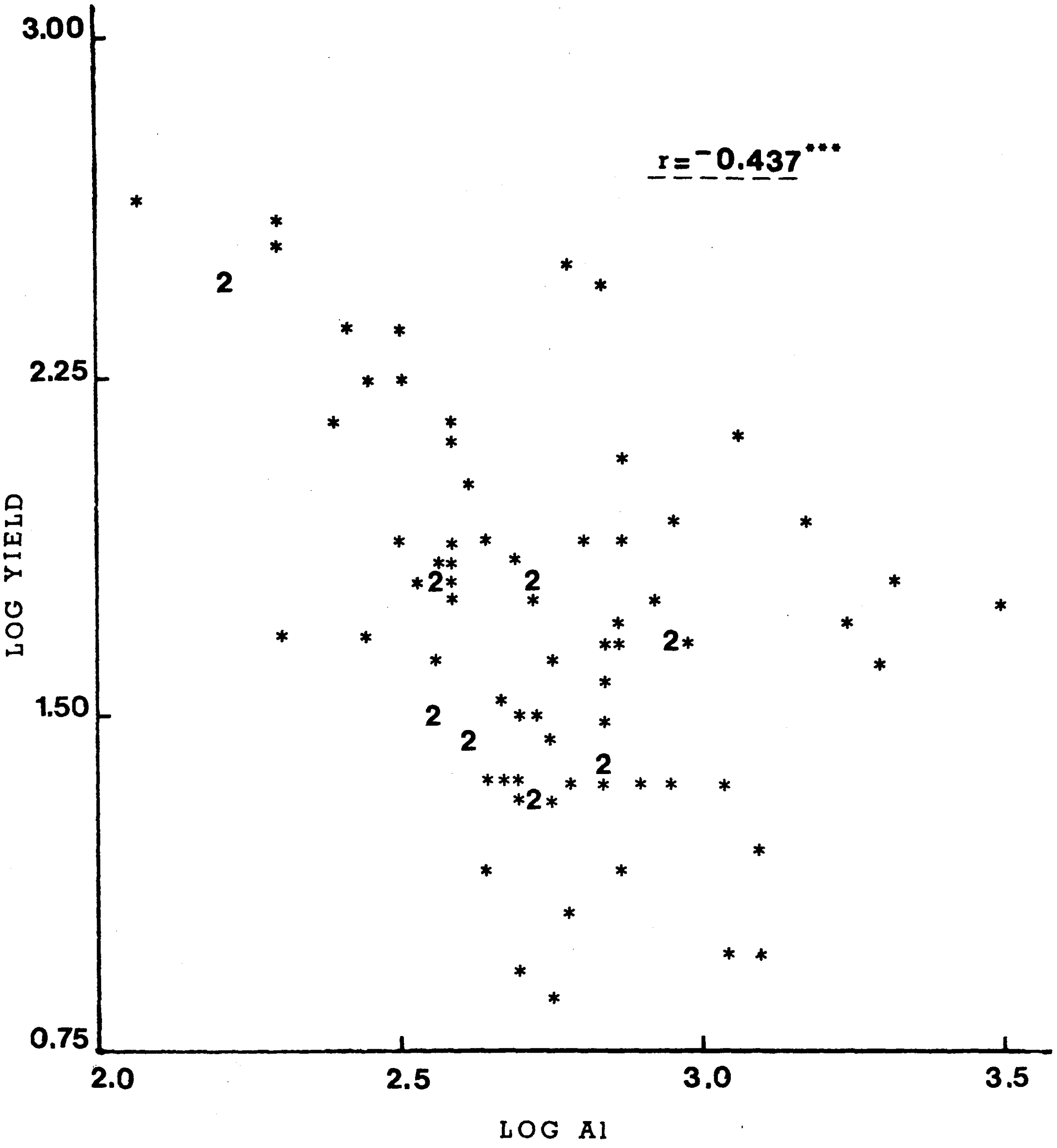


Figure 2.12. Plot of logarithm (Aℓ(herbage)) against logarithm (yield).

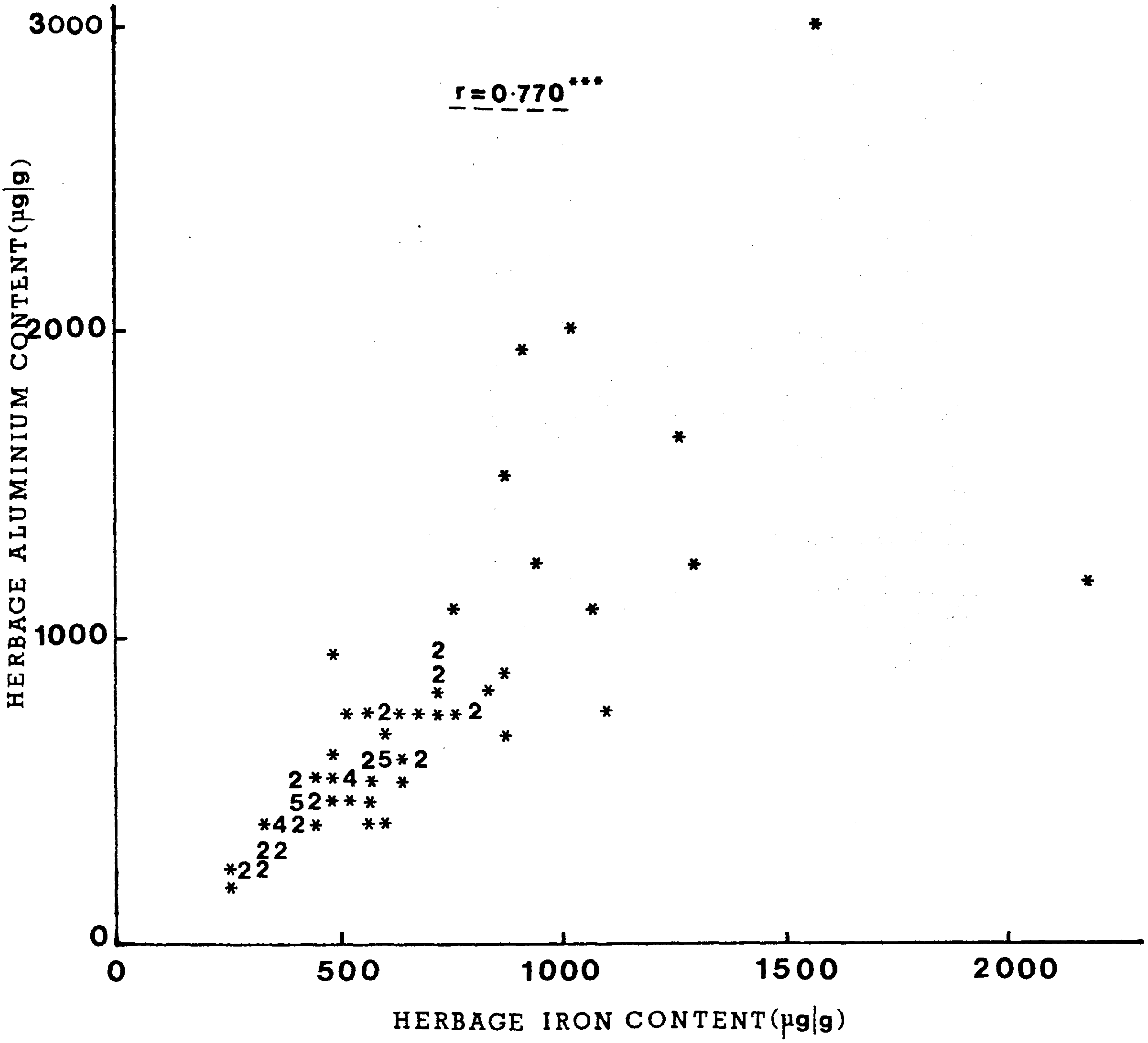


Figure 2.13. Relationship between herbage iron and aluminium content.

Table 2.8. Significance of the correlation coefficient between Fe and Aℓ levels in spoil and herbage

Treatment	No. of plots	Herbage Fe v Aℓ	Spoil Fe v Aℓ
All treatments	82	***	***
Unlimed plots	10	**	n.s.
25 tonnes/ha lime	38	***	**
50 tonnes/ha lime	34	***	*
Peat added	17	***	n.s.
Chicken manure added	20	**	n.s.
Alginure added	21	***	***
Sewage sludge added	18	***	n.s.

As Table 2.8 shows, variations in the level of Fe and Aℓ in the spoil are not reflected in the herbage, the relationship within the plant being independent of the spoil levels. In order to maintain the balance in the plant these elements must be taken up in equivalent amounts from the spoil solution by a regulatory system which is selective when they are not in balance in the spoil.

2.11.4 Herbage manganese content

Herbage manganese content ranges from 98-306 μg/g, with acetic extractable levels in the spoil of 18-118 μg/g. Fig. 2.14 shows the variation in Mn (herbage) with pH, and although there is no significant correlation over all plots (r = 0.074), there is a general trend of higher

Mn (herbage) values at higher pH - by omitting those values of Mn (herbage) > 300 µg/g the correlation is significant at the 5% level (n = 80, r = 0.276*).

The behaviour of Mn in the spoil contrasts with that of the other trace elements studied in that the level of acetic extractable Mn increases with pH, being positively correlated at the 0.1% level over all plots (n = 82, r = 0.551). This is in contrast to spoil data of MacLean and Dekker (1976) and soil data of Kunishi (1982) who found an increase in spoil/ soil levels at lower pH, while many workers have reported higher plant Mn content at lower pH (McNaught and Dorofaeff 1973, Dick et al. 1985). Pulford et al. (1982) studied seven colliery spoil sites in the Central Scottish coalfield and found a significant positive correlation (at 0.1% level) between Mn (spoil) and pH, as did Cornwell and Stone (1973) and Down (1975) working with spoils of Pennsylvania and Somerset respectively.

Release of Mn to solution from the acid breakdown and dissolution of primary Mn-containing minerals under oxdising conditions leads to the formation of hydrous manganese oxides (Kimber 1982, Pulford et al. 1982), unreacted solution Mn being leached from the spoil. As a result there is not a large pool of acetic extractable Mn present in the spoil, and this explains the lack of correlation between Mn (herbage) and Mn (spoil) (r = 0.169). Mn oxides in soils are known to exist in many more forms than iron and aluminium oxides, including mixed-valence oxides, some of which are acetic extractable and may be able to supply Mn to plants. This accounts for the fact that Mn (herbage) and spoil pH are positively correlated.

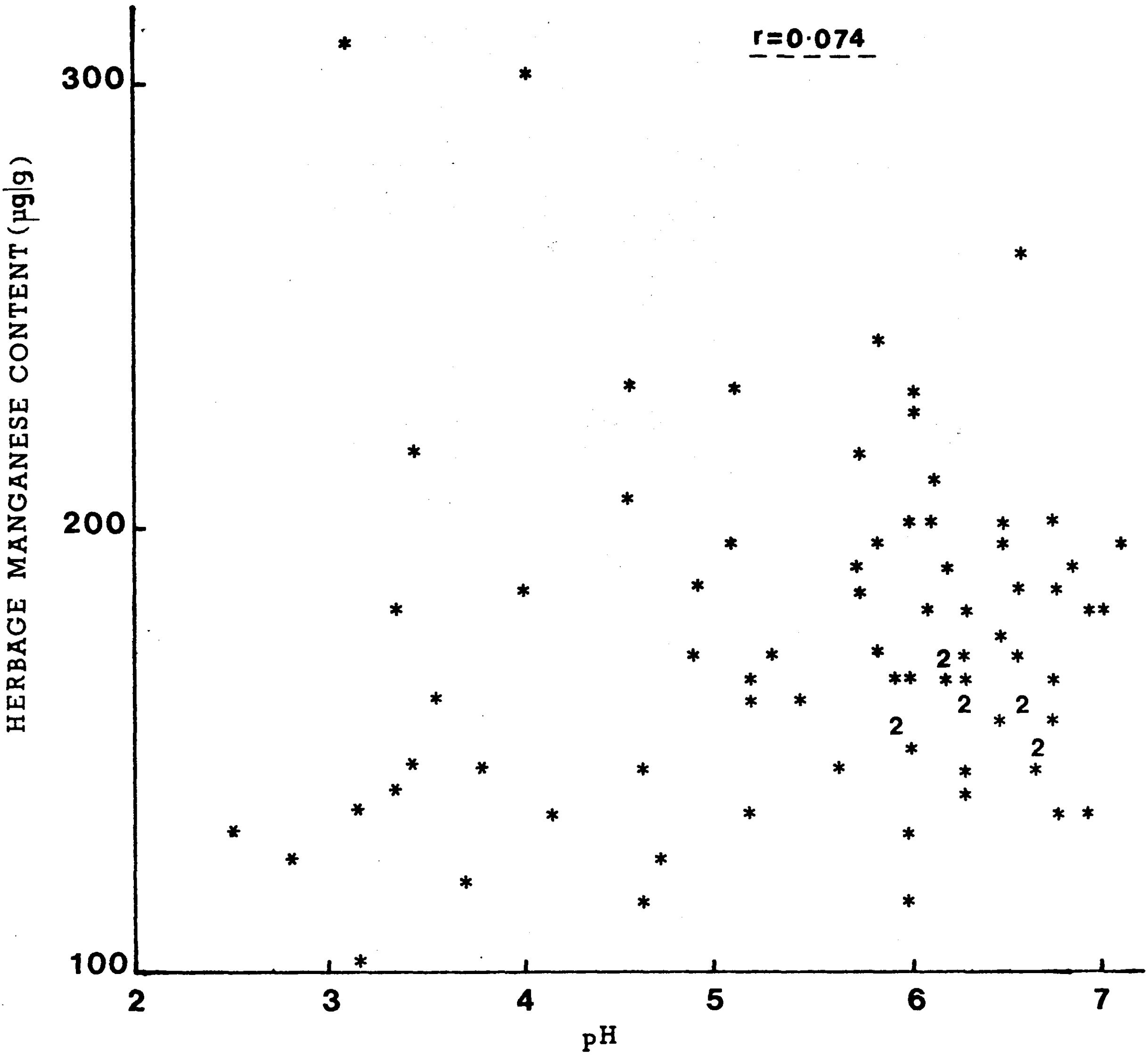


Figure 2.14. Relationship between spoil pH and herbage manganese content.

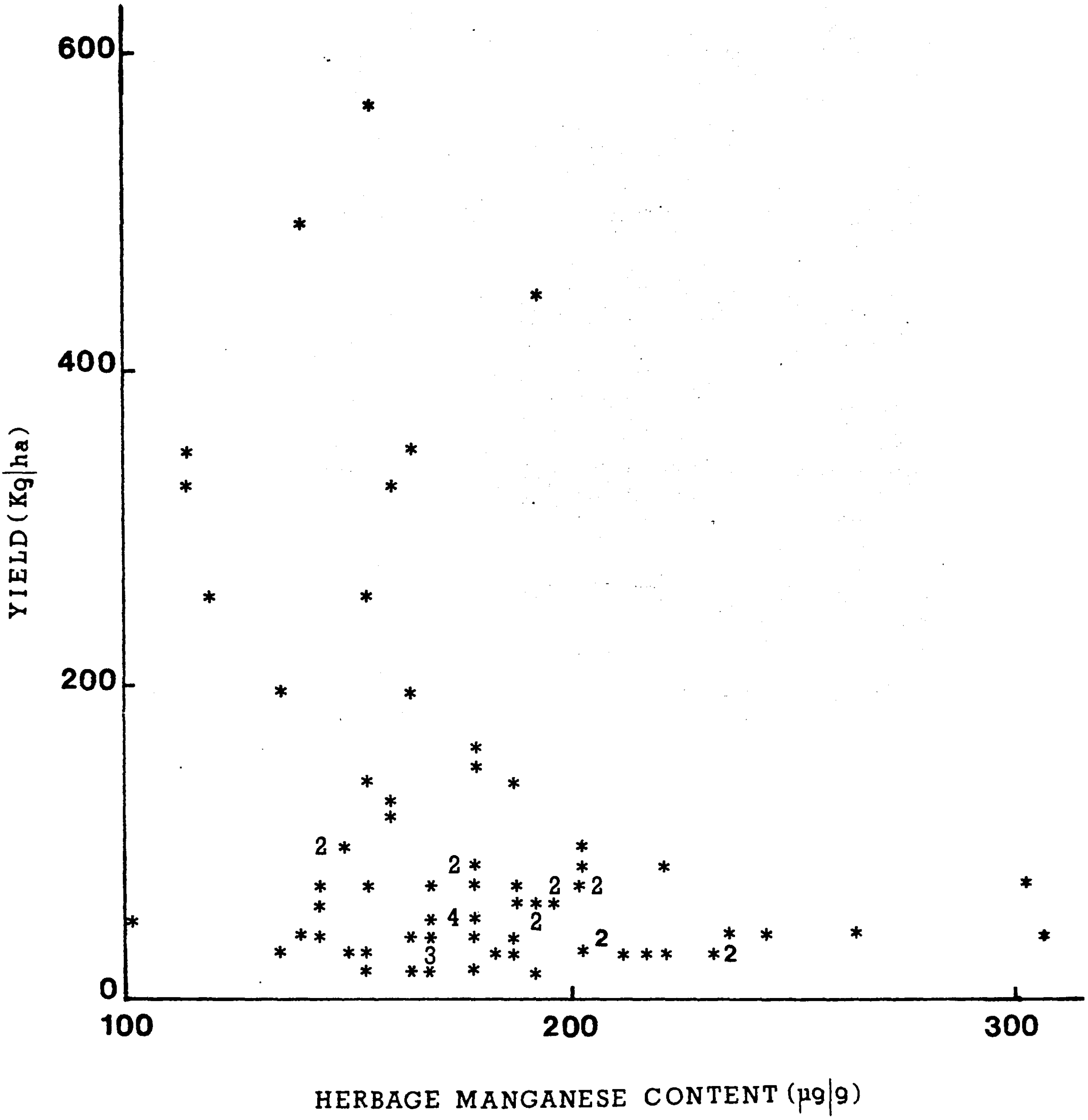


Figure 2.15. Relationship between herbage manganese content and yield.

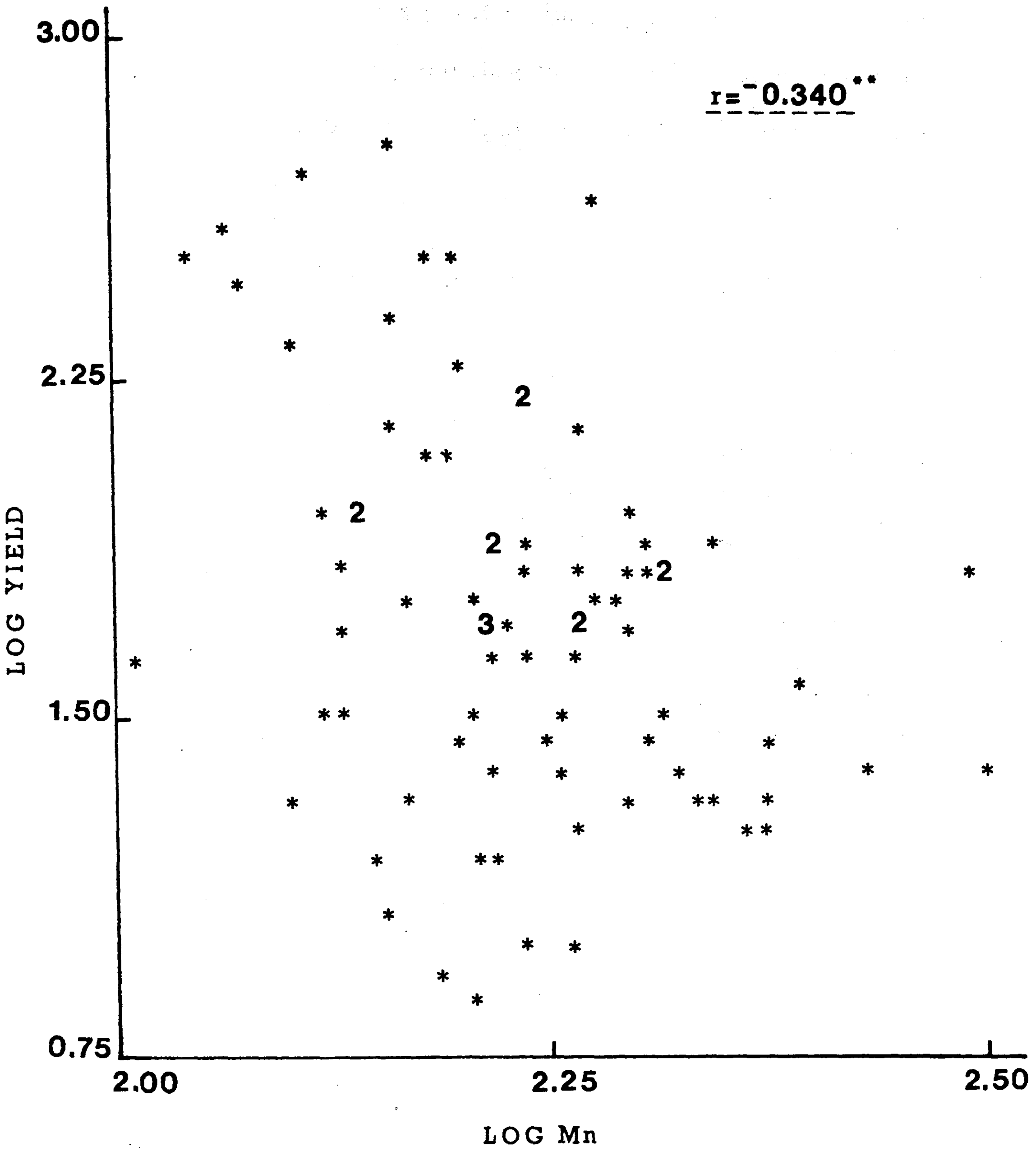


Figure 2.16. Plot of logarithm (Mn(herbage)) against logarithm (yield).

Mn uptake reduces plant yield, particularly when Mn (herbage) > 100 μg/g (Fig. 2.15). By plotting the logarithms of Mn (herbage) against yield the relationship is significant at the 1% level ($r = -0.340$) (Fig. 2.16).

2.11.5 Herbage copper and zinc content

The levels of copper and zinc found in spoil and herbage are:

Element	Spoil (μg/g)	Herbage (μg/g)
Cu	0.5-16.5	12-62
Zn	1.3-15.8	39-351

Neither element shows any significant correlation with spoil pH or herbage yield, although some workers have reported a reduction in herbage content through increasing soil pH by liming (Brown and Jurinak 1964, Black 1968).

Stucky _et al_. (1980) found that phytotoxic effects were induced in perennial ryegrass (_Lolium perenne_) grown on acid mine spoil by Cu and Zn when the herbage content exceeded 20 μg/g and 200 μg/g respectively, while Cunningham _et al_. (1975) reported a yield reduction at 23 μg/g Cu and 289 μg/g Zn. In the present study the levels of Cu (herbage) exceed that quoted as being potentially phytotoxic in many cases, while Zn (herbage) levels exceed 200 μg/g in only 4 plots, most samples having values < 100 μg/g, therefore Zn toxicity is not likely to be a problem in this case.

There is a relationship within the plant, which is unaffected by spoil pH, between the levels of Cu and Zn (Table 2.9, Fig. 2.17), although there is no relationship between the acetic extractable levels of these elements in the spoil (r = 0.089).

Table 2.9. Correlation between herbage Cu and Zn levels

Zn (herbage)	No. of plots	Correlation coefficient
All data	82	$0.181^{n.s.}$
< 170 μg/g	75	0.261^{*}
< 160 μg/g	74	0.380^{***}

Note. For all values of Zn (herbage) < 160 μg/g the Cu (herbage) v Zn (herbage) relationship remains significant at the 0.1% level.

The results in Table 2.9 show that there is a relationship between Cu and Zn uptake, up to a level of Zn (herbage) of 160 μg/g, above which there may be interference with Cu uptake or substitution of Zn^{2+} for Cu^{2+} in metabolic reactions. Reduction of Cu uptake by Zn has been reported by Chaudhry and Loneregan (1970) and Loneregan (1975), while Chaudhry and Loneregan (1972) found that Cu reduced uptake of Zn, although no such relationship was found in this study.

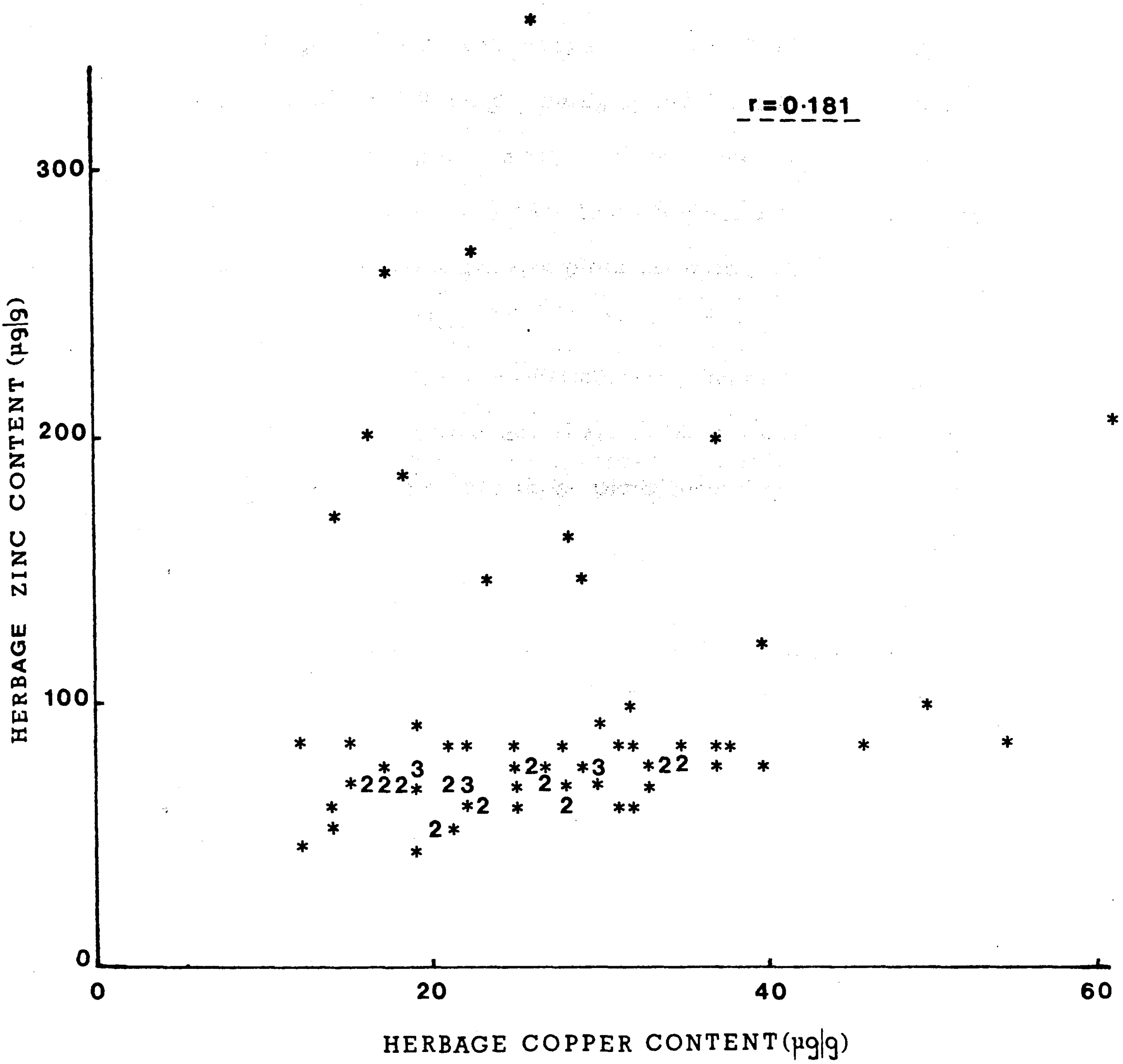


Figure 2.17. Relationship between herbage copper and zinc content.

2.11.6 Herbage calcium and magnesium content

Herbage calcium levels range from 2566-9097 μg/g, with magnesium levels of 959-2023 μg/g being found. Addition of lime to the spoil increases Ca (herbage) in most cases in agreement with Wear (1956) and Kunishi (1982), however, there is no further increase when comparing the levels of Ca in the herbage from plots receiving 25 tonnes lime/ha with those receiving 50 tonnes/ha.

There is a positive correlation, significant at the 0.1% level between spoil pH and Ca (herbage) (Fig. 2.18, r = 0.419) (Dick et al. 1985), however, when considering only those plots receiving lime, no such relationship exists (Table 2.10). Costigan et al. (1982) reported that herbage yield was significantly, positively correlated with the level of both Ca (herbage) and Mg (herbage) in limed spoil although this relationship was not found here. These workers, however, found considerably higher levels of Ca and Mg in the herbage (Ca 20,000-50,000 μg/g, Mg 3,000-6,000 μg/g) as a result of addition of magnesian limestone at rates of up to 100 tonnes/ha.

Over all the plots, herbage Ca and Mg levels are closely associated (Fig. 2.19, $r = 0.352^{**}$), this relationship being independent of spoil pH (Table 2.10).

Table 2.10. Relationship between herbage Ca and Mg content and spoil pH

Ca(herbage) v Mg(herbage)		Ca(herbage) v pH	Mg(herbage) v pH
All data (82)	0.352^{**}	0.419^{***}	$-0.124^{n.s.}$
Limed plots (72)	0.506^{***}	$0.136^{n.s.}$	$-0.027^{n.s.}$

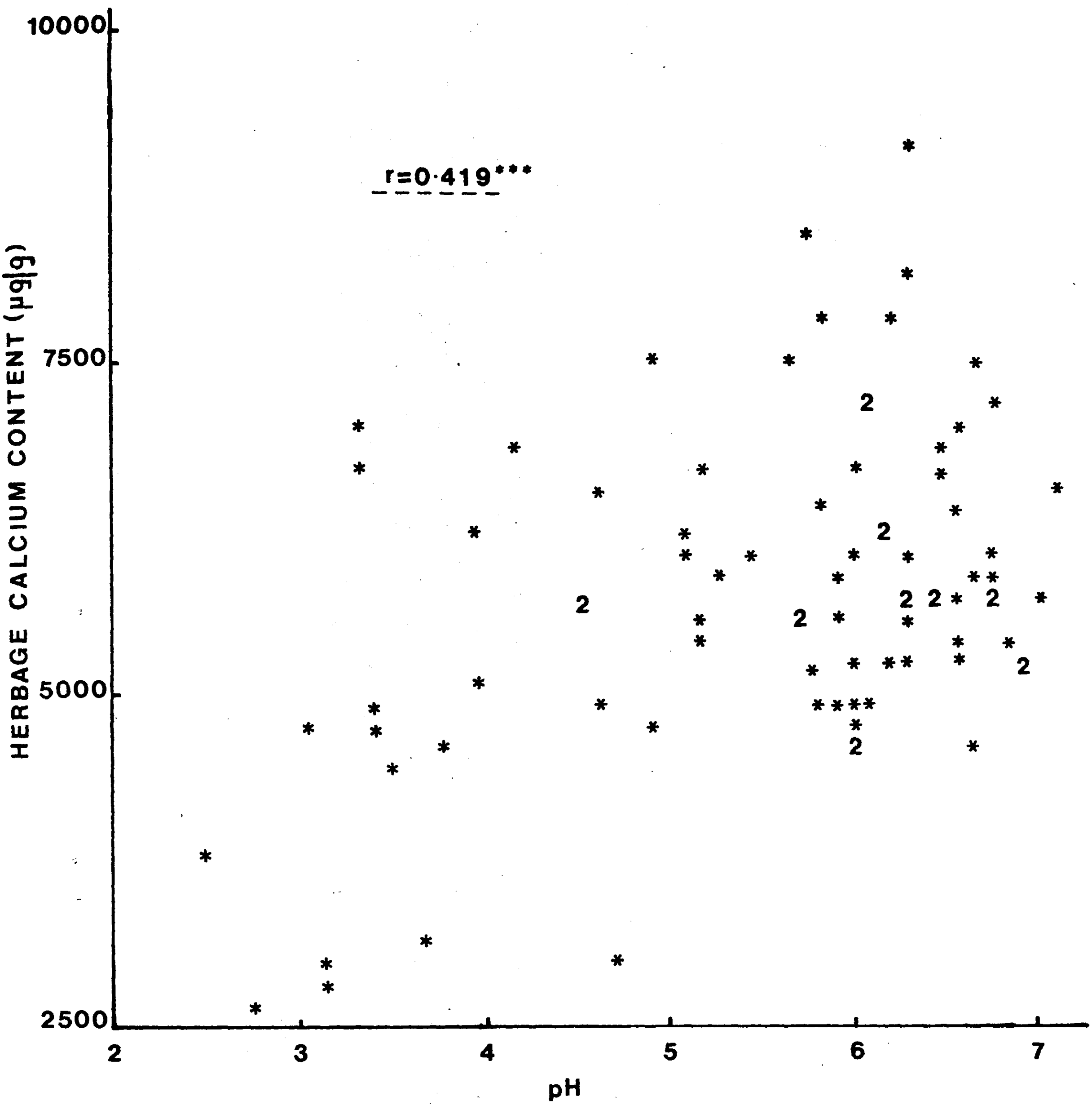


Figure 2.18. Relationship between spoil pH and herbage calcium content.

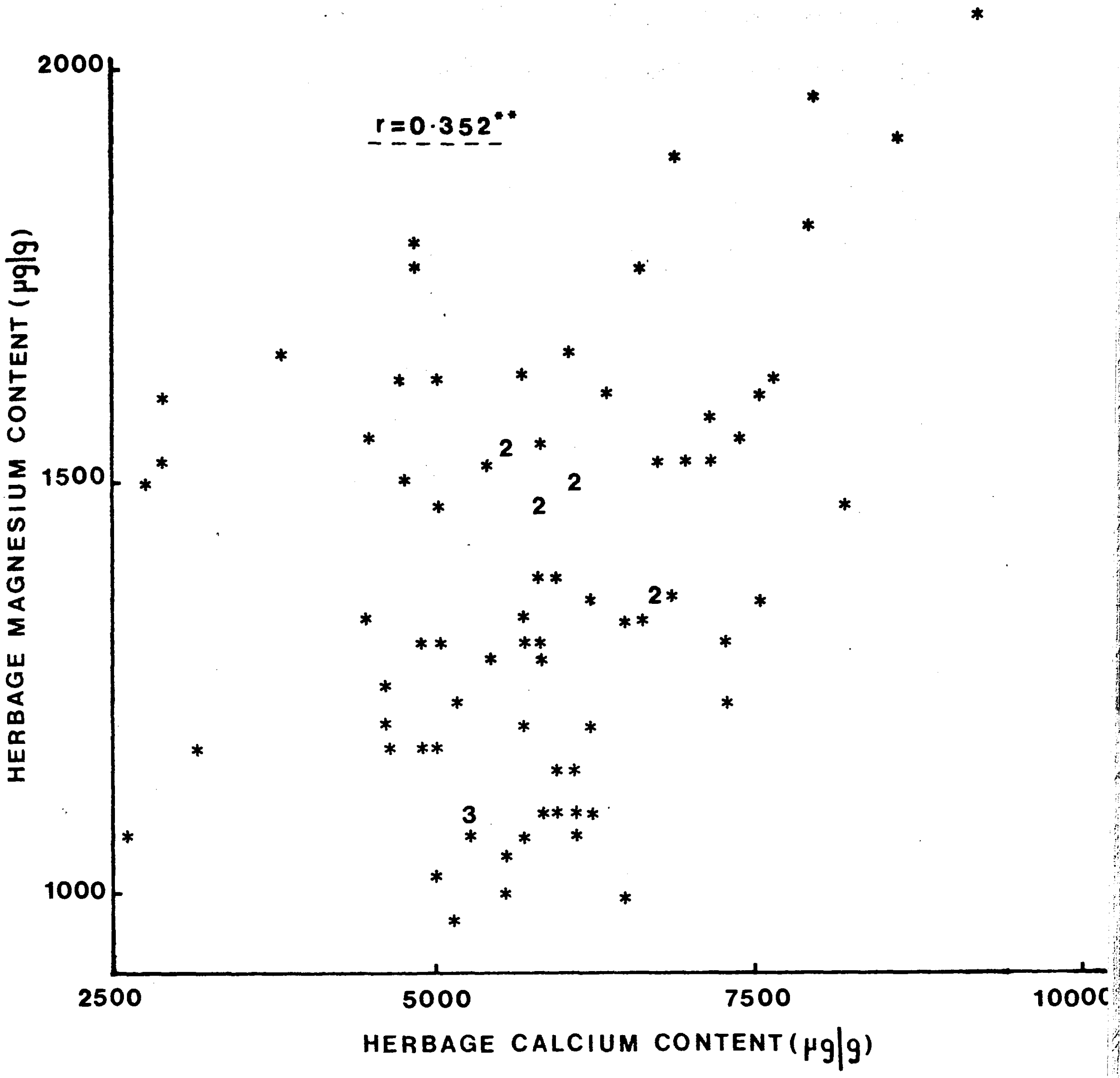


Figure 2.19. Relationship between herbage calcium and magnesium content.

The level of Ca in the herbage is generally 4-5 times greater than Mg (herbage) (Table 2.5). Ca is required by plants as a constituent of the cell wall while Mg is a major ion of the chlorophyll molecule, essential for cell function and plant growth, therefore an increase in plant yield, which is a result of an increase in cell wall production and associated chlorophyll production, must raise Ca and Mg levels together in the plant.

2.12 Mineral Intake of Animals Grazing on Colliery Spoil

Table 2.11 shows the maximum tolerable levels of dietary Fe, Aℓ, Mn, Cu, Zn, Ca and Mg for cattle and sheep, together with the range found in the herbage in this study (unlimed and limed plots).

Table 2.11. Maximum tolerable levels of dietary Fe, Aℓ, Mn, Cu, Zn, Ca and Mg and levels found in herbage

Element	*Cattle(μg/ g)	*Sheep(μ g/ g)	Herbage content(μg/g)
Fe	1,000	500	217-2,179
Aℓ	1,000	1,000	120-3,026
Mn	1,000	1,000	98-306
Cu	100	25	12-62
Zn	500	300	39-351
Ca	20,000	20,000	2,566-9,097
Mg	5,000	5,000	959-2,023

* Data from National Research Council (United States) (1980)

There are many factors which determine the amount of each mineral taken up by the grazing animal from the forage. The stage of maturity of the plant as well as the form of the element in the plant, and the concentration of other essential and non-essential elements affect the animal's digestive tract and its ability to absorb each element. Furthermore, the physiological state of the animal and its nutrititional status will also influence the level at which an element will cause adverse effect.

As Table 2.11 shows there may be cause for concern in some cases if animals were grazed solely on herbage grown on reclaimed colliery spoil, particularly in the cases of Fe, Aℓ and Cu in sheep.

It is estimated that grazing animals may take over 10% of their dry matter intake as soil (National Research Council 1980) and this factor may be of greater importance in a spoil situation, due to high levels of trace elements in spoils.

Although no conclusions can be drawn from the data presented in this work concerning mineral intake by grazing stock the possibility exists that animals may suffer mineral imbalance or toxicity if grazed for prolonged periods on pasture, and fed hay or silage grown on reclaimed spoil.

2.13 Summary

The levels of acetic acid-extractable Fe and Aℓ in the spoil are reduced at higher pH although herbage content is not related to spoil levels. For those samples with Fe (herbage) $<$ 1000 μg/g there is a reduction in Fe uptake at higher pH, possibly due to interference by carbonate and bicarbonate from unreacted lime. Over all plots there is a significant correlation between Fe and Aℓ in the herbage, this relationship existing regardless of lime and organic matter treatments and independent of variations in acetic acid-extractable levels in the spoil. Both elements reduce yield at all levels, most significantly when Fe $>$ 500 μg/g and Aℓ $>$ 600-700 μg/g.

In contrast to other trace elements studied Mn levels in the spoil increase at higher pH and although there is no correlation between spoil and herbage levels, spoil pH and Mn (herbage) are also positively correlated. Mn reduces yield at all levels, most marked when Mn $>$ 100 μg/g.

Cu and Zn levels in the plant show no correlation with spoil pH or herbage yield although their uptake is closely associated up to a level of Zn (herbage) of 160 μg/g, above which there may be interference with Cu uptake.

Ca (herbage) is increased by liming and is significantly correlated with pH but no such relationship exists for Mg, despite the levels of these elements in the plant being closely related.

The problem of trace element imbalance in animals grazing on colliery spoil is an aspect which requires attention, and may alter at later harvests, but early indications are that there may be a problem due to Fe, Aℓ and Cu in sheep.

CHAPTER 3

SPOIL PHOSPHORUS STATUS AND FACTORS AFFECTING PHOSPHATE ADSORPTION BY SPOIL

3.1 INTRODUCTION

After nitrogen, phosphorus is the element most restrictive to plant growth in soil (Thompson and Troeh 1973) and colliery spoil (Gemmell 1977, Bloomfield et al. 1982). Whereas nitrogen levels can be raised by the inclusion of legumes among the species grown, no such biological aid is available for phosphorus addition. However, the fixation of nitrogen by legumes is governed to a large extent by the supply of phosphorus in the soil (Palmer and Iverson 1983, Hue and Adams 1984), therefore soil phosphorus takes on a role more important than simply that of a plant nutrient per se.

Low levels of plant available phosphorus have been found to be a major inhibitory factor to plant growth in many situations; in acid soils (Hsu 1964, Haynes 1982), metalliferous mine waste (Berg et al. 1975), China clay waste (May et al. 1973) and colliery spoil (Pulford 1976, Bradshaw and Chadwick 1980, Palmer and Iverson 1983), the characteristics of phosphorus deficiency being a stunted root system and reduction in top growth. Unamended colliery spoil will usually support a sparse vegetation cover which is maintained only if nutrients are supplied through mineral weathering and rainfall. However, in order to achieve and maintain a reasonable cover, nutrients, notably nitrogen and phosphorus, must be added as fertilizer.

3.2 Soil Phosphorus Cycle

Figure 3.1 shows the soil phosphorus cycle in a simplified form

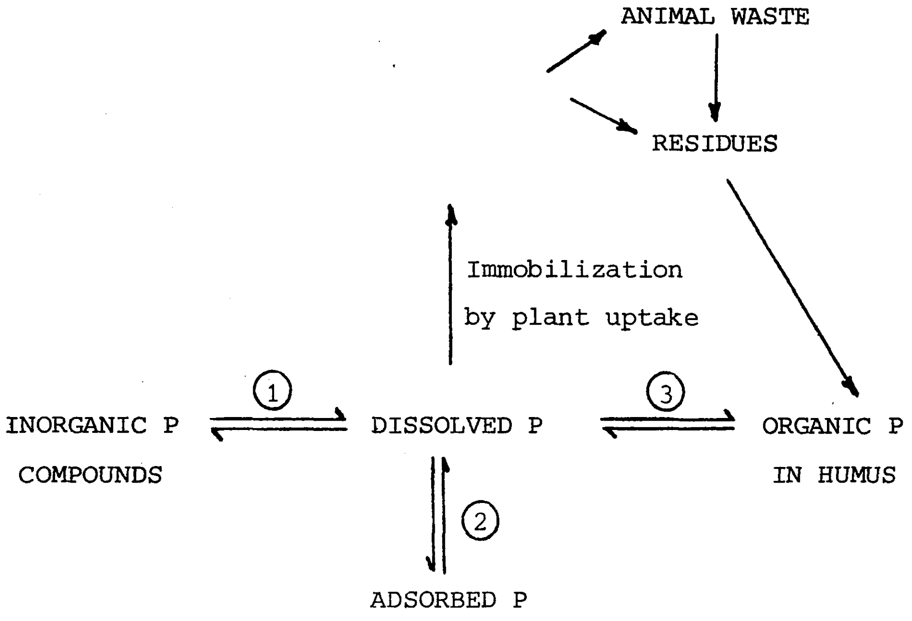


Fig. 3.1. Soil phosphorus cycle

Dissolved phosphate, available for plant uptake, exists in the soil mainly as $H_2PO_4^-$ and HPO_4^{2-}, the proportion present in these two forms depending on the soil pH; at pH 7.2 phosphate in solution occurs equally in both forms, while the $H_2PO_4^-$ ion is dominant below this pH and HPO_4^{2-} dominates under more alkaline conditions. It is estimated that the proportion of the TOTAL phosphorus content of a soil which is in solution is approximately 0.1% (Talibudeen 1957, Donahue *et al.* 1971) not all of which may be immediately taken up by plants due to competition from soil microorganisms and leaching losses.

① Inorganic phosphorus compounds $\rightleftharpoons$ Dissolved phosphorus

Inorganic phosphorus compounds in soil, mainly iron, aluminium and calcium compounds include:

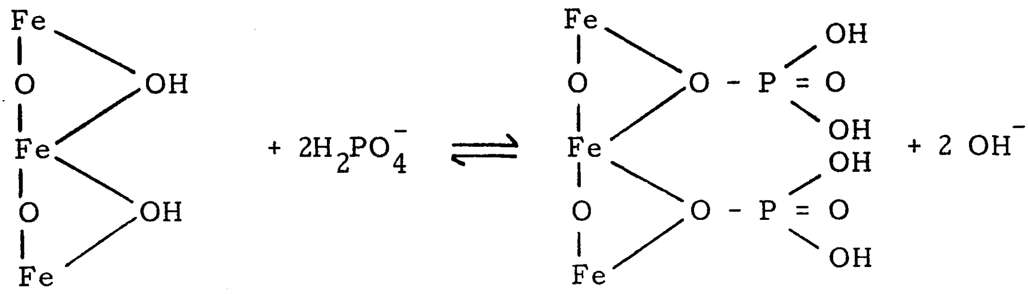
Monocalcium phosphate	$Ca(H_2PO_4)_2$
Dicalcium phosphate	$CaHPO_4$
Tricalcium phosphate	$Ca_3(PO_4)_2$
Octacalcium phosphate	$Ca_4H(PO_4)_3$
Hydroxyapatite	$Ca_{10}(PO_4)_6(OH)_2$
Fluorapatite	$Ca_{10}(PO_4)_6F_2$
Strengite	$FePO_4.2H_2O$
Variscite	$A\ell PO_4.2H_2O$

The simpler calcium phosphate compounds such as the mono- and di-calcium phosphates are the most soluble of these compounds and therefore the most readily available to plants. However, except in recently fertilized soils, these compounds are present in extremely small amounts since they are easily converted to the more insoluble calcium forms. Iron and aluminium phosphates in soil exhibit maximum stability under acid conditions, and since Scottish colliery spoil is almost invariably acid it can be assumed that these compounds contribute little phosphorus to plants. In order to establish a plant cover, therefore, phosphorus must be added to the spoil. However, addition of phosphorus in a readily available form does not overcome the problem, since reaction of the added phosphorus with spoil greatly reduces the amount which is available for plant uptake.

In acid mineral soils iron and aluminium ions present in solution react with the soluble anionic forms of phosphate, resulting in the formation of insoluble hydroxy phosphates. In most strongly acid soils and in colliery spoil, the concentration of iron and aluminium ions greatly exceeds that of $H_2PO_4^-$ and so the reaction moves in favour of the formation of the stable hydroxy phosphates, which are more insoluble under acid conditions. In order to reduce the loss of phosphorus in this way, lime may be added to raise the pH and thereby increase the solubility of the iron and aluminium phosphates and also reduce the concentration of iron and aluminium ions in solution. Liming, however, increases phosphorus loss through reaction with calcium ions leading to the formation of calcium phosphates which are insoluble under alkaline conditions (Thompson and Troeh 1973, Holford and Mattingly 1975). Spoil receiving high rates of lime may in the short term behave as a calcareous soil, as the liming material dissolves giving raised calcium ion concentration in the soil solution.

② Adsorbed phosphorus $\rightleftharpoons$ Dissolved phosphorus

Phosphate ions not only react with soluble iron and aluminium but also with their insoluble hydrous oxides (Barrow 1984). Active iron and aluminium may be present as surface coatings of their oxides or hydroxides on clay surfaces, and since all anionic forms of phosphates are able to form these compounds, fixation by this mechanism takes place over a wide pH range. Hsu (1964) and Van Riemsdijk and Lyklema (1980) working with sandy soils containing iron and aluminium oxides/hydroxides identified the formation of a metal-phosphate coating on the oxide particle by a ligand exchange reaction with surface -OH groups:



Phosphate adsorption on hydrous oxide surfaces has been shown to be a major factor in the loss of phosphorus in colliery spoil (Doubleday 1972, Pulford and Duncan 1975, Pulford 1976).

Soil phosphorus reactions have been studied with the aid of radioactive ^{32}P, which allows the tracing of phosphate added to a soil and the location of its uptake or fixation (Williams and Knight 1963). Studies in soil phosphate adsorption from the soil solution show a two-stage reaction; an initial fast reaction (1-2 hrs) due to an exchange reaction at the oxide/hydroxide surface, and a slower second stage reaction due possibly to exchange with inaccessible sites within micropores or crystal defects (Arambarri and Talibudeen 1959, Bolan et al 1985), through isomorphous substitution in the crystal lattice, or with freshly formed surface active components formed during ageing (Hsu 1964).

Phosphate adsorption also occurs on positively charged clay mineral surfaces, where it tends to be held too tightly to be exchangeable at low pH - raising the pH by liming increases the level of extractable phosphate due to ion exchange with carbonate and bicarbonate ions (Talibudeen 1957).

③ Organic phosphorus $\rightleftharpoons$ Dissolved phosphorus

Organic phosphorus compounds in the soil are not directly available to plants but are tied up in the structure of organic compounds of plant and animal residues. The largest group of organic phosphorus compounds is the inositol phosphates, which are basically sugar molecules with one or more -OH groups replaced by $-H_2PO_4$ groups. Such compounds contain approximately 1/3 of the organic phosphorus in soils, with nucleic acids and their decomposition products containing about 10% and phospholipids about 1%. Enzymic breakdown of these compounds releases phosphate occurring in reactive positions to the soil solution. However, much of the phosphorus is built into the structure and is only released in the later stages of decomposition.

Mechanisms by which organic phosphorus compounds are held in the soil include adsorption through orthophosphate groups to iron and aluminium oxides by mechanisms similar to those for inorganic phosphates, and sorption by interaction of the organic portion of the phosphate ester with inorganic soil components e.g. clay minerals.

Colliery spoil, low in decomposable organic matter, will not contain a significant organic phosphorus fraction. However, amendment at reclamation with fertilizer and manure may result in some of the available fertilizer phosphorus being taken up in an organic form (Donald and Williams 1954) thus reducing its plant availability until microbial breakdown releases it once more to solution. Liming, however, facilitates the release of phosphorus from the organic fraction, as the organic-phosphate bond is less stable under alkaline conditions and also microbial activity is increased by liming, therefore lime + fertilizer +

organic matter additions combine to increase the spoil phosphorus status.

3.3 Problems of Low Phosphorus Availability and High Fixing Capacity of Colliery Spoil

Phosphate adsorption, and its removal from the available pool, is a problem which has long been recognised in soil and spoil. Williams et al. (1958) and Saunders (1965) found phosphate adsorption to be significantly correlated with the level of aluminium extracted by Tamm's acid oxalate in Scottish and New Zealand soils respectively, thus concluding that the amorphous aluminium fraction was responsible for phosphate adsorption, while Doubleday (1971, 1972) and Pulford and Duncan (1975) reported a correlation with the amorphous iron oxide fraction in coal waste.

3.4 Experimental Methods

The level of extractable phosphorus and the capacity to adsorb added phosphate were determined in samples of colliery spoil from six unreclaimed sites (Fig. 3.2) in order to study the relationship between these factors and the levels of iron, aluminium and manganese extracted by Tamm's acid oxalate and spoil pH, and the effects of pH, liming and addition of available phosphorus were measured by pot experiments under controlled conditions.

3.4.1 Measurement of spoil pH

Method

A 10g sample of air-dry < 2 m.m. spoil was weighed into a shaking bottle to which 25 ml de-ionized water was added. After shaking on a wrist shaker for 20 mins. the pH of the suspension was measured.

3.4.2 "Available phosphorus"

The choice of extracting method was based on the following criteria:

(a) The amount of phosphorus extracted should correlate with plant uptake and yield response to added phosphorus.

(b) There should be a minimum of secondary precipitation and adsorption of extracted phosphorus.

(c) The method should be adaptable to rapid, routine analysis.

(c) The method adopted should be applicable to the specific type of sample under study.

Commonly used extractants to measure available phosphorus include:

(a) 0.5M sodium bicarbonate (pH 8.5) (Olsen et al. 1954), used by ADAS in soils of England and Wales.

(b) 0.5M acetic acid (Williams and Stewart 1941) used by the Advisory Service in Scotland.

(c) 1% citric acid (Nagarajah et al. 1968).

(d) 0.03M NH_4F + 0.025M $HC\ell$ (Bray and Kurtz 1945).

(e) Isotopically exchangeable phosphorus using radioactive ^{32}P (Larsen 1952).

(f) Anion exchange resin (Cooke and Hislop 1963).

Extraction of colliery spoil with 0.5M $NaHCO_3$ and dilute citric acid gives coloured extracts of varying intensity due to extraction of organic matter and iron respectively, and involves a clean-up step with charcoal or other adsorbent to remove the colour component before colorimetric determination of phosphorus. In this respect it was difficult to find a suitable adsorbent which did not itself contain phosphorus, and therefore further steps were required to first clean the adsorbent material; for this reason these methods were not used for spoil phosphorus extraction. Work carried out in the Agricultural Chemistry Department, Glasgow University (Khalil 1981) showed that phosphorus extracted from soils using 0.5M acetic acid, acid ammonium fluoride and anion exchange resin correlated significantly with plant uptake. Acetic acid, however, removed very little phosphorus (compared to other methods), while anion exchange methods require a 24 hr extraction time. For these reasons the method chosen for the analysis of a large number of samples was extraction with acid-NH_4F, which has a short extraction time, good correlation with plant uptake (Williams and Knight 1963, Khalil 1981) and is suitable for acid spoil. The combination of $HC\ell$ and NH_4F is designed to remove acid-soluble forms of phosphorus (largely calcium phosphates) and the NH_4F dissolves iron and aluminium phosphates by complex formation with these ions in acid solution.

Reagents

1M Ammonium fluoride - Dissolve 37g Analar NH_4F in de-ionized water and dilute to 1 litre (store in polyethylene bottle).

0.5M Hydrochloric acid - Dilute 40.4 ml Analar $HC\ell$ to 1 litre with de-ionized water.

Extracting solution - Add 15 ml 1M NH_4F and 25 ml 0.5M $HC\ell$ to 460 ml de-ionized water. This gives a solution 0.03M in NH_4F and 0.025M in $HC\ell$.

Stannous chloride (stock) - Dissolve 10g Analar $SnC\ell_2.2H_2O$ in 25 ml Analar $HC\ell$. Store in a brown, glass-stoppered bottle in a fridge and prepare fresh every 6 weeks.

Stannous chloride (working solution) - Mix 1 ml $SnC\ell_2$ stock with 333 ml de-ionized water. Make fresh every 2 hrs.

Ammonium molybdate - Dissolve 15g Analar $(NH_4)_6Mo_7O_{24}$ in 350 ml de-ionized water. Add 350 ml 10M $HC\ell$ slowly with stirring. Cool and make to 1 litre. Store in a brown, glass-stoppered bottle. Prepare fresh every 2 months.

Method

Approximately 1g of air-dry < 2 m.m. spoil was accurately weighed into a shaking bottle. 7 ml of extracting solution was added and shaken by hand for 1 min. The extract was filtered through a Whatman's No. 42 filter paper. To 2 ml of filtrate were added, in order, 5 ml de-ionized water, 2 ml ammonium molybdate solution and 1 ml $SnC\ell_2$ working solution. After mixing, the colour was measured photometrically at 660 n.m. after exactly 10 mins. The concentration of phosphorus was calculated with reference to a standard graph of suitable range.

3.4.3 Phosphate adsorption

The phosphate adsorption capacity of the spoil was determined following the method of Pulford and Duncan (1975).

Reagents

Buffered phosphorus working solution - Dissolve 272g Analar $NaCH_3CO_2.3H_2O$ in approximately 1ℓ de-ionized water. Add 0.312g $NaH_2PO_4.2H_2O$ + 120 ml glacial CH_3CO_2H and make to 2ℓ. This gives a solution of 30 μ g P/ml buffered at pH 4.6.

3M Sulphuric acid - Dilute 83.3 ml Analar H_2SO_4 to 1ℓ with de-ionized water.

Ammonium molybdate - Dissolve 5g Analar $(NH_4)_6Mo_7O_{24}.4H_2O$ in de-ionized water and make to 250 ml.

Ascorbic acid - Dissolve 4.4g Analar $C_6H_8O_6$ in de-ionized water and make to 250 ml. This solution deteriorates slowly on standing and must be prepared fresh as required.

Reducing solution - Dilute a mixture of 125 ml 3M H_2SO_4, 38 ml ammonium molybdate solution and 60 ml ascorbic acid solution to 250 ml. This solution should be faint-green in colour and should be prepared immediately before use.

Method

Approximately 5g of air-dry < 2 m.m. spoil was shaken with 25 ml of 30 μ gP/ml buffered phosphorus solution on a reciprocating shaker for 2 hrs. The extract was filtered through a Whatman's No. 42 filter paper. To 5 ml filtrate was added 20 ml reducing solution and the volume made to 50 ml with de-ionized water. After standing overnight the colour was measured photometrically at 660 n.m. The amount of phosphorus retained by the spoil was calculated by difference.

3.4.4 Amorphous iron, aluminium and manganese

The amorphous oxide fraction was extracted with acid ammonium oxalate (Tamm's reagent).

Reagents

Ammonium oxalate - $(CO_2NH_4)_2.H_2O$

Oxalic acid - $(CO_2H)_2.2H_2O$

Extracting solution - Dissolve 24.9g Analar ammonium oxalate + 12.6g Analar oxalic acid in de-ionized water and make to 1ℓ; this gives a solution 0.175M in ammonium oxalate and 0.1M in oxalic acid at pH 3.3.

Method

Approximately 5g of air-dry < 2 m.m. spoil was shaken with 50 ml extracting solution on a reciprocating shaker for 2 hrs. After filtering through a Whatman's filter paper the concentration of Fe, Aℓ and Mn was measured on a Perkin Elmer model 370A.

3.5 Selection of Samples

86 samples of unburnt colliery spoil covering the pH range 2.9-8.4 from six sites in the Central Scottish coal belt were analysed for pH, Tamm Fe, Aℓ, Mn, NH_4F-extractable phosphorus and phosphate adsorption. The sites chosen for analysis are all located in West and Central Scotland (Fig. 3.2), Their choice was based on extensive background work carried out in the Agricultural Chemistry Department, Glasgow University, and represent a range of "typical" colliery spoil bings of this area. Through experience in this Department it has been found that spoils of the West of Scotland contain higher levels of iron pyrites compared to those of the East coast and, therefore, the sites chosen presented the spoil conditions most limiting to plant growth.

I am indebted to Dr. A.J. Kimber for his permission to use his results for some of the Tamm extractable Fe, Aℓ, Mn data presented here.

3.6 Results and Discussion

A complete table of results is presented in Appendix I.

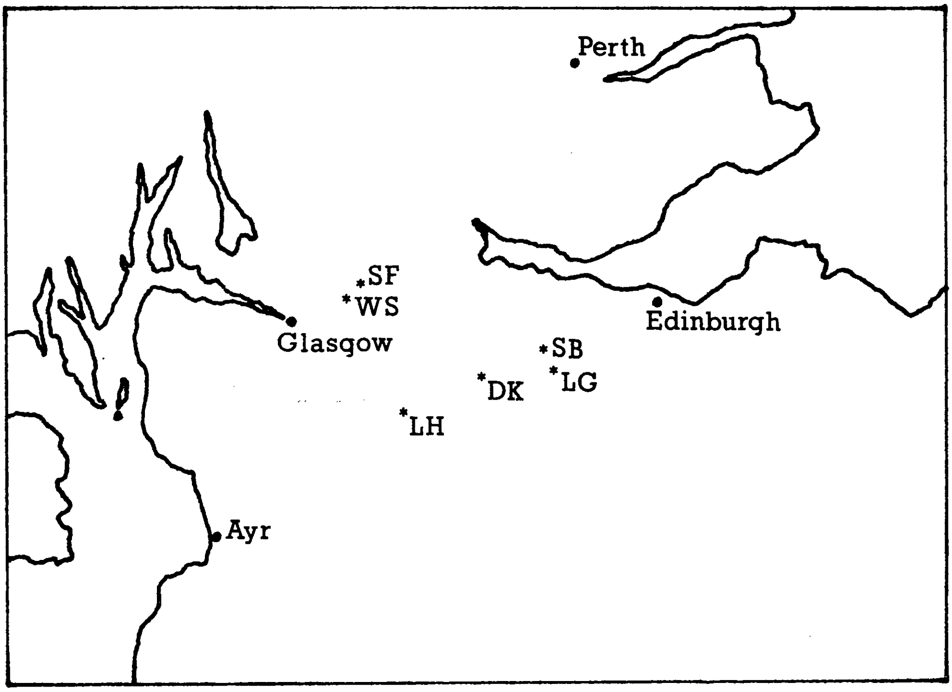
Table 3.1 shows the mean and range of values for each parameter for each site and for all sites combined.

Table 3.2 shows the correlation coefficient and significance of the relationships between pH, Tamm Fe, Aℓ, Mn, NH_4F-extractable phosphorus and phosphate adsorption over all sites.

Table 3.1. Ammonium oxalate-extractable Fe, Aℓ and Mn, NH_4F-extractabale P and P-adsorption of colliery spoil (range and mean values)

SITE	pH	Ammonium oxalate extractable (µg/g) Fe	Aℓ	Mn	Extractable-P (µg/g)	P-adsorption (mg/100g)
SAINT FLANNANS (SF)	3.5 - 8.4 (5.5)	1560-6150 (3862)	151-900 (574)	5-512 (149)	1.1 - 10.3 (4.4)	6.3 - 13.4 (8.7)
WATERSIDE (WS)	4.1 - 6.7 (5.1)	1950-5590 (3716)	230-723 (441)	15-134 (66)	4.5 - 17.2 (12.7)	5.0 - 8.6 (7.3)
LOGANLEE (LG)	2.9 - 3.9 (3.4)	2970-9230 (4990)	75-1840 (945)	1-8 (4)	0.1 - 5.4 (3.1)	10.8 - 13.9 (12.2)
LARKHALL (LH)	3.2 - 5.5 (3,7)	1040-5700 (3344)	380-1650 (762)	3-225 (39)	n.d.-12.2 (3.0)	5.1 - 13.4 (11,9)
STONEYBURN (SB)	3,0 - 5.6 (4.4)	1450-17500 (6148)	180-1930 (694)	3-151 (39)	0.9 -19.3 (8.2)	4.0 - 14.6 (10.3)
DYKEHEAD (DK)	4.5 - 7.6 (5.9)	900-5650 (2368)	151-340 (255)	59-179 (117)	0,5 - 10.8 (5.4)	4.1 - 6.8 (5.8)
ALL SITES	2.9 - 8.4 (4,7)	900-17500 (4380)	75-1930 (620)	1-512 (75)	n.d.-19.3 (6.2)	4.0 - 14.6 (9.4)

Fig. 3.2. Location of sites chosen for spoil analysis



SF = SAINT FLANNANS - Grid reference NS 688749

WS = WATERSIDE - " " NS 686731

LG = LOGANLEE - " " NS 976622

LH = LARKHALL - " " NS 762523

SB = STONEYBURN - " " NS 977634

DK = DYKEHEAD - " " NS 868608

Table 3.2. Correlations between NH_4F-extractable P, P-adsorption and pH, oxalate extractable Fe, Aℓ and Mn

	NH_4F-extractable P	P-adsorption
pH	0.224^{*}	-0.739^{***}
Fe	-0.293^{**}	0.558^{***}
Aℓ	-0.039	0.448^{***}
Mn	0.064	-0.408^{***}

Since pH and P-adsorption are closely correlated, this was taken into account in analysing the relationships between P-adsorption and oxalate extractable metal levels. The resulting partial correlation coefficients are 0.346^{**}, 0.244^{*} and 0.05 for P-adsorption vs. oxalate Fe, Aℓ and Mn respectively, showing that the P-adsorption vs. Mn correlation is simply a reflection of the P-adsorption vs. pH relationship. The formula used to calculate partial correlation data is as follows:

$$P_{12/3} = \frac{P_{12}-(P_{13}xP_{23})}{[(1-P_{13}^{2})(1-P_{23}^{2})]^{\frac{1}{2}}}$$

where P_{12} = correlation coefficient between parameters 1 and 2

P_{13} = correlation coefficient between parameters 1 and 3

P_{23} = correlation coefficient between parameters 2 and 3

$P_{12/3}$ = partial correlation coefficient between parameters 1 and 2 taking into account parameter 3.

P-adsorption is found to be related inversely to pH in agreement with soil analysis of Beauchamp et al. (1976), Parfitt (1978), Haynes (1982) and spoil analysis, Pulford and Duncan (1975). Precipitation of insoluble iron and aluminium hydrous oxides, which have a positive surface charge under acid conditions, increases the phosphate adsorption capacity of the spoil, while at higher pH anion exchange of OH^- for $H_2PO_4^-$ occurs, thus reducing the amount of phosphate adsorbed (Talibudeen 1981).

Figs. 3.3-3.10 show the relationships between the data in Appendix I for all 86 spoil samples.

Table 3.2 shows that both the amorphous Fe and Aℓ fractions adsorb phosphate over the pH range 2.9-8.4 while there is no indication that either fraction holds "native" phosphate in a form extractable by acid NH_4F, although the results show that the extractability of the adsorbed phosphate decreases due to the greater energy of adsorption at lower pH.

By splitting the data into two groups, pH $<$ 4.0 (39 samples) and pH $>$ 4.0 (47 samples) a better understanding of the phosphate reactions in spoil is seen. Tables 3.3 and 3.4 show the correlation coefficients between pH, oxalate-extractable Fe and Aℓ, NH_4F extractable phosphorus and phosphate adsorption for samples with pH $<$ 4.0 and pH $>$ 4.0, respectively.

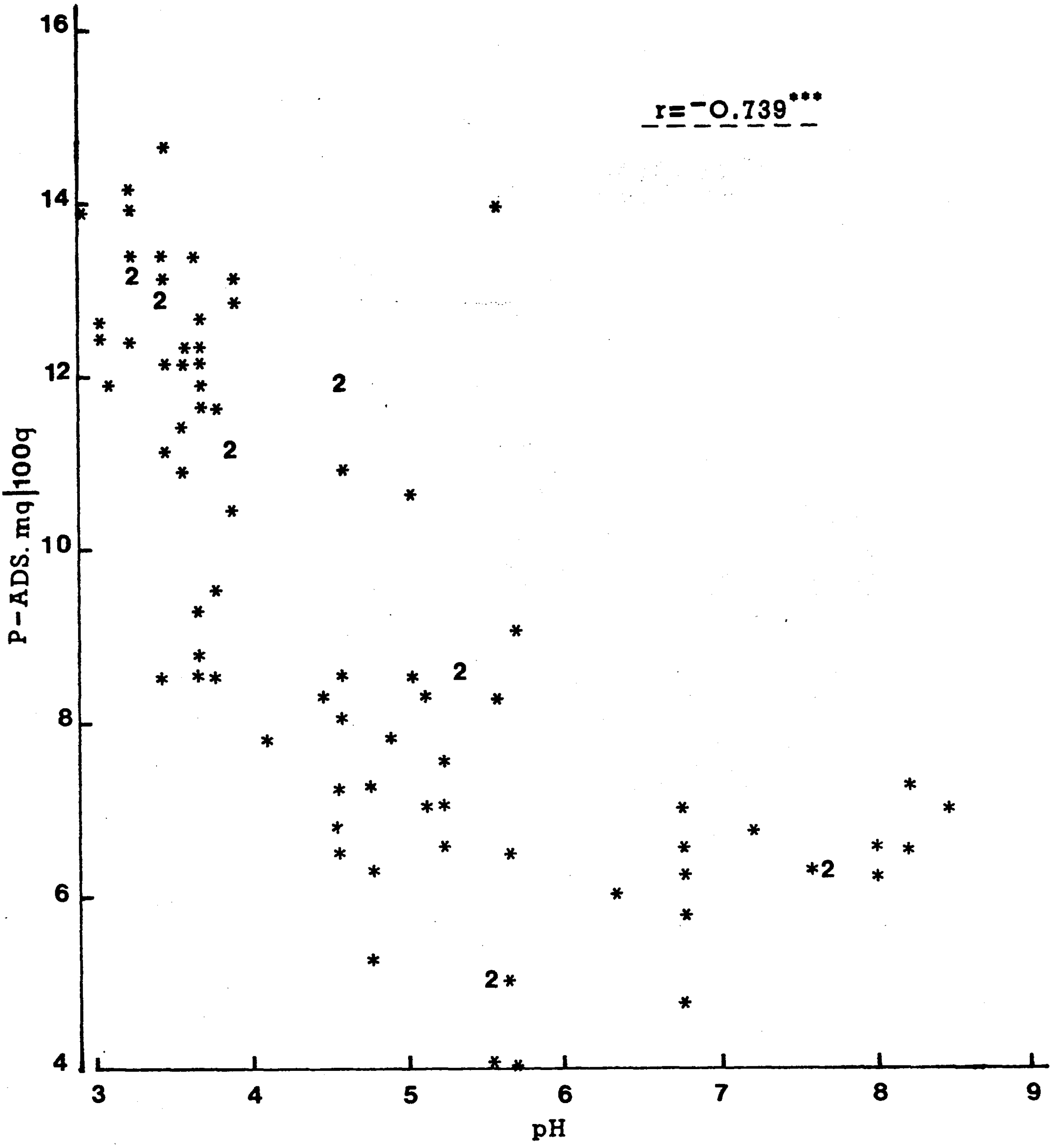


Figure 3.3. Relationship between spoil pH and phosphate adsorption.

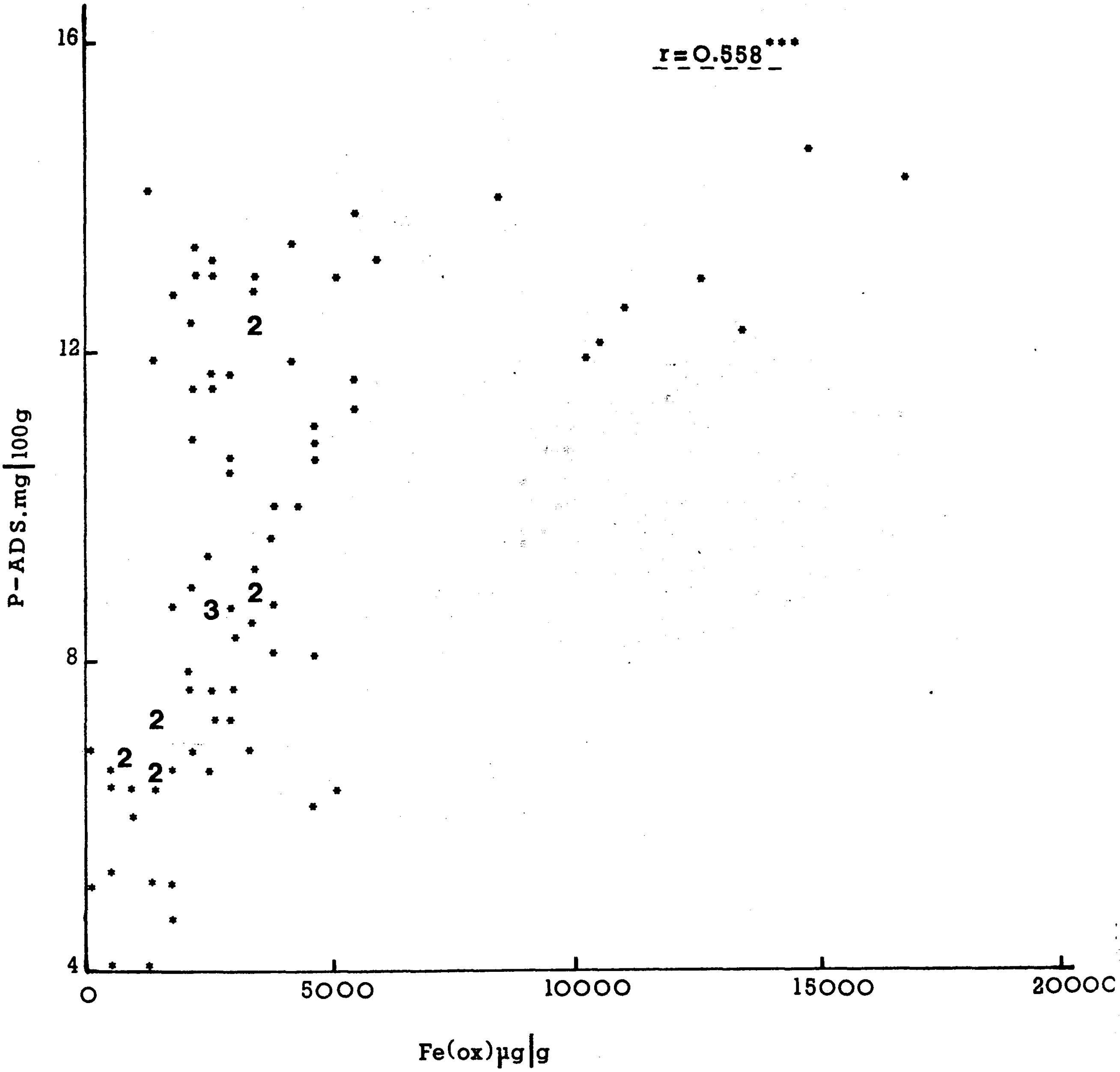


Figure 3.4. Relationship between oxalate extractable iron and phosphate adsorption.

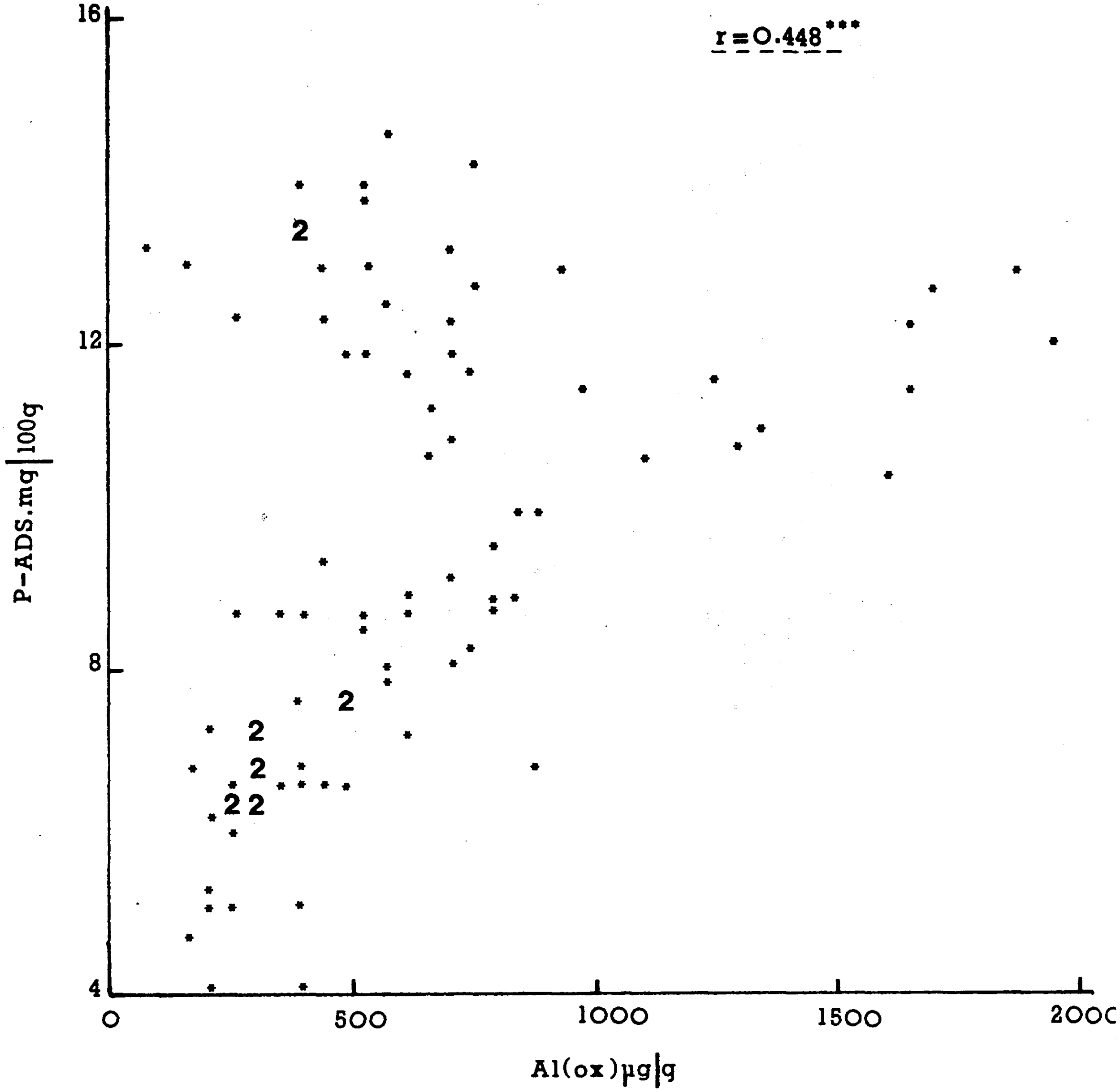


Figure 3.5. Relationship between oxalate extractable aluminium and phosphate adsorption.

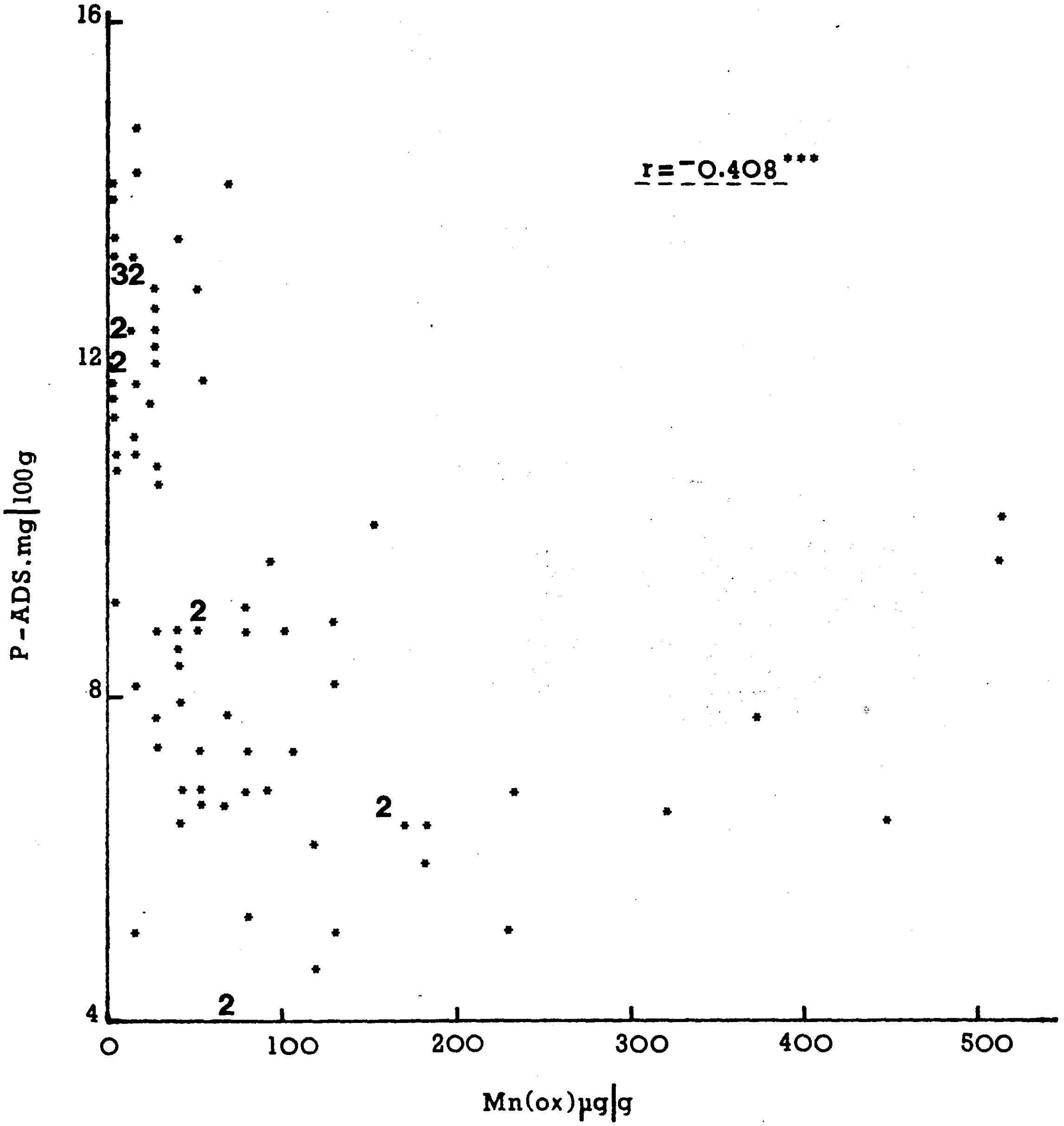


Figure 3.6. Relationship between oxalate extractable manganese and phosphate adsorption.

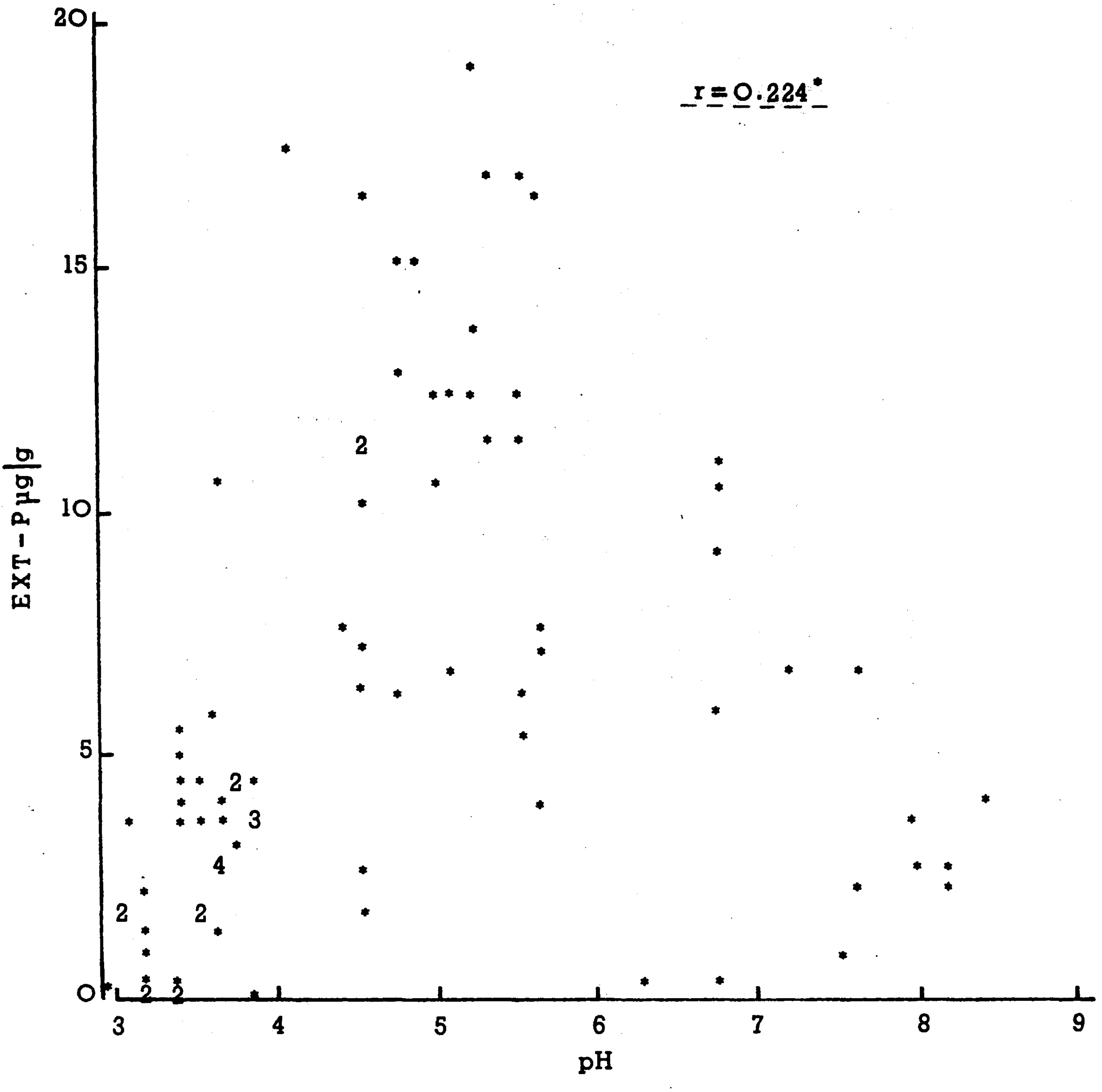


Figure 3.7. Relationship between spoil pH and NH_4F extractable phosphorus.

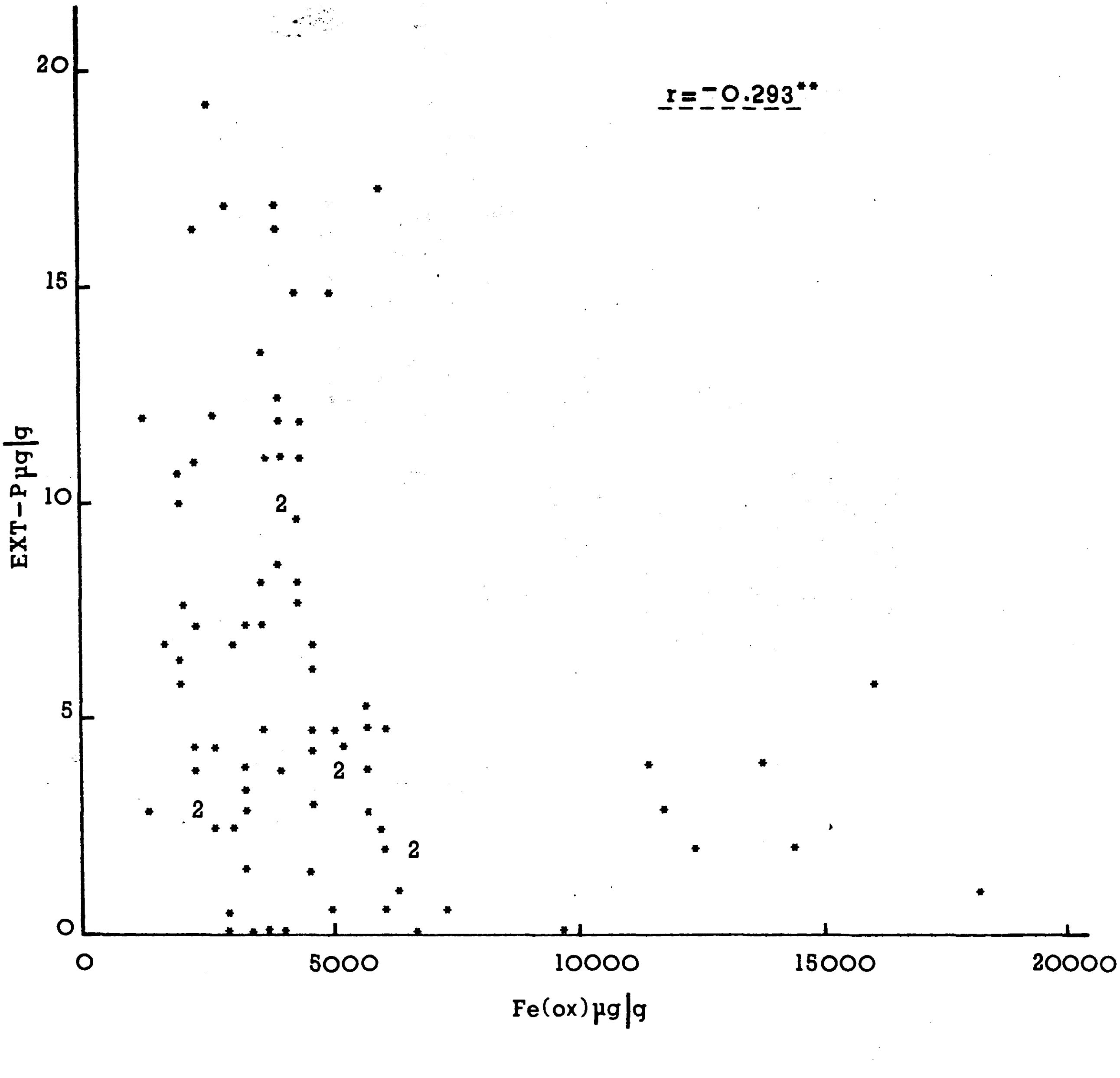


Figure 3.8. Relationship between oxalate extractable iron and NH_4F extractable phosphorus.

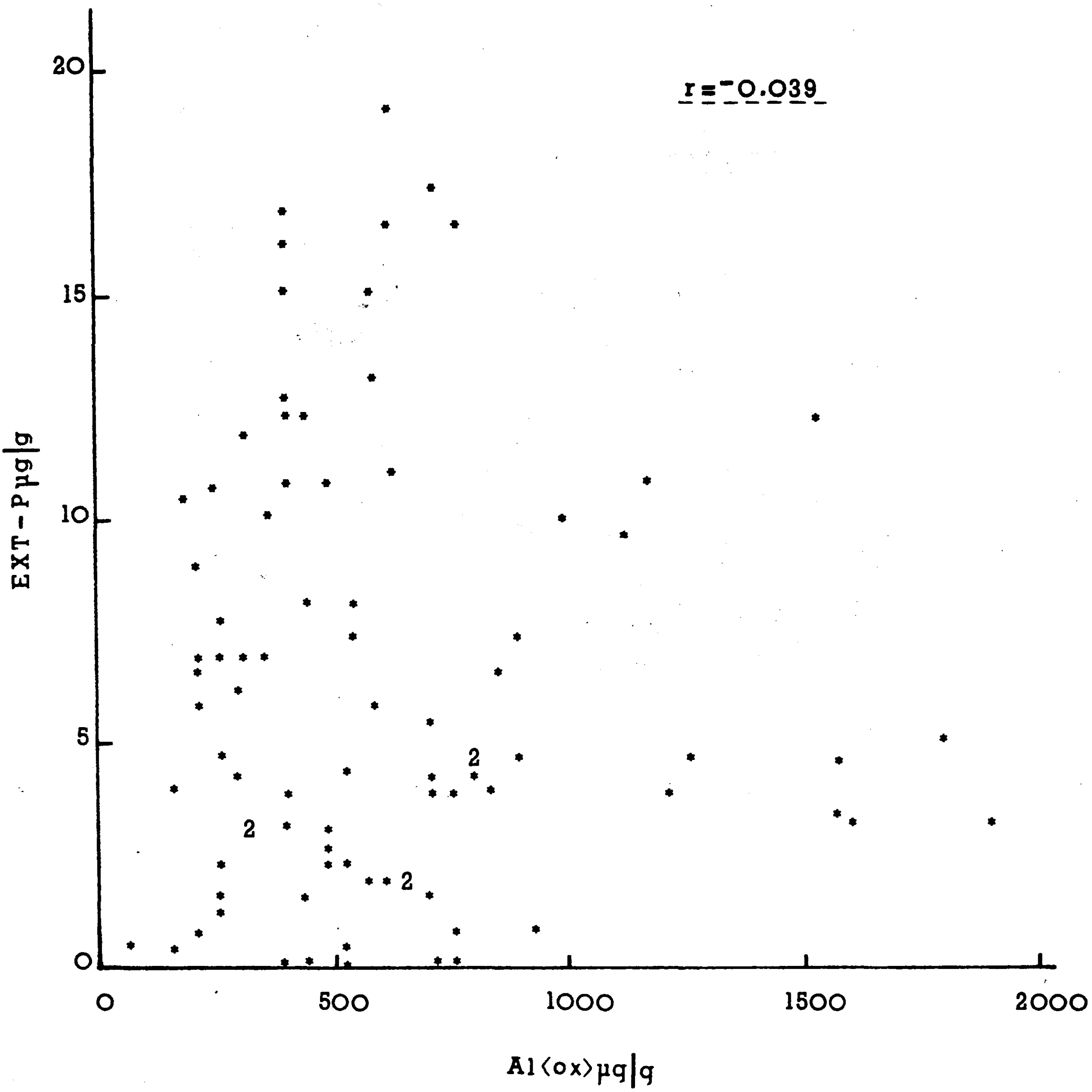


Figure 3.9 Relationship between oxalate extractable aluminium and NH_4F extractable phosphorus.

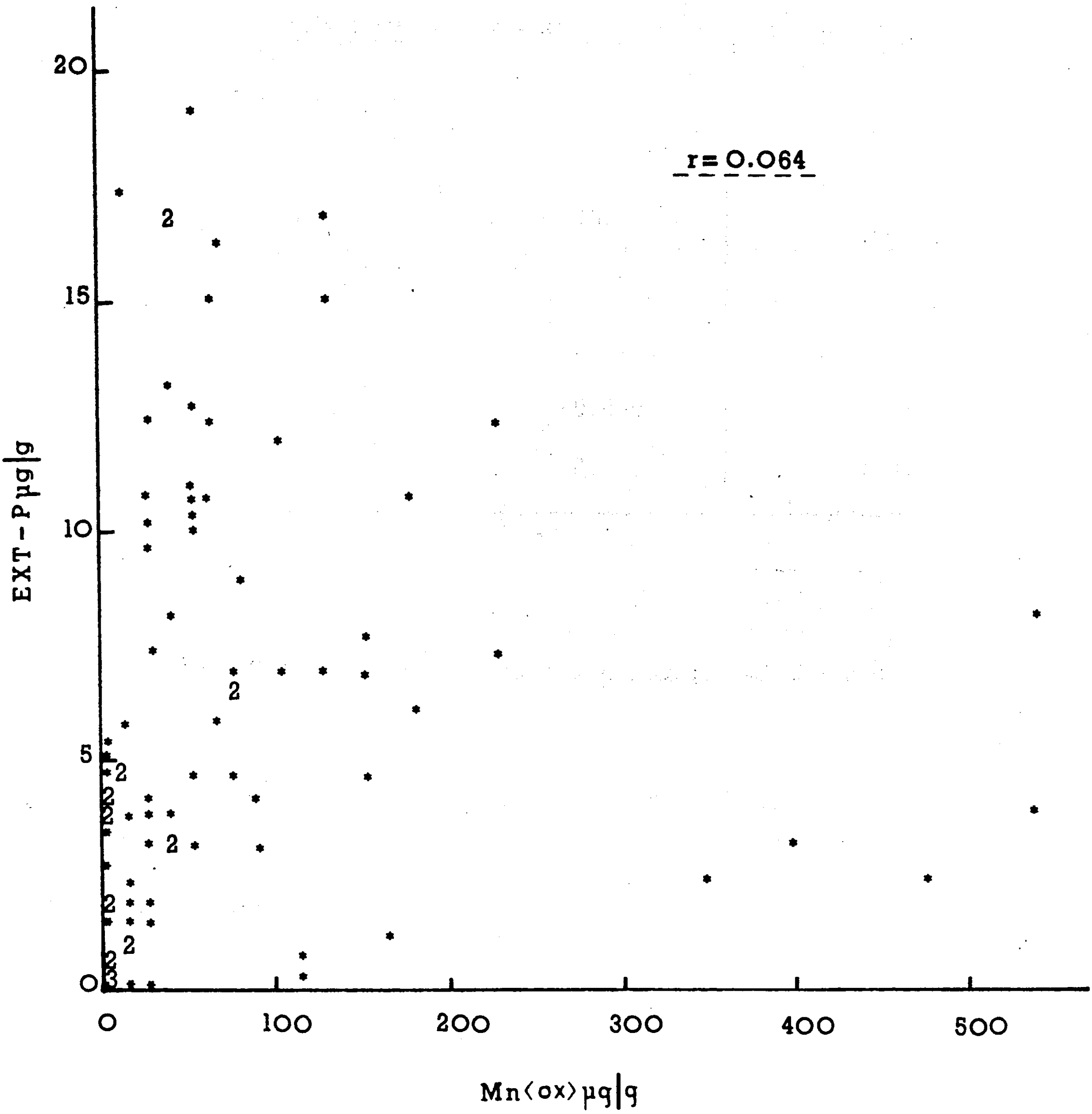


Figure 3.10. Relationship between oxalate extractable manganese and NH_4F extractable phosphorus.

Table 3.3. Correlation coefficient and significance of the relationships between pH, oxalate-extractable Fe, and Aℓ, NH_4F-extractable phosphorus and phosphate adsorption for samples with pH < 4.0

	pH	Fe	Aℓ
Fe	-0.323^{*}		
Aℓ	0.416^{**}	-0.179	
NH_4F-P	0.452^{**}	-0.060	0.367^{*}
P-ads.	-0.478^{**}	0.378^{*}	-0.184

Table 3.4. Correlation coefficient and significance of the relationships between pH, oxalate-extractable Fe, and Aℓ, NH_4F extractable phosphorus and phosphate adsorption for samples with pH > 4.0

	pH	Fe	Aℓ
Fe	-0.345^{*}		
Aℓ	-0.322^{*}	0.318^{*}	
NH_4F-P	-0.543^{***}	0.060	0.357^{*}
P-ads.	-0.360^{*}	0.412^{**}	0.589^{***}

Table 3.3 shows that for those samples with pH < 4.0 the amorphous Fe fraction is the active component involved in phosphate adsorption reactions, and that there is no contribution from the Aℓ fraction. Aluminium hydrous oxides enter solution as the pH falls below 4.0 (Kittrick and Jackson 1955), as shown by the positive correlation between pH and oxalate-extractable Aℓ (Table 3.3), and therefore there is a reduced effect of the Aℓ fraction towards phosphate adsorption as the pH falls below this value. The Fe hydrous oxides remain insoluble down to pH 2.7-3.0 and, as shown in Table 3.3, continue to adsorb phosphate below pH 4.0. At pH > 4.0 there is a significant relationship between both the amorphous Fe and Aℓ fractions and phosphate adsorption indicating that adsorption occurs on both fractions in the insoluble form.

For both sets of results (Tables 3.3 and 3.4) the level of NH_4F-extractable phosphate is correlated significantly with the hydrous Aℓ fraction only, indicating that it is this fraction which holds available or exchangeable phosphate in colliery spoil, with no contribution from the hydrous Fe fraction.

3.7 The Effects of Lime and Phosphorus Addition on Colliery Spoil Properties and Plant Growth (Pot Experiment)

The effects of lime and phosphorus addition on plant growth were determined by pot experiment using spoil from three of the sites discussed in previous sections:

Waterside - acid spoil with medium-high levels of available phosphorus.

Loganlee - most acid spoil with high levels of Tamm-extractable aluminium, high phosphate adsorption capacity and low available phosphorus.

Dykehead - least acid spoil with low levels of Tamm-extractable iron and aluminium and low phosphate adsorption capacity.

The experiment was carried out in a growth room maintained at 20°C with 16 hr day length and the pots were watered with deionized water three times per week.

3.8 Materials and Methods

3.8.1 Spoil properties

Table 3.5 shows the pH, % iron pyrites and particle size distribution of the spoils used in the pot experiment.

Table 3.5. Analysis of spoil for pot experiment

SITE	pH	% iron pyrites	Particle size distribution (% by weight)		
			<2mm	2mm-1cm	>1cm
WATERSIDE	4.0	0.19	17	72	11
LOGANLEE	3.7	0.10	48	44	8
DYKEHEAD	7.0	0.14	22	60	18

The samples were sieved to pass a 1 cm grid and the material > 1 cm discarded. As the particle size distribution of the < 1 cm fraction shows, Waterside and Dykehead spoils have a coarser, free-draining texture, while Loganlee has a heavy texture.

3.8.2 Liming rates

Two lime rates were used with each spoil, the lower rate representing the amount of lime required to raise the pH of the spoil to pH 6.2 (the optimum for a grass sward) and a higher rate which included an allowance for the pyrite content, being equivalent to the addition of 4 tonnes $CaCO_3$/ha for every 0.1% pyrite (for lime requirement determination see Chapter 2). Table 3.6 shows the lime requirements of each spoil. Loganlee spoil (pH 3.7) has a higher lime requirement (low rate) than Waterside (pH 4.0) due to the greater proportion of < 2 mm material, giving a greater pH buffering capacity.

Table 3.6. Liming rates for Waterside, Loganlee and Dykehead spoils for pot experiment

	$Ca(OH)_2$ tonnes/ha	
	Low Rate	High Rate
WATERSIDE	2.27	8.02
LOGANLEE	7.64	10.67
DYKEHEAD	Nil	4.27

Each treatment was carried out in triplicate and compared to control pots receiving N, P and K fertilizer only.

3.8.3 Fertilizer treatments

The levels of nitrogen and potassium added to each pot were the same for each lime and phosphate treatment, being equivalent to the addition of 60kg N/ha and 30kg K/ha. Nitrogen was added as $NaNO_3$ and potassium as $KC\ell$ (Analar grades) each added in solution to the spoil (after liming, where required) in order to give an even distribution through the sample. Two rates of phosphorus addition were compared, representing the addition of 20 and 40 kg P/ha added as $CaH(PO_4)_2.H_2O$ (Analar grade) in solution. Each phosphorus treatment at each rate was carried out in triplicate and compared to control pots receiving lime, N and K only.

3.8.4 Seeding rate

Perennial ryegrass (Lolium perenne) was chosen because it is widely used in reclamation schemes, as it has a wide tolerance to spoil conditions, has a quick emergence and gives a leafy growth. This was sown at a rate of 55 seeds/pot (10 cm diameter), equivalent to 100kg/ha.

WS and LG spoil received 9 treatments (Table 3.7)

Since there is only one level of lime added to Dykehead spoil, this material received only 6 treatments. T1, T2 and T3 in Table 3.7 represent the addition of lime to acid spoil. However, since Dykehead already has an initial pH of 7.0 these three treatments do not apply to this site.

Table 3.7. List of treatments for Waterside and Loganlee spoils

Treatment No.	Treatment
T_1	No lime , no P
T_2	" " , low P
T_3	" " , high P
T_4	Low lime , no P
T_5	" " , low P
T_6	" " , high P
T_7	High lime, no P
T_8	" " , low P
T_9	" " , high P

3.9 RESULTS AND DISCUSSION

3.9.1 Effect of lime and phosphorus addition on seed germination

At germination it was noted that there was a difference in the number of seeds germinating between spoils, and between treatments on each spoil. Table 3.8 shows the number of germinating seeds from each treatment.

As Table 3.8 shows, there is no significant effect of phosphorus addition on germination due to the fact that the seed contains sufficient nutrient reserves to meet demands at germination.

The effect of lime is most marked for LG spoil which shows that there is inhibition to seed germination in the unamended spoil. Kimber (1982) made an extensive study of each of the three sites used in this experiment - see Table 3.9.

Inhibition to seed germination in LG spoil may be the result of higher levels of available Fe and Aℓ, however, these factors require more intensive study.

3.9.2 Effect of lime and phosphorus addition on total plant yield

The grass from each pot was harvested after 46 days (leaving a 1 cm stubble) and again after 111 days. Table 3.8 shows the average yield of replicate treatments at both harvests and the average combined yield, the combined yields shown in Fig. 3.11. Plates 4, 5 and 6 show the pots after 32 days.

The effect of lime added at the lower rate to acid spoil was to increase yield at each level of phosphorus addition, the most dramatic result shown for Loganlee spoil which showed virtually no growth without

Table 3.8. Germination count and yield from two harvests

TREATMENT	Germination(seeds/pot)	1st Yield(mgDM/pot)	2nd Yield(mgDM/pot)	Total yield(mgDM/pot)
WS 1	27	148	150	298
2	30	199	225	424
3	18	108	142	250
4	29	144	180	324
5	27	284	261	545
6	30	332	435	767
7	32	176	147	323
8	34	220	321	541
9	41	306	270	576
LG 1	1	6	n.d.*	6
2	1	6	n.d.	6
3	2	8	n.d.	8
4	27	74	19	93
5	39	190	228	418
6	37	235	354	589
7	31	64	28	92
8	36	87	117	204
9	33	133	159	292
DK 4	35	114	60	174
5	35	306	278	584
6	37	327	338	665
7	34	115	17	132
8	26	165	138	303
9	20	105	117	222

* n.d. = No re-growth after 1st harvest.

NOTE: In this and following Tables, only the average result of the triplicate treatments is given for clarity and ease of presentation of data.

Table 3.9. Levels of acetic-extractable iron and aluminium in WS, LG and DK spoils (selected data from Kimber 1982)

SITE	Acetic extractable µg/g (range & mean)	
	Fe	Aℓ
WATERSIDE	14-143 (61)	32-387 (102)
LOGANLEE	7-372 (112)	54-371 (183)
DYKEHEAD	9-129 (32)	8-67 (35)

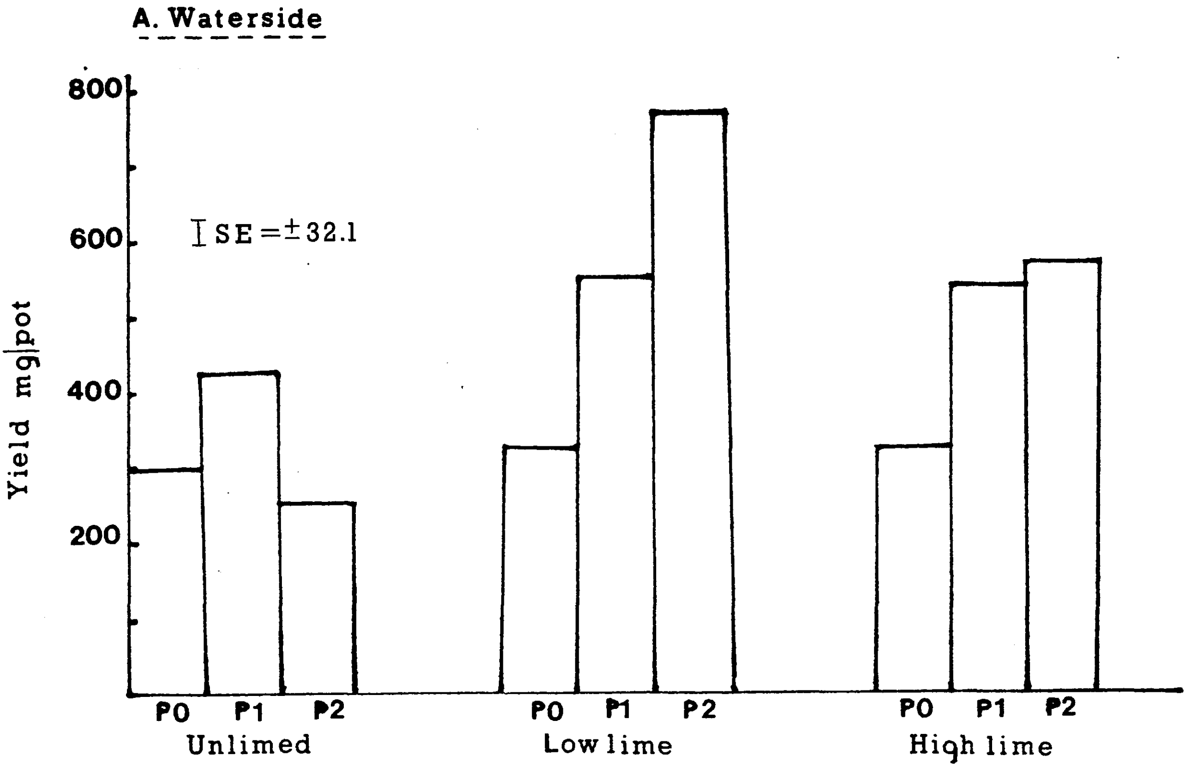


Figure 3.11a. Total yield of perennial ryegrass in pot experiment.

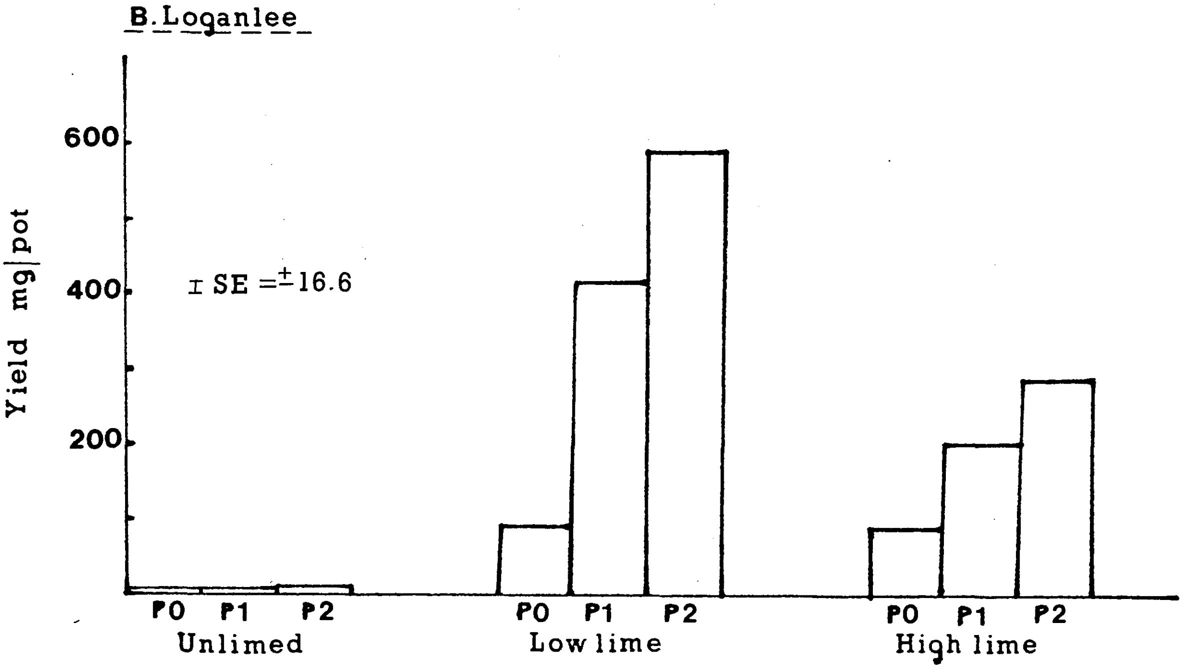


Figure 3.11b. Total yield of perennial ryegrass in pot experiment.

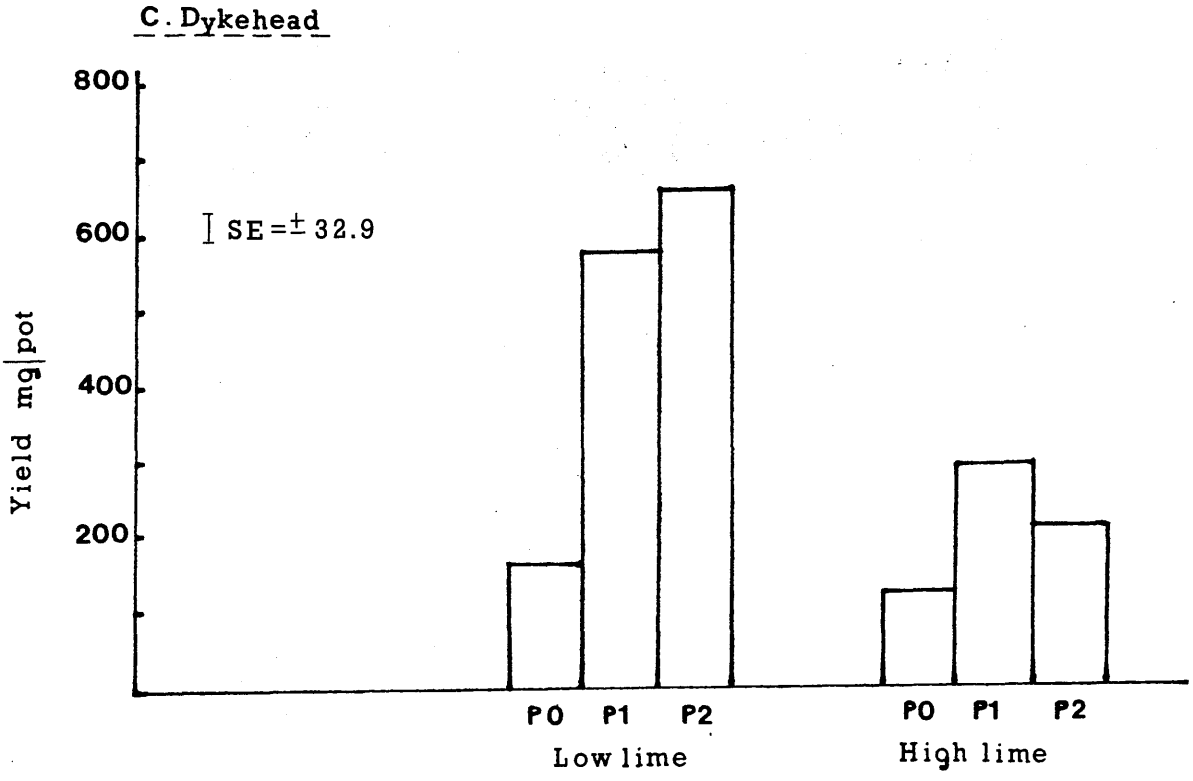


Figure 3.11c. Total yield of perennial ryegrass in pot experiment.

PLATE 4 - WATERSIDE - growth of perennial ryegrass (<u>Lolium perenne</u>) after 32 days.

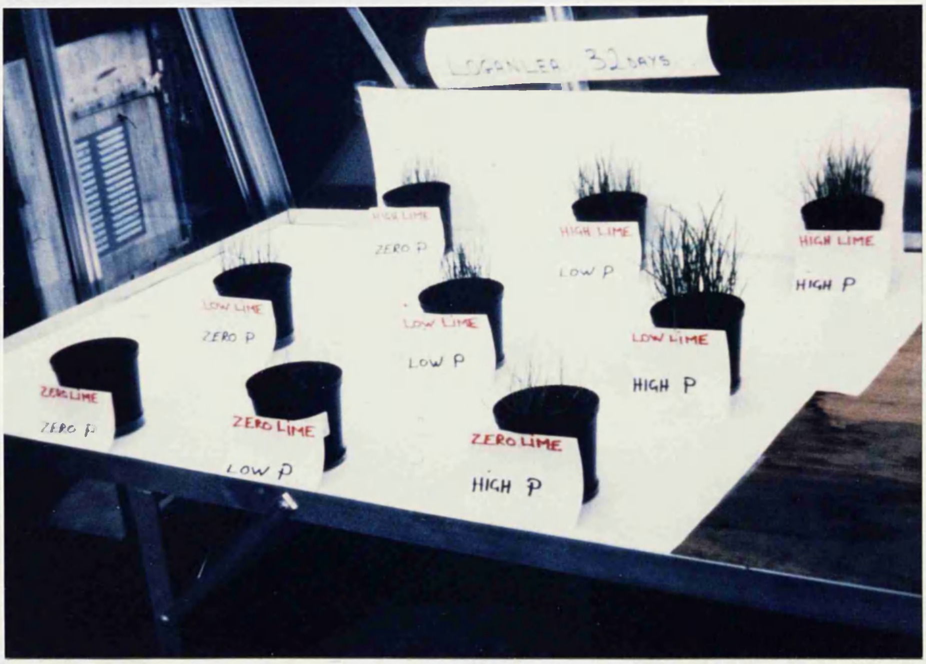


PLATE 5 - LOGANLEE - growth of perennial ryegrass (<u>Lolium perenne</u>) after 32 days.

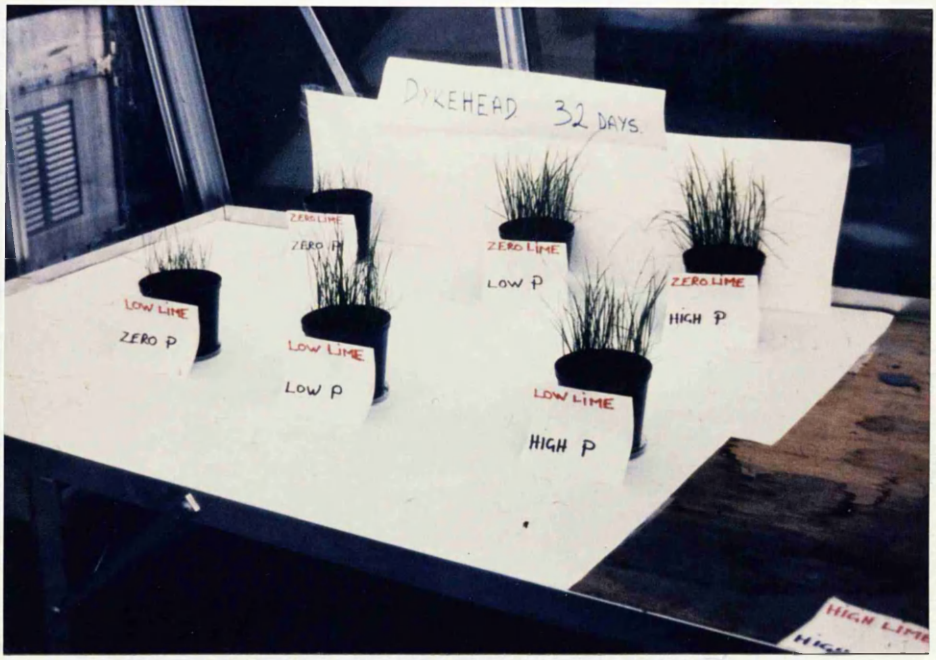


PLATE 6 - DYKEHEAD - growth of perennial ryegrass (<u>Lolium perenne</u>) after 32 days.

liming (see Fig. 3.12). On each of the three spoils addition of lime at the higher rate reduced yield compared to the low lime treatment if phosphorus was added, the yield reduction being greater at the high level of phosphorus addition, although without phosphorus, liming above the immediate spoil requirement had no effect on yield. Kunishi (1982) and Costigan et al. (1982) also reported that the addition of high levels of lime to acid spoil decreased plant yield, although the latter authors found, in contrast to the above results, that the inhibitory effect of high liming was reduced by high phosphorus fertilization. Reduction in yield due to high liming with phosphorus fertilization indicates that the extra lime added to combat future pyrite oxidation either:

(a) fixes some phosphorus in an unavailable form in the spoil,
(b) reduces uptake of phosphorus by plant roots,
(c) affects the transport mechanism within the plant.

(a) and (b) are discussed in later sections.

The effect of phosphorus fertilization on plant yield is shown in Fig. 3.13. Plant yield was increased by addition of 20 kg P/ha to all spoils (unlimed and limed) although no yield increase was found resulting from the addition of phosphorus at 40 kg /ha to unlimed acid spoil or to spoil at pH 7.0 receiving lime, indicating that pH is the factor of greatest influence in spoil with pH $<$ 4.0 and pH $>$ 7.0. All other lime treatments show an increase in plant yield with increasing phosphorus fertilization. However, there is a diminishing effectiveness of phosphorus at the higher rate compared to the effect on yield of addition of 20 kg P/ha.

ANALYSIS OF VARIANCE SHOWS THAT BOTH LIME AND PHOSPHATE ADDITION HAVE A SIGNIFICANT EFFECT ON YIELD AT THE 1% LEVEL ON ALL SITES, AND THAT THE LIME\PHOSPHATE INTERACTION IS ALSO SIGNIFICANT AT 1%.

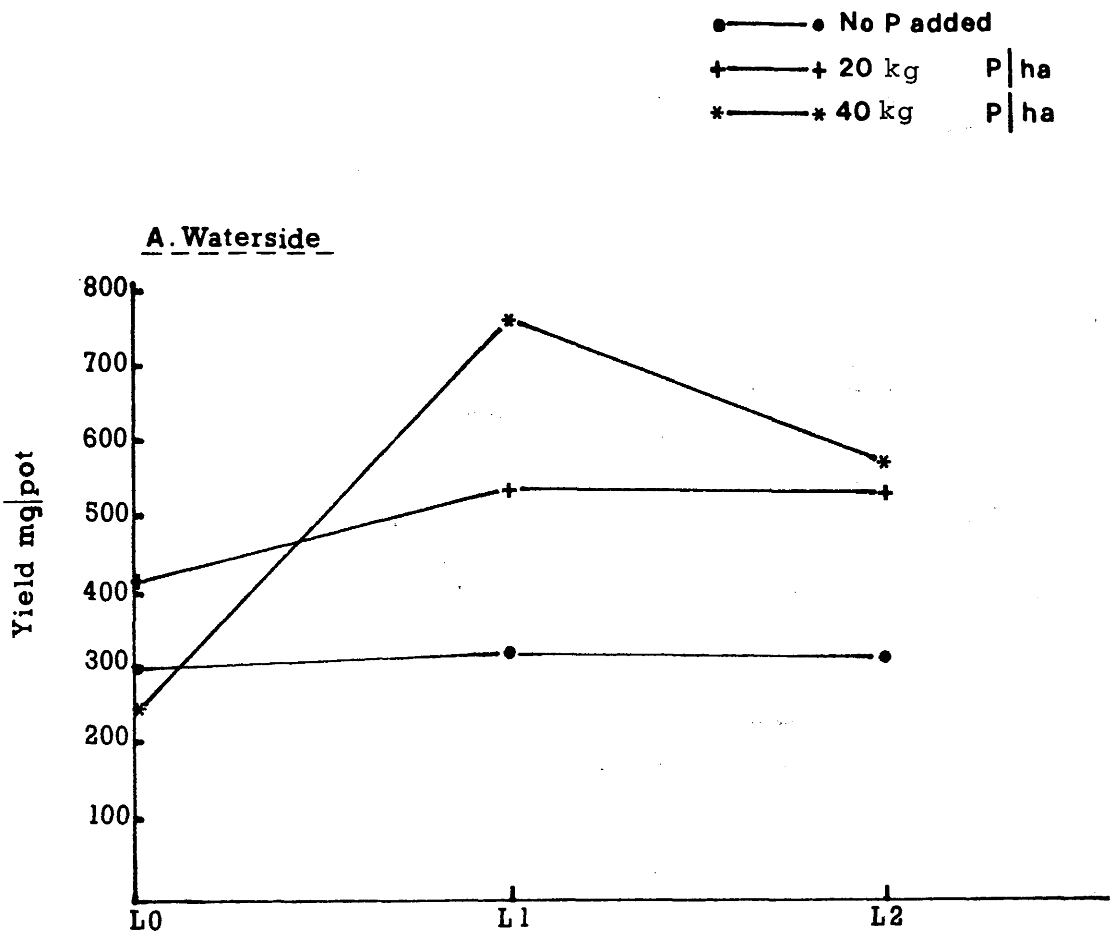


Figure 3.12a. Effect of lime on yield of perennial ryegrass.

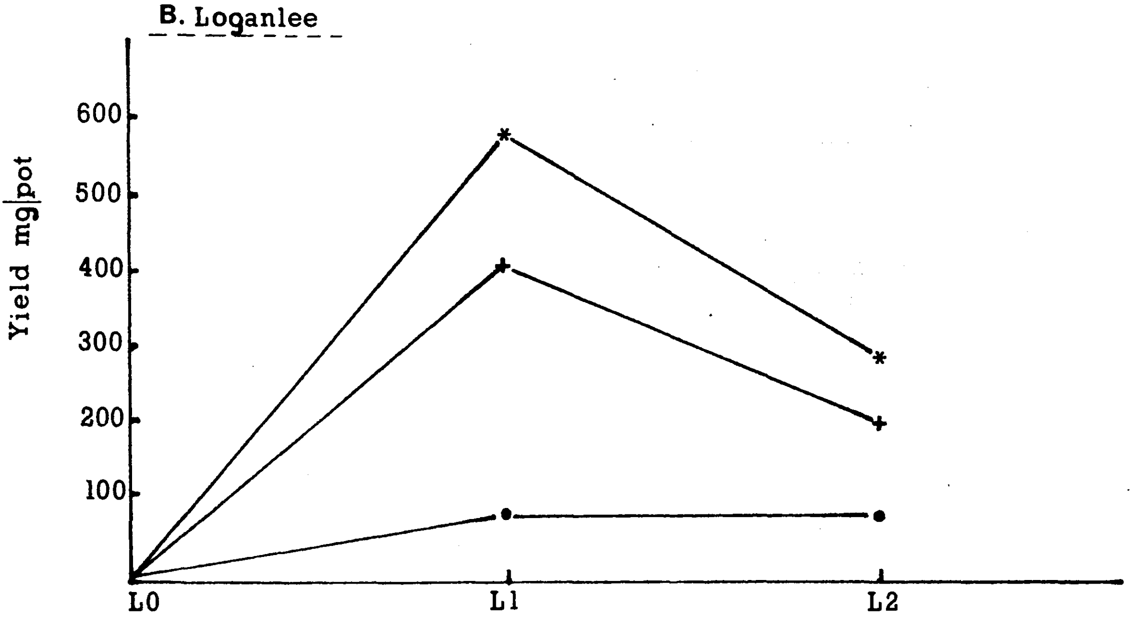


Figure 3.12b. Effect of lime on yield of perennial ryegrass.

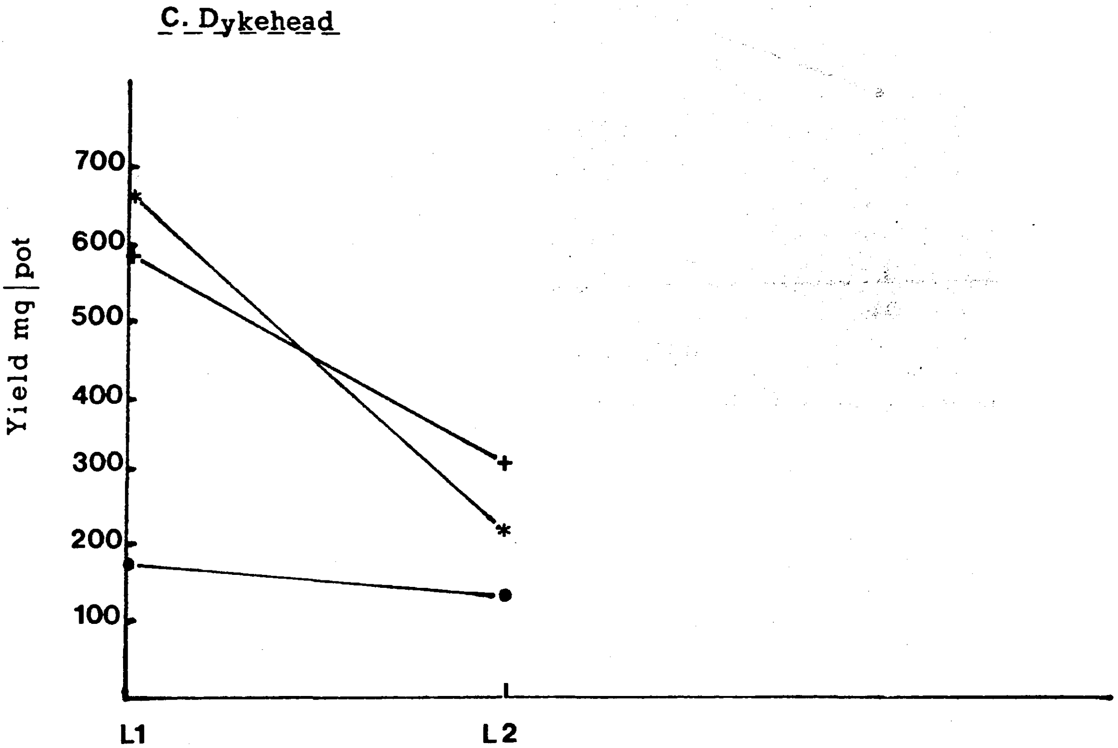


Figure 3.12c. Effect of lime on yield of perennial ryegrass.

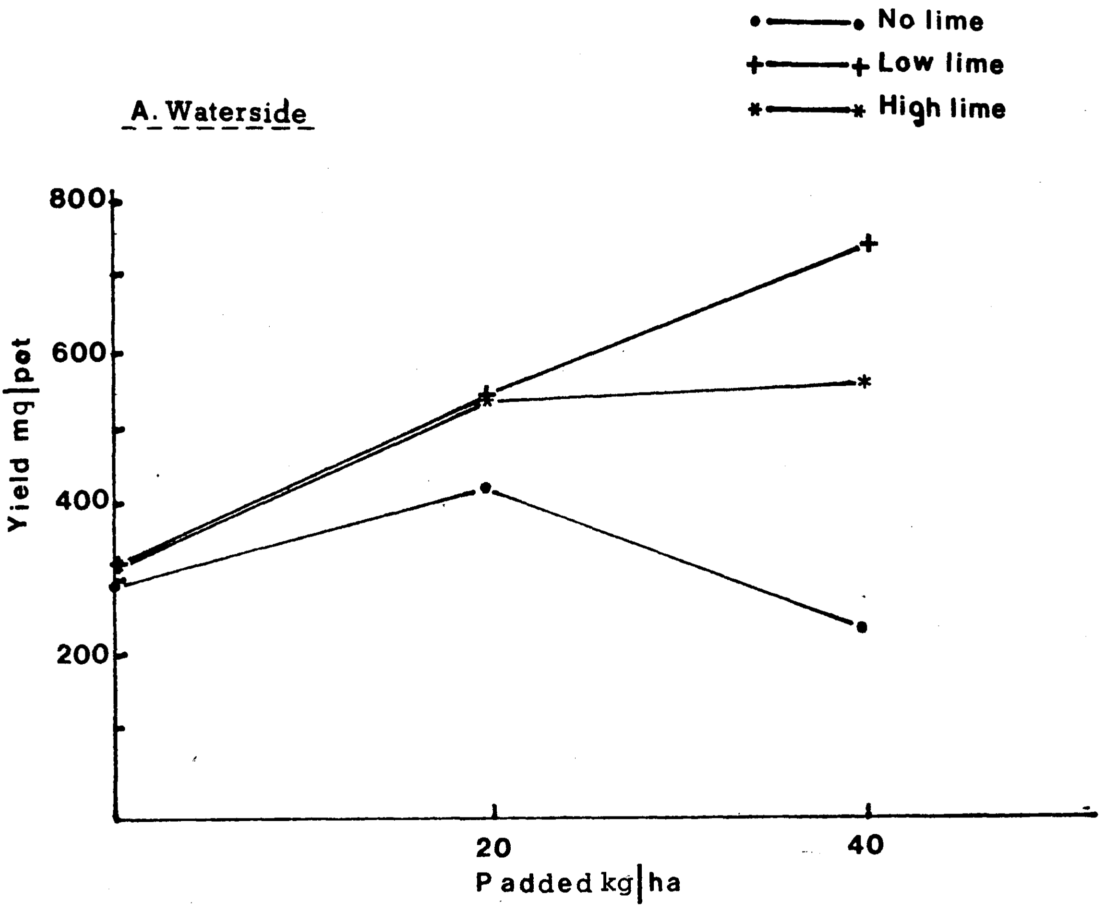


Figure 3.13a. Effect of phosphorus addition on yield of perennial ryegrass.

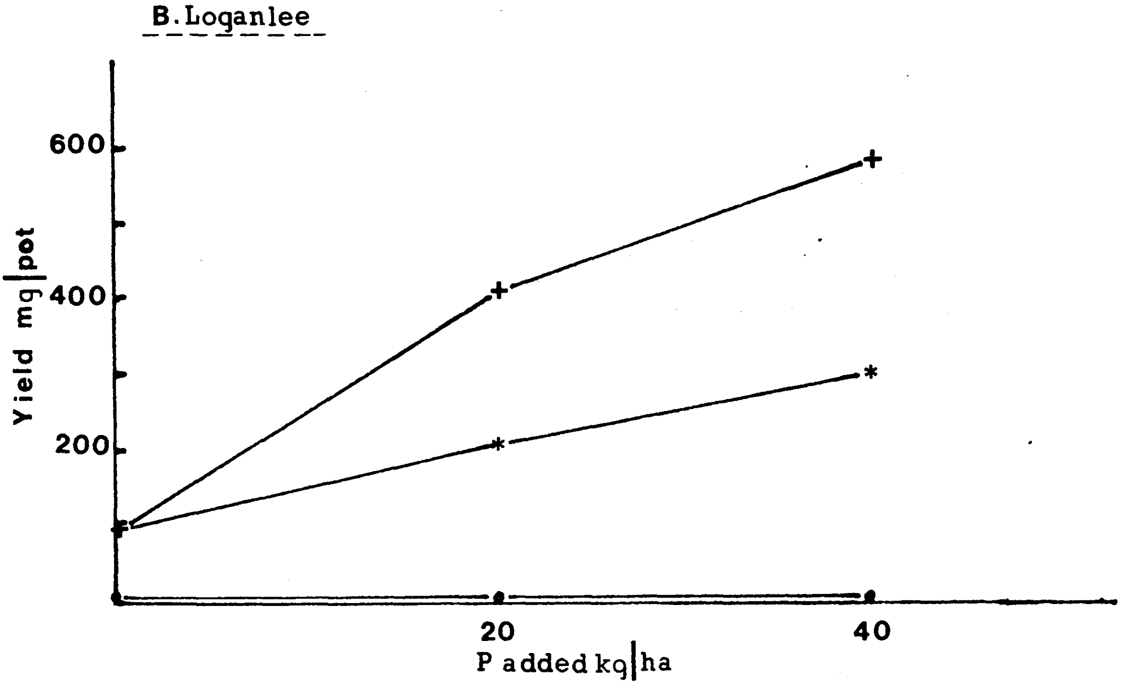


Figure 3.13b. Effect of phosphorus addition on yield of perennial ryegrass.

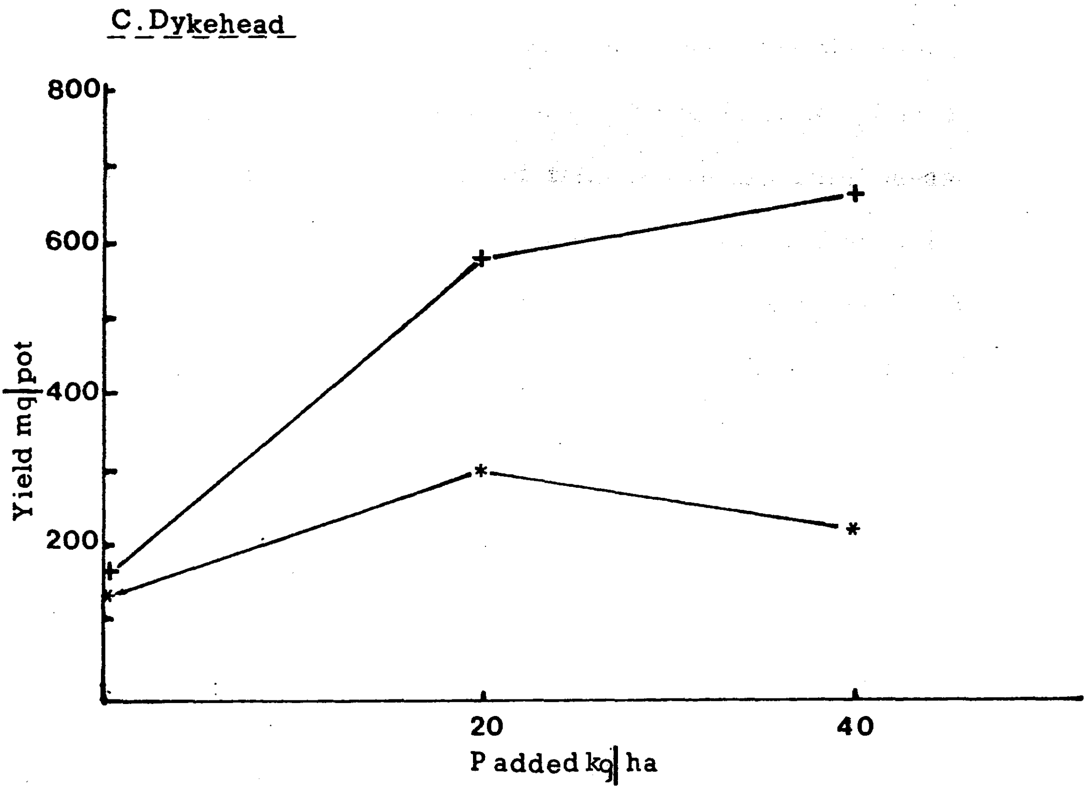


Figure 3.13c. Effect of phosphorus addition on yield of perennial ryegrass.

3.9.3 Effect of lime and phosphorus addition on the phosphorus content of spoil and herbage

The three replicate yields from each treatment were bulked and the total sample divided to give duplicate samples which were digested using nitric acid/perchloric acid (see Chapter 2). The phosphorus content of the digests were measured using the ascorbic acid procedure given in section 3.4.3. Table 3.10 shows the phosphorus content of the grass from each harvest and the total phosphorus content of the combined harvests, together with the level of NH_4F-extractable phosphorus in the spoil (average of triplicate treatments) measured after the second cut and the % recovery of phosphorus (native and added).

In all discussions of these results, treatments LG 1-3 are excluded due to the lack of re-growth after the first cut and the anomalous results found in phosphorus content analysis.

As Table 3.10 shows the phosphorus content of the first harvest is higher in most cases than that of the second harvest (Neller 1953).

The effect of liming is to greatly reduce the level of NH_4F-extractable phosphorus, both native and applied, and to generally reduce the amount of phosphorus taken up by the grass. Neller (1953) in a growth experiment with oats grown on acid fine sand also found a reduction in the phosphorus content of those plants grown in pots receiving lime, similar results being reported by Winsor and Long (1963) in a pot experiment with carnations and lettuce grown in soil receiving lime and phosphorus, the conclusion in both these studies being that phosphorus is inefficiently taken up by plant roots in the presence of excessive calcium, i.e. unreacted lime.

The effect of phosphorus fertilization is, as expected, to increase the level of NH_4F-extractable phosphorus in the spoil and consequently to increase the plant phosphorus content at all levels.

Column 6 of Table 3.10 shows the percentage of the native + added phosphorus which is recovered in both the grass and spoil using the following formula:

$$\frac{\text{P in spoil + P in grass}}{{}^{*}\text{Native P + P in grass}^{*}\text{ + added P}} \times 100$$

* Data from T1 for WS and LG spoils and T4 for DK spoil.

Since no loss occurs through leaching, as each pot stood in a separate cup and watering was controlled so that no percolating water escaped, any unrecovered phosphorus must be fixed in a form which is not extractable by NH_4F, i.e. adsorbed or amorphous oxides, and is not taken up by the plants after 111 days. These results show that the addition of lime greatly reduces the % recovery of phosphorus and that the addition of high rates of phosphorus in general gives a lower recovery compared to the lower rate, a result also reported by McConaghy and Stewart (1963).

Table 3.10. Phosphorus content of grass from each harvest, total phosphorus yield, NH_4F-extractable phosphorus in spoil and % phosphorus recovery

TREATMENT	Plant Phosphorus Content (mg/g)			Spoil Phosphorus (NH_4F-extractable µg/g)	% Phosphorus Recovery
	1st Harvest	2nd Harvest	Combined Harvests		
WS 1	2.04	1.34	1.68	16.6	100
2	1.91	1.68	1.79	44.8	78.8
3	2.28	1.74	1.97	54.2	55.2
4	1.20	1.48	1.36	0.4	7.4
5	1.63	1.70	1.66	12.0	23.6
6	2.37	2.05	2.19	22.8	26.2
7	1.19	0.95	1.08	0.4	6.4
8	1.52	1.06	1.25	0.6	3.4
9	1.71	1.30	1.52	1.5	3.3
LG 1	6.34	n.d.	6.34	5.4	100
2	6.12	n.d.	6.12	24.4	50.7
3	4.87	n.d.	4.87	41.3	45.4
4	1.10	0.89	1.05	0.7	16.7
5	1.77	1.25	1.49	4.0	11.0
6	2.35	1.33	1.74	10.1	13.5
7	1.30	0.84	1.16	0.4	11.5
8	1.56	1.10	1.30	0.2	1.6
9	1.96	1.37	1.64	0.8	2.0
DK 4	0.87	1.25	1.00	2.6	100
5	2.11	1.66	1.90	22.5	62.6
6	3.03	2.30	2.66	38.6	55.5
7	0.95	0.86	0.94	0.4	21.4
8	1.66	1.47	1.57	0.4	3.2
9	1.78	1.46	1.61	0.6	1.7

3.9.4 Effect of lime and phosphorus addition on root growth

After the second harvest a vertical core (1 cm diameter) was taken from each pot and the weight of roots/pot calculated (Table 3.11).

There is no consistent overall effect of lime on rooting in WS (pH 4.0) or DK (pH 7.0) spoils. However, the weight of roots produced at each phosphorus level in LG (pH 3.7) spoil increases with liming. Fitter and Bradshaw (1974) found that liming increased root growth in colliery spoil in the glasshouse if the spoil had pH < 4.1 but had no effect in spoils above this pH, concluding that Aℓ, or another trivalent metal ion was the operative factor in restricting rooting.

The effect of phosphorus fertilization is to increase root mass; WS and LG spoils giving maximum root yield at the lower application rate while DK gives increasing root yield with increasing phosphorus application.

3.9.5 Effect of lime on spoil pH, phosphate adsorption and NH_4F-extractable phosphorus

At the end of the growth experiment the spoil from each pot receiving no fertilizer phosphorus was air dried, and the fraction < 2 m.m. analysed (Table 3.12).

The calculated lime requirement increases the pH of the acid spoils above the target value of 6.2, suggesting that there is unreacted $Ca(OH)_2$ in the spoil. The lime requirement method used is based on field soils and it is not unreasonable to assume that in the field, over a full growing season, reaction with the spoil and leaching losses would

Table 3.11. Effect of lime and phosphorus treatments on root growth

Treatment	Root weight(mg/pot)
WS 1	76
2	124
3	87
4	16
5	129
6	35
7	85
8	152
9	96
LG 1	23
2	23
3	16
4	41
5	90
6	53
7	44
8	150
9	87
DK 4	53
5	150
6	253
7	67
8	69
9	90

Table 3.12 . pH, NH_4F-extractable phosphorus and phosphate adsorption capacity of pots receiving no phosphorus fertilizer

TREATMENT	$Ca(OH)_2$(tonnes/ha)	pH	P adsorption(mg/100g)	NH_4F-extractable P(µg/g)
WS 1	0	4.1	9.3	16.6
4	2.27	7.4	9.8	0.4
7	8.02	8.2	10.4	0.4
LG 1	0	3.8	10.2	5.4
4	7.64	7.2	12.4	0.7
7	10.67	7.9	12.6	0.4
DK 4	0	7.0	6.7	2.6
7	4.24	8.1	7.4	0.4

reduce the effect of the lime. It may be, therefore, that the lime requirement is over-estimated for growth-room conditions by this method. The capacity of the spoil to adsorb phosphorus is increased by liming (thereby reducing the effectiveness of any added phosphorus) by precipitation of calcium phosphate compounds which will, however, break down if the pH drops again, while the extractability of native phosphorus is also reduced by liming (Winsor and Long 1963).

3.9.6 Effect of liming acid spoil on the level of acetic acid extractable iron and aluminium

Waterside and Loganlee pots receiving the low lime treatment and no added phosphorus were extracted with 0.5M acetic acid to measure the effect of liming on the level of "available" Fe and Aℓ, i.e. soil solution and readily exchangeable (Mitchell 1971). - See Table 3.13.

Extraction procedure

Method

Approximately 10g of air-dry < 2 m.m. spoil was shaken with 100 ml of 0.5M acetic acid on an end-over-end shaker for 18 hrs. After filtering through a Whatman's No. 42 filter paper the concentration of Fe and Aℓ was measured on a Perkin Elmer model 370A.

As Table 3.13 shows, liming reduces the availability of Fe and Aℓ in agreement with Pulford (1976), Kimber (1982) who found a negative relationship between pH and the levels of acetic acid-extractable Fe and Aℓ in colliery spoil.

Table 3.13. Effect of liming on pH, acetic acid-extractable iron and aluminium

	Acetic Acid Extractable (μg/g)		
	Fe	Aℓ	pH
WATERSIDE UNLIMED	84	105	4.1
LIMED	49	66	7.4
LOGANLEE UNLIMED	158	185	3.8
LIMED	88	74	7.2

3.10 SUMMARY

Yield of Lolium perenne on the less acid spoil (Waterside pH 4.1) is far greater than on Loganlee spoil (pH 3.8) which showed very poor growth without amendment and no re-growth after the first harvest.

Liming at the lower rate increases yield on both acid spoils and at each level of phosphorus addition (20 and 40 kg/ha) despite reducing the level of NH_4F-extractable phosphorus (both native and applied), suggesting that shortage of phosphorus is not the most limiting factor in unlimed spoils (COMPARE T2 WITH T5 AND T3 WITH T6). The level of acetic-extractable Aℓ is reduced by liming and it may be this factor which increases plant growth (Foy et al. 1965, Kunishi 1982). Liming also increases the phosphate adsorption capacity of the spoil (Mokwunye 1975) and greatly reduces the phosphorus content of the herbage (Neller 1953, Winsor and Long

1963) due to fixation by excess calcium.

Addition of lime at the higher rate, to include an allowance for pyrite content, further reduces NH_4F-extractable phosphorus and decreases yield in phosphorus-fertilized spoil compared to the low lime treatments, indicating that in limed spoil, phosphorus is the limiting factor. Fitter and Bradshaw (1974) found similar results in a pot experiment with Lolium perenne on acid colliery spoil, i.e. liming increased yield on spoil with pH $<$ 4 but reduced yield if pH $>$ 4.

Phosphorus fertilization increases the level of NH_4F-extractable phosphorus and consequently the plant phosphorus content (Volk and McLean 1963). Addition of 20 kg P/ha increases plant yield in all spoils, Waterside giving a better response than the more acid Loganlee spoil (Fitter and Bradshaw 1974).

These results show that it is not only a shortage of phosphorus which is limiting plant growth, but that the acidic nature of the spoil also has a major influence on plant yield. The highest yield obtained on acid spoil is with the lower liming rate and addition of phosphorus at 40 kg/ha, while with Dykehead spoil, high phosphorus rate and no lime, i.e. treatment 6 in each case; despite the relatively high pH of Dykehead (pH 7.0) there is no "free" Ca in treatments 4, 5 and 6. Adding phosphorus to each spoil, limed and unlimed, increases the level of NH_4F-extractable phosphorus, but this does not in itself induce greater yield since Waterside and Loganlee spoils have higher spoil phosphorus levels when no lime is added (treatments 2 and 3) than the highest yielding treatment 6.

In order to achieve highest yield, therefore, acid spoil should be limed to pH 6-7, while spoil with neutral reaction should receive no lime, regardless of pyrite content, since "overliming" results in loss of applied phosphorus and a reduction in the level of native spoil phosphorus. Provided that N and K levels are adequate *Lolium perenne* benefits from the addition of lime and phosphorus, yield increasing with higher phosphorus fertilization up to and including a rate of 40 kg/ha, the highest rate used in this study, with possibly greater yield if higher application rates of P were used.

By not including an allowance in the level of lime added for the pyrite content of the spoil in the field, acid production due to pyrite oxidation and loss of lime by leaching may reduce the pH to an injurious level. If this occurred, the remedy for long-term benefit of the spoil would be to re-apply lime, at a rate determined by analysis, and ploughing the vegetation under, with fertilization if necessary, thereby re-establishing suitable spoil conditions.

CHAPTER 4

CATION EXCHANGE REACTIONS IN COLLIERY SPOIL

4.1 INTRODUCTION

Cation exchange is the reversible process by which cations are exchanged between solid and liquid phases, and between solid phases if in close contact with each other. The solid component of the soil is made up of primary minerals, clay minerals and hydrous oxides, together with organic matter and living organisms, forming a system of more or less discrete particles or aggregates. In this heterogeneous system the soil solution acts as the medium by which chemical reactions between members of the different phases and of the same phase are made possible when the reactants are not in direct contact.

Chemical and physical processes more or less intimately connected with ion exchange include weathering of minerals, nutrient absorption by plants, swelling, shrinkage, and leaching of electrolytes, thus ion exchange may be regarded as one of the most important of all the processes occurring in a soil.

The cations held on the surface of soil minerals, and within the crystal framework of some mineral species, can be reversibly replaced by those of salt solutions, the sum of all cationic species liberated by such reactions being a measure of the cation exchange capacity (CEC) of the soil. The cation exchange capacity of a soil or spoil is the total number of exchange sites of both organic and mineral origin, these being occupied principally by Ca^{2+}, Mg^{2+}, K^+ and Na^+ in temperate soils. CEC therefore represents the total nutrient supplying capacity of a soil, i.e. it is a measure of the total number of exchange sites available to hold plant nutrients for uptake. The proportion of these sites actually occupied can be measured by leaching the soil with a salt solution, e.g. ammonium acetate, replacing all cations with NH_4^+ by mass action, and the Ca^{2+}, Mg^{2+}, K^+ and Na^+ content of the leachate determined - this represents the level of exchangeable bases in the soil.

Colliery spoil is by nature a poor growth medium and requires the addition of nutrients, notably nitrogen and phosphorus, for plant growth. However, little work has been done to evaluate spoil as a reservoir of exchangeable cations or to measure its capacity to hold added cations in an exchangeable form. The work described in this chapter was carried out to investigate these factors, and to determine the role of the amorphous oxide fraction in cation exchange reactions.

4.2 The Cation Exchange Complex

The negative charges active in cation exchange reactions in the soil arise in 3 ways:

1. Cation substitution within mineral layers
2. Broken edge bonds
3. H^+ ionizing from organic soil colloids.

4.2.1 Cation substitution

Cation substitution occurs within the mineral layers of all 2:1 layer silicates, which consist of an aluminium octahedral sheet lying between two silica tetrahedral sheets. During formation some of the aluminium atoms of the octahedral layer may be replaced by magnesium atoms due to their similar atomic size. The substitution of each divalent atom for a trivalent atom leaves one unsatisfied negative charge on the lattice, which may be balanced by cations held near the exchange surface. Charges arising from cation substitution contribute towards the cation exchange capacity if the counterions are held at the outer surface of the particle, while those held between the layers are exchangeable only if the lattice expands, as in montmorillonite and vermiculite, but are non-exchangeable due to inaccessibility in the non-expanding lattices. Table 4.1 lists the cation exchange capacity of the major silicate clays. Charge due to cation substitution is a permanent charge and is not pH dependent (Foth 1984).

Table 4.1. Cation exchange capacities of soil clays (Thompson and Troeh 1973)

Mineral	Lattice	CEC range(me/100g)
Kaolinite	1:1 non-expanding	3-15
Chlorite	2:1 partially expanded	20-40
Illite	2:1 non-expanding	20-40
Montmorillonite	2:1 expanding	60-100
Vermiculite	2:1 partially expanded	100-200

4.2.2 Broken edge bonds

All mineral lattices which are held together by continuous sequences of ionic bonds possess unsatisfied charges at the edge of the structure. The amount of charge associated with these bonds increases with decreasing particle size and reaches a maximum in allophane, an amorphous clay which includes the building blocks of minerals; tetrahedra containing silica and octahedra containing aluminium, magnesium and iron, and which has a large pH-dependent cation exchange capacity, increasing rapidly in the range pH 4-10 due to surface hydroxyl group ionization (Fields and Claridge 1975). All charges arising from broken edge bonds are pH-dependent and may be represented in a simple form as shown in Fig. 4.1.

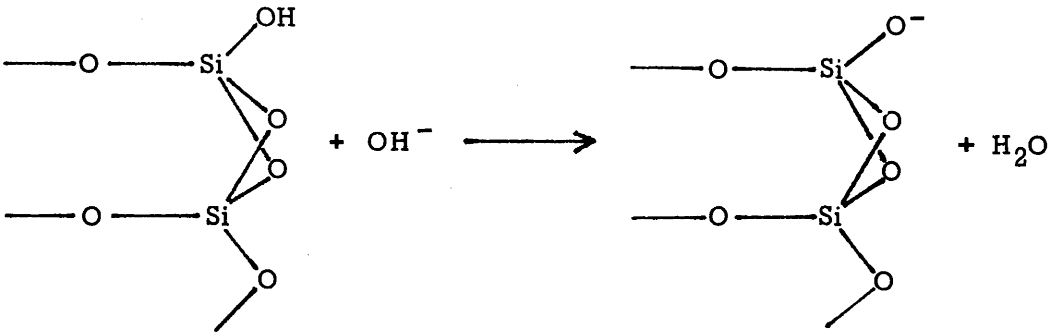


Fig. 4.1 Charge arising from hydroxyl group ionization in clay minerals.

4.2.3 Organic soil colloids

The colloidal organic fraction, or humus, in the soil does not have a single structural formula but is composed basically of carbon, hydrogen and oxygen. It has a much higher cation exchange capacity than the silicate clays, 200-300 me/100g of well-humified organic matter, the charges arising from the ionization of H^+ from carboxyl and phenolic hydroxyl functions (Fig. 4.2)

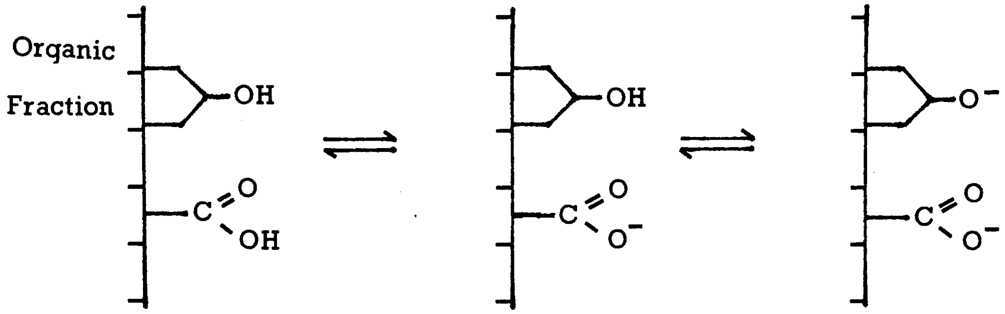


Fig. 4.2. Charge arising from hydroxyl group ionization in organic matter.

The charge on the humus colloid is pH-dependent (Sawhney et al. 1970, Foth 1984), decreasing under acid conditions when hydrogen is tightly bound to the colloidal surface. As well as surface adsorption shown in Fig. 4.2, functional groups within the humus structure also contribute to the cation exchange capacity. Microbial and chemical breakdown of organic matter in the soil exposes further functional groups for reaction, thus increasing the cation exchange capacity. As a result, organic-rich soils have high cation exchange capacities, primarily due to the organic fraction (pH-dependent) with smaller contributions from the mineral fraction (mainly a permanent charge) (Wada and Wada 1985).

4.3 Soil Exchange Reactions

The cation species adsorbed by the cation exchange complex in the soil is governed by 3 factors.

1. The available supply of each cation
2. The intensity of leaching and other removal processes
3. The strength with which each cation is adsorbed.

4.3.1 Cation availability

The available supply of each cation consists of ions which have been recycled through plants and soil fauna and returned to the soil, and fresh supplies either from mineral weathering or added as fertilizer. Weathering is the process by which rocks are broken down chemically and physically, with the subsequent release of cations and anions from the mineral components. Some of the soluble products may be removed from the reaction site by leaching, but some may be adsorbed on the clay minerals, or may combine with other products to form clay minerals. Ca^{2+}, Mg^{2+}, K^+ and Na^+, together with other cations, adsorbed by the clay and organic matter are in this way made available to plants, and although the amount brought into the exchangeable pool represents only a fraction of that stored in the mineral fraction it provides a continuous supply of plant nutrients.

Addition of compound fertilizers increase the level of available nitrogen, phosphorus and potassium but the purity of inorganic fertilizers means that minor elements are not included, although formulations are available which supply specific cations, e.g. Zn^{2+}, Cu^{2+}, Mn^{2+} etc. if these are specifically required by a crop. Organic fertilizers such as farmyard manure, plant residues and sewage sludge supply a wider range of nutrients than inorganic forms and also add organic matter to the soil which will improve soil structure and may accumulate as humus, thus increasing the soil's cation exchange capacity.

4.3.2 Cation removal processes

Leaching removes excess soluble salts, i.e. those not held on the exchange complex, while removal of adsorbed cations involves an exchange reaction. CO_2 dissolving in rainwater, and decomposing organic matter are sources of H^+ ions which replace adsorbed cations, the latter being leached from the soil. The other main cation removal process is loss through plant uptake, the exact amount taken up by a crop depending on soil conditions and the plant species. This loss is only temporary if the crop is ploughed under as a green manure, as breakdown of the plant material in the soil releases its constituent minerals. However, crop removal represents a continuous drain on the soil nutrient reserves.

4.3.3 Cation adsorption

The strength of the attractive force between a cation and the exchange complex depends on the charge of the cation, the charge density on the exchange surface and how close the cation can come to the exchange sites. The closeness of approach is limited by the amount of water of hydration carried by the ion, with higher charge and smaller non-hydrated size both giving a larger hydrated ion. The sequence of relative attractive forces, reflecting both the charge and hydrated size of the cations is $A\ell^{3+} > Ca^{2+} > Mg^{2+} > K^+ = NH_4^+ > Na^+$, and this so-called lyotrophic series represents a preferential adsorption sequence, i.e. cation exchange sites adsorb more of a cation early in the sequence than of one later in the sequence if the two are present in equivalent amounts, however, preferential adsorption can be overcome by an ion that is much more concentrated than the others in the soil solution.

4.4 Sources of Exchangeable Bases

Calcium ions are strongly adsorbed on cation exchange sites, due to the small hydrated size of the divalent ion, and account for 80-85% of the exchangeable bases in arable soils (Kelley 1964, Thompson and Troeh 1973). Calcium minerals include feldspar $CaA\ell_2Si_2O_8$, apatite $Ca_5(F,C\ell)/(PO_4)_3$, gypsum $CaSO_4$, dolomite $CaMg(CO_3)_2$ and calcite $CaCO_3$, which release available Ca^{2+}, the form in which calcium is taken up by plants. There is no significant fixation of Ca^{2+} into unavailable forms, losses being mainly as calcium bicarbonate formed by rainwater reacting with carbonate minerals.

Magnesium minerals include biotite $K_2(Si_6A\ell_2)(Mg,Fe)_6O_{20}OH_4$, serpentine $Mg_6Si_4O_{10}(OH)_8$, olivine $(Mg,Fe)_2SiO_4$ and dolomite $CaMg(CO_3)_2$, however, the main supply of Mg^{2+} to plants is the breakdown of clay minerals. Up to 65% of soil magnesium is in the clay fraction (Salmon 1963), which weathers sufficiently rapidly in the soil to make good any net loss of magnesium through leaching or crop removal. 12-18% of the exchangeable bases are normally Mg^{2+} ions (Thompson and Troeh 1973) and if lime is applied as dolomitic limestone, magnesium levels should not be limiting to plant growth.

Potassium-containing minerals include feldspar $(K,Na)A\ell Si_3O_8$, biotite $K_2(Si_6A\ell_2)A\ell_4O_{20}(OH)_4$ and clay minerals which break down to release available potassium, and a continuous movement of K^+ between the fixed K^+ (in the lattice), exchangeable K^+ (on the cation exchange complex) a solution K^+ (plant available) occurs. These reactions are reversible since K^+ ions released by weathering can be adsorbed on the exchange complex or may re-enter the clay or biotite lattice and become fixed. Diffusion of K^+ from exchange sites through the soil solution accounts for about 90% of K^+ uptake by plants (Foth 1984) and removal from the soil solution causes dissociation of K^+ from the exchange sites to maintain the equilbrium of the system. Acid breakdown of clay minerals in colliery spoil ensures an adequate supply of potassium for plant uptake (Bloomfield 1982), the minimum level of 0.1 me/100g or 2-3% of the total exchangeable base content recommended by Foth (1984) for plant requirements being exceeded in all spoils in this work (Table 4.6).

Sodium occurs in feldspar $(K,Na)A\ell Si_3O_8$ and hornblende $NaCa_2(Mg,Fe)_4(A\ell,Fe)(SiA\ell)_8O_{22}(OH,F)_2$, sodium feldspar weathering more rapidly than potassium feldspar. Na^+ is the easiest basic cation leached from soil as it is less tightly held on cation exchange sites than Ca^{2+}, Mg^{2+} or K^+. Na^+ accounts for 1-2% of the total exchangeable bases in arable soils (Foth 1984) and although it has not been shown to be essential for plant growth some evidence shows that it may partially substitute for K^+ in meeting plant requirements (Thompson and Troeh 1973).

4.5 Cation Exchange Capacity of Colliery Spoil

The cation exchange capacity of colliery spoil, which is almost invariably low in organic matter, will be due principally to isomorphous substitution and broken edge bonds, and may be compared to a mineral soil low in organic matter. Smith et al. (1971) found that the CEC of surface soils was consistently higher than that of iron ore spoil in West Virginia. However, in many cases the spoils had higher CEC than the soil horizons below 15 cm, the decrease in CEC of the soils with depth being due to lower organic matter levels, values for the spoils being 6.8 and 11.6 me/100g which may be compared to results reported by others for colliery spoil. Abbott and Bacon (1977) reported the CEC of colliery spoil in Eastern Canada varying between 8.6 and 9.6 me/100g, while Stucky et al. (1980) working on Illinois strip-mine waste with an organic matter content of 2.7% found an average CEC of 11.7 me/100g.

Stucky et al. (1980) found in growth experiments on spoil treated with sewage sludge that plants would grow under conditions in which the level of available trace metals (both available in the spoil and added in the sewage sludge) exceeded amounts known to be capable of inducing phytotoxic effects or reducing growth. Among the mechanisms proposed for the reduction in the amount of trace metals available to the plant were (a) reaction of cations with carboxyl and phenolic functional groups of the organic colloid, and (b) adsorption of cations on clay minerals. This example illustrates the cation exchange properties of colliery spoil, in this case the cations being trace metals.

Another example can be drawn from trace metal analysis which shows spoil exchange reactions of significance when considering the cation exchange properties of a spoil. In soils the hydrous oxides of iron, aluminium and manganese hold significant proportions of some trace metal ions by exchange adsorption, the amount of adsorbed species varying with soil pH due to the pH-dependency of the charge on the oxide fraction (Childs and Leslie 1977, Jones and Jarvis 1981, Kimber 1982).

Extraction of colliery spoil with acid ammonium oxalate (Tamm's reagent) shows that there is a significant oxide fraction, the oxalate-extractable component varying greatly from site to site. Kimber (1982) found levels of oxalate-extractable iron, aluminium and manganese in unburnt Scottish colliery spoil ranging from 1-4,420, 8-960 and 1-536µg/g respectively, and the possibility is investigated here that these oxides may influence the cation exchange properties of the spoil. Tamm's reagent (see Chapter 3) specifically dissolves amorphous oxides, the aged or crystalline fraction being unaffected, therefore analysis of the oxalate extract and measurement of spoil CEC and exchangeable bases will reveal any relationship which may exist between the amorphous oxide fraction and cation adsorption properties.

Under acid conditions, interaction of H^+ with the oxide surface will occur, leading to a net positive charge, whereas addition of OH^- to the system, i.e. raising the pH, will create a negative surface with cation exchange properties (Fig. 4.3).

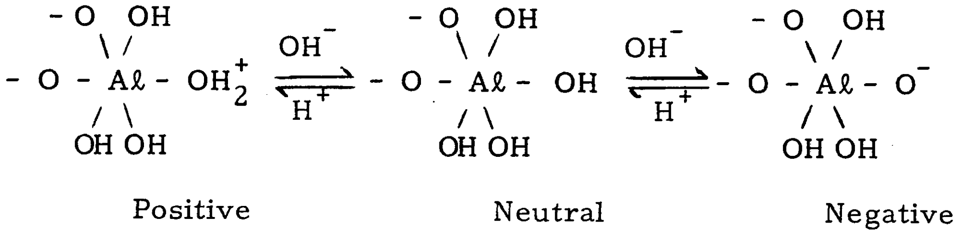


Fig. 4.3. Variation of surface charge on aluminium oxide surface with pH.

In the following sections the role of the amorphous oxide fraction as cation exchange reaction sites in the spoil is investigated.

4.6 Measurement of Cation Exchange Capacity

There a number of methods used for determining the cation exchange capacity of soils and these can be grouped into the following categories:

(a) Those in which the soil is leached with a dilute acid, e.g. HCℓ, and the hydrogen-saturated exchange material titrated to pH 7 with barium hydroxide, or to pH 8.5 with sodium hydroxide.

(b) Those in which the exchangeable cations are stoichiometrically replaced by the acetate of K^+, NH_4^+, Ba^{2+}, Ca^{2+} or Na^+, and the amount of replacing cation adsorbed determined.

(c) Those which involve equilibrating soils (pre-leached with calcium acetate) with a dilute solution (100μg Ca/ml) of calcium nitrate containing Ca^{40} and radioactive Ca^{45} and the uptake of radioactive isotope determined.

Most CEC investigations employ methods based on saturating the exchange complex with a given cation and determining the total amount of cation adsorbed. However, there is discussion as to whether to use a replacing salt at pH 8.2 or at pH 7.0. Those who prefer the former do so because it is closer to the equilibrium pH between soil and $CaCO_3$ at the partial pressure of CO_2 in the atmosphere, while those who prefer working with replacing solutions at pH 7.0 do so because it is the neutral point of water and may more nearly represent the pH of the soil-bicarbonate-carbonic acid buffer system at the partial pressure of CO_2 likely to prevail in the atmosphere of a fertile soil during the season of active growth.

All CEC determinations carried out in this work used the method of saturating the exchange complex at pH 7.0 with K^+, and after removal of excess saturating salt, displacing all adsorbed K^+ ions using NH_4^+, the amount of displaced K^+ being a measure of the spoil's cation exchange capacity. Two methods of equilibrating the spoil with the saturating solutions may be used, either by shaking or leaching, and the suitability of each method was investigated.

4.6.1 Materials and Methods

Four samples of spoil from an unreclaimed site at Auchencruive, Ayrshire, were chosen for this investigation. Each sample was air-dried and the fraction $<$ 2 m.m. analysed.

(a) Shaking method

5g of $<$ 2 m.m. sample were washed with 3 x 30 ml portions of 90% ethanol, with shaking and centrifuging to remove soluble salts not held as exchange ions. This was followed by 3 x 40 ml shakings with 1M potassium acetate at pH 7.0 to saturate the exchange sites. The K^+-saturates sample was then washed with a further 3 x 30 ml of 90% ethanol and finally shaken with 3 x 30 ml portions of 1M ammonium acetate at pH 7.0, the supernates being collected and made to 100 ml for K^+ determination by flame emission. All results are expressed as milli-equivalents of K^+ per 100g of spoil (me/100g) and all analyses were carried out in duplicate.

(b) Leaching method

10g of $<$ 2 m.m. sample were mixed with an equal volume of acid-washed sand to aid percolation, and placed in a leaching tube (2.5 cm I.D.). The column was washed with 100 ml of 90% ethanol and the leachate discarded. All exchangeable cations were replaced by K^+ by leaching with 200 ml of 1M potassium acetate at pH 7.0, followed by washing with 100 ml of 90% ethanol to remove excess K^+ in the column. The K^+-saturated sample was then leached with 200 ml of 1M ammonium acetate at pH 7.0 and the leachate made to 250 ml for K^+ determination. All analyses were carried out in duplicate.

4.6.2 Results

Table 4.2 shows the results of CEC determinations on each sample using both methods.

Table 4.2. Comparison of CEC determinations using shaking and leaching methods

Sample	CEC (me/100g) Shaking	Leaching
AC1	25.8	26.7
AC2	30.4	29.4
AC3	27.0	25.6
AC4	27.9	27.3

As table 4.2 shows there is close agreement between the results using the shaking and leaching methods, the mean and standard deviation for each method being 27.77 ± 1.95 and 27.25 ± 1.60, respectively. For all further work carried out on CEC determinations of spoil the column leaching method was chosen due to its convenience in handling a large number of samples at once.

4.7 Determination of Exchangeable Bases in Colliery Spoil

Exchangeable bases are defined as the alkali earth metals, principally Ca^{2+}, Mg^{2+}, K^+ and Na^+, attached to the exchange complex (organic and inorganic) which can be exchanged with each other, and with other positively charged ions in the soil solution. Measurement of the exchangeable base content of a soil or spoil involves replacement of the adsorbed cations with NH_4^+, and the Ca^{2+}, Mg^{2+}, K^+ and Na^+ content of the leachate determined.

4.7.1 Method

10g of sample (<2 m.m. fraction) were mixed with an equal volume of acid-washed sand and placed in a leaching tube (2.5 cm I.D.). The column was washed with 100 ml of 90% ethanol to remove soluble salts, and the leachate discarded. All exchangeable cations were displaced by leaching with 200 ml of 1M ammonium acetate at pH 7.0, the leachate collected and made to 250 ml. Ca^{2+} and Mg^{2+} were determined by atomic absorption spectrophotometry using a Perkin Elmer model 370A, and K^+ and Na^+ were determined by flame emission. The level of exchangeable bases is considered to be the sum of these elements in the leachate expressed in milliequivalents per 100g sample (me/100g). All analyses were carried out in duplicate.

4.8 Base Saturation

The percentage base saturation of a soil is defined as the percentage of the cation exchange sites occupied by bases, i.e. Ca^{2+}, Mg^{2+}, K^+ and Na^+. Base saturation may be used as an index of leaching in a soil, highly leached soils having a lower value. Exchangeable base determinations measure the pool of available bases at any one moment, but percentage base saturation may be used to relate the exchangeable base content to the potential cation supplying power of the soil.

4.9 Results and Discussion

Cation exchange capacity and exchangeable base determinations were carried out on 21 spoil samples chosen from those described in Chapter 3, those chosen for further analysis representing a range of pH values from 5 sites. Both measurements were carried out on the $<$ 2 m.m. fraction using the column leaching methods. Table 4.3 shows the results, together with pH and oxalate-extractable Fe, Aℓ and Mn levels. All analyses were carried out in duplicate.

CEC values range from 11.6-24.2 me/100g, with 19 samples having values $<$ 20.0 me/100g, compared to values of 10-15 me/100g for a sandy loam soil (Hausenbuiller 1972) and 25-30 me/100g for productive silt or clay loam (Bradshaw and Chadwick 1980).

Table 4.4 shows that the oxalate-extractable Aℓ fraction is significantly positively correlated with CEC at the 0.1% level ($r = 0.747$). This relationship is unaffected by spoil pH, since the partial correlation coefficient between these two parameters, taking into account the variability in pH is 0.742*** (the formula for calculating the partial correlation coefficient is shown in Chapter 3). This relationship suggests that amorphous aluminium oxides occupy a significant proportion of the cation exchange sites in the spoil. The influence of the oxide fraction on spoil properties was highlighted in Chapter 3, where the relationship between amorphous iron and aluminium oxides on the phosphate adsorbing capacity of spoil showed the existence of a positively charged surface on the oxide fraction. Soil and spoil amorphous oxides, as well as synthetic oxides, have been extensively studied in this respect, i.e. having an ANION holding capacity (Pulford and Duncan 1975, Borggaard 1984, Beyrouty

Table 4.3. Cation exchange capacity, exchangeable bases, pH and oxalate-extractable Fe, Aℓ and Mn of a range of spoil samples

Sample	pH	Oxalate extract (μg/g)			CEC (me/100g)	Exchangeable Bases (me/100g)	% Base Saturation
		Fe	Aℓ	Mn			
SF 5	6.7	1560	151	46	13.4	10.3	77.2
10	3.7	4395	725	7	13.8	2.5	18.0
12	3.5	4190	800	56	12.9	1.0	7.8
17	7.5	5870	275	172	13.1	12.6	96.1
22	5.6	3290	425	507	13.4	7.9	59.0
WS 2	5.2	3160	584	43	14.2	6.9	48.7
3	4.1	5590	723	17	13.5	3.5	25.6
5	4.9	4480	564	131	13.0	4.7	36.2
7	6.7	3570	230	74	15.4	9.4	61.3
10	4.8	3580	383	51	11.6	6.3	54.5
LG 6	3.9	3030	1240	8	24.2	2.4	10.0
10	3.4	5140	550	5	14.0	1.3	9.4
SB 4	3.7	11300	1930	28	24.1	4.9	20.4
8	4.6	3950	1160	26	19.9	5.1	25.7
12	5.6	1450	240	151	15.4	10.4	67.3
15	5.1	3350	280	102	19.2	10.1	52.4
21	3.2	17500	790	12	16.5	3.0	18.1
DK 2	5.5	1445	230	59	13.3	8.1	61.1
4	6.8	1440	285	175	12.7	10.0	78.7
8	6.3	5650	232	122	14.8	9.9	66.8
10	4.5	4100	315	82	12.5	6.1	48.9

Table 4.4. Correlation coefficients and significance of the relationships between CEC, pH, oxalate-extractable Fe, Aℓ and Mn

	Correlation coefficient
CEC vs pH	-0.332
CEC vs Fe	0.297
CEC vs Aℓ	0.747***
CEC vs Mn	-0.265

Table 4.5 Correlation coefficients and significance of the relationships between exchangeable bases, pH, oxalate-extractable Fe, Aℓ and Mn

	Correlation coefficient
Exchangeable bases vs pH	0.926***
" " " Fe	-0.360
" " " Aℓ	-0.608**
" " " Mn	0.450*

et al. 1984). Hatcher et al. (1967) found that freshly precipitated aluminium hydroxide adsorbed relatively large amounts of boron, whereas aged or polymerized hydroxides had a reduced boron adsorption capacity, indicating that surface hydroxyl groups were the active boron adsorption sites. The positively charged aluminium hydroxide fraction was also found to be adsorbed in an exchangeable form by montmorillonite by Shen and Rich (1962) and Turner and Brydon (1962) and the above results indicate an interaction between the cationic aluminium fraction and the negatively charged cation exchange complex in the spoil, since acid oxalate extraction of the spoil dissolves the amorphous, or non-crystalline hydrous oxide fraction. The amorphous iron oxide fraction, like the aluminium fraction, is positively correlated with CEC ($r = 0.297$) although not at a significant level, indicating that iron may also be held on cation exchange sites in its oxide form although it does not show such a close association as aluminium. Figures 4.4-4.7 show the relationships between CEC and pH, oxalate Fe, Aℓ and Mn respectively.

CEC and spoil pH are negatively correlated ($r = -0.332$), however, by removing the influence of the aluminium oxide fraction, since this occupies a significant proportion of the exchange sites, the correlation between pH and CEC has a positive sign ($r = 0.305$). This relationship, although not significant at the $< 5\%$ level agrees with other findings, e.g. Helling et al. (1964), Bhumbla and McLean (1965), Sawhney et al (1970) working with clay minerals, indicating a pH-dependent charge on the exchange complex.

The level of exchangeable bases in the 21 samples analysed is significantly correlated with spoil pH ($r = 0.926^{***}$, Fig. 4.8), oxalate

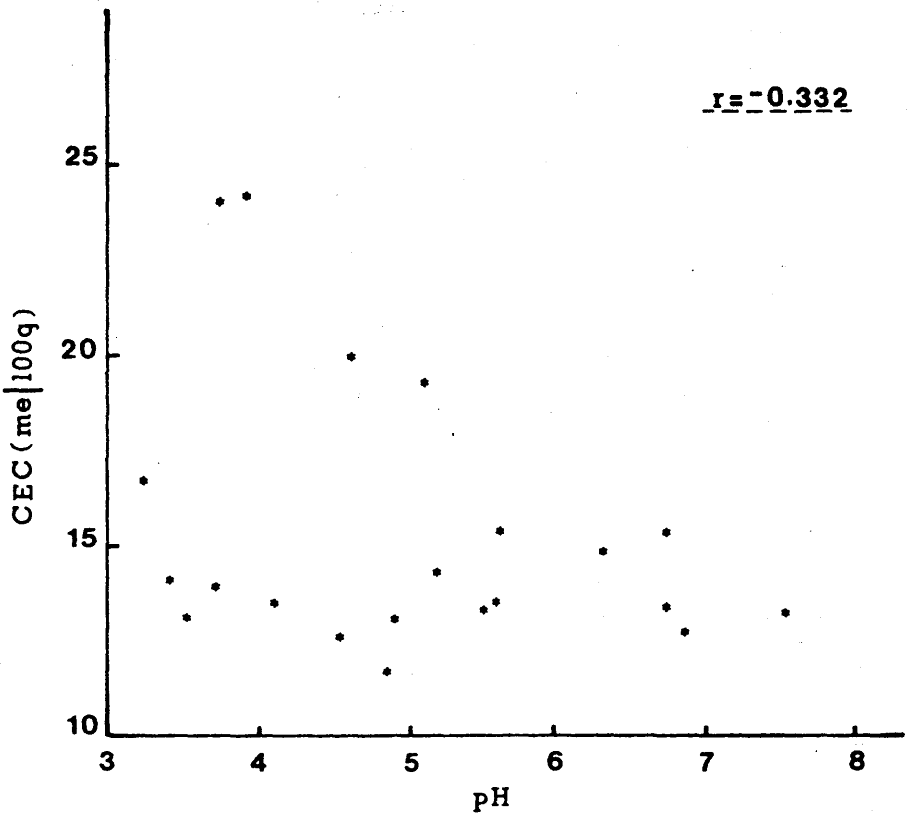


Figure 4.4. Relationship between spoil pH and cation exchange capacity.

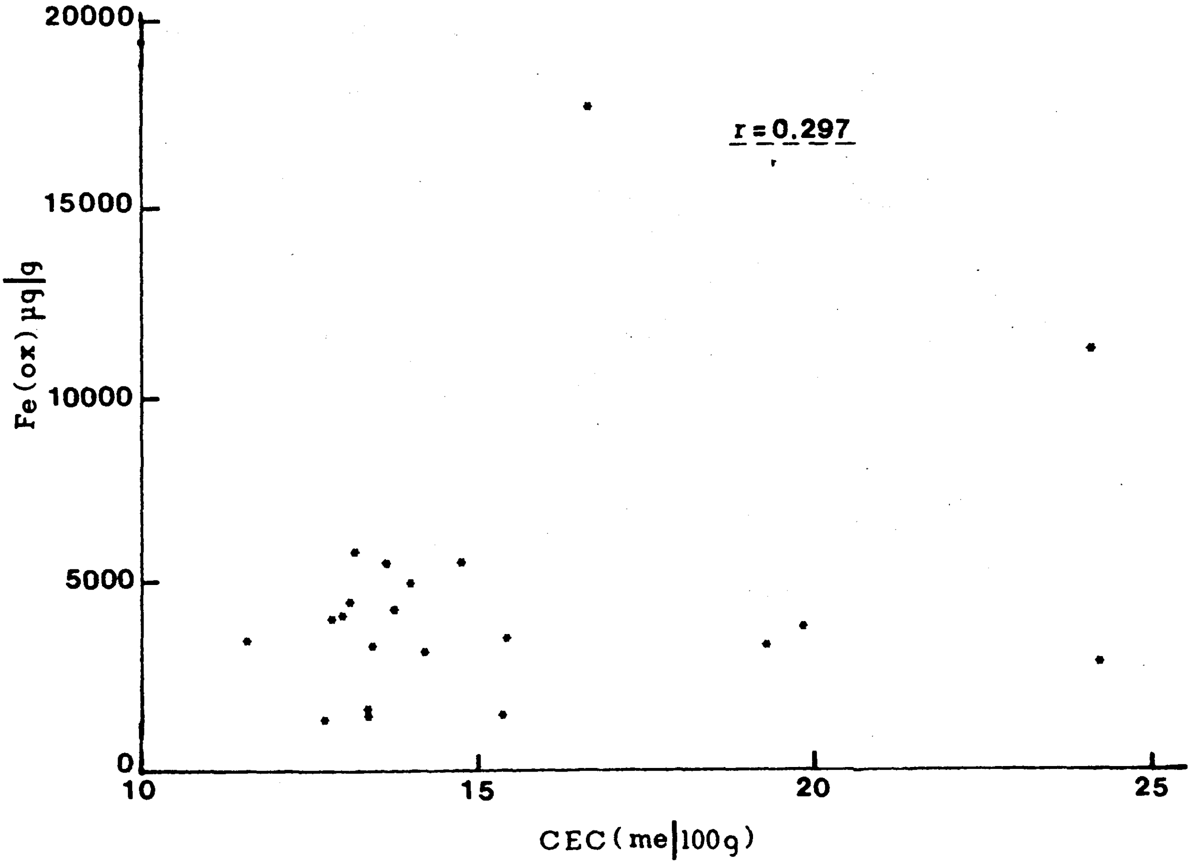


Figure 4.5. Relationship between cation exchange capacity and oxalate extractable iron.

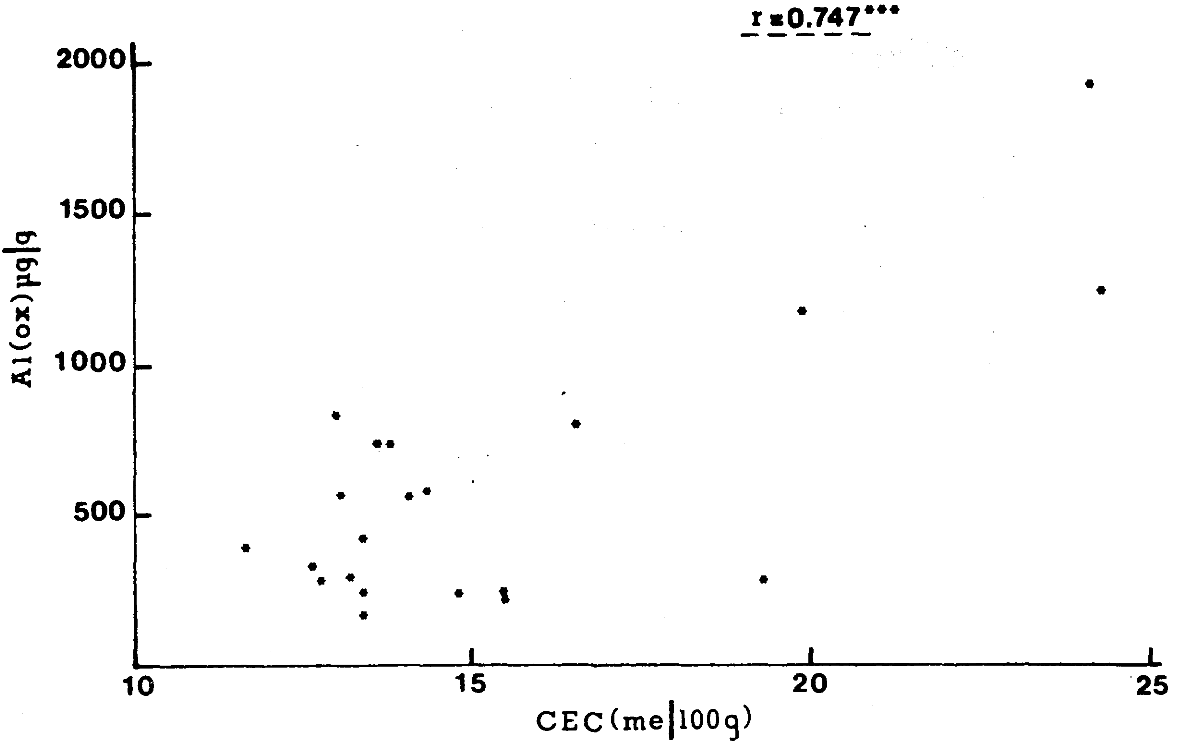


Figure 4.6. Relationship between cation exchange capacity and oxalate extractable aluminium.

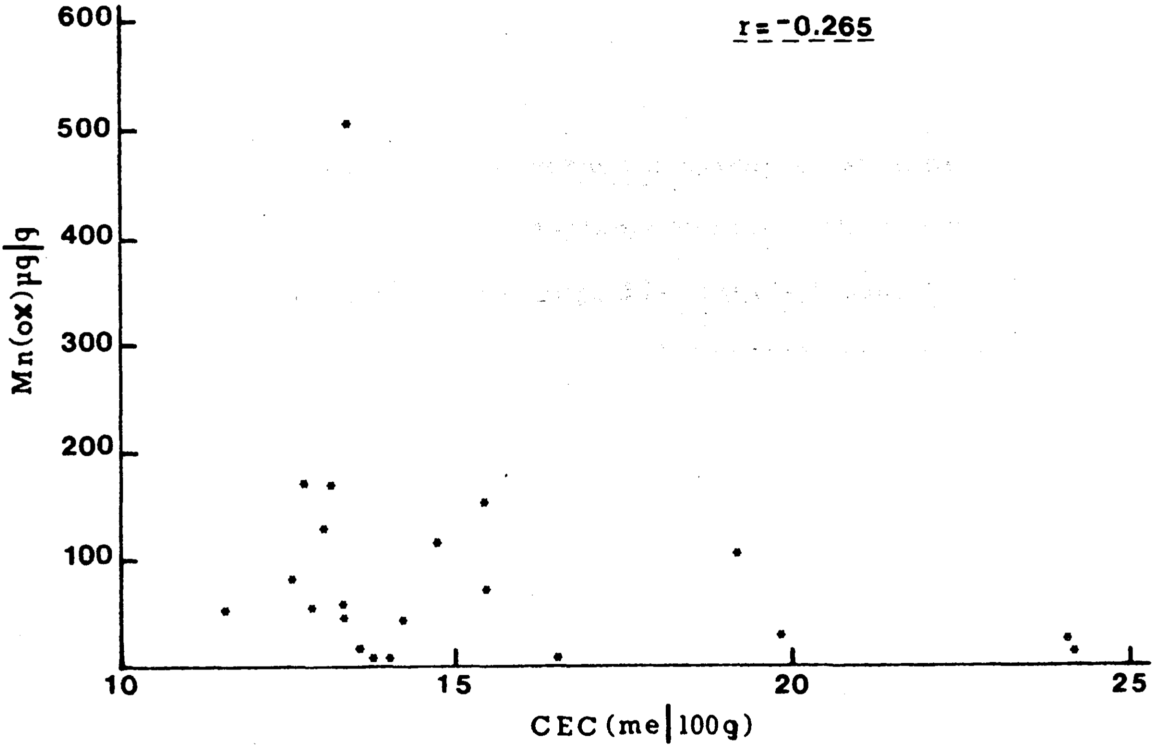


Figure 4.7. Relationship between cation exchange capacity and oxalate extractable manganese.

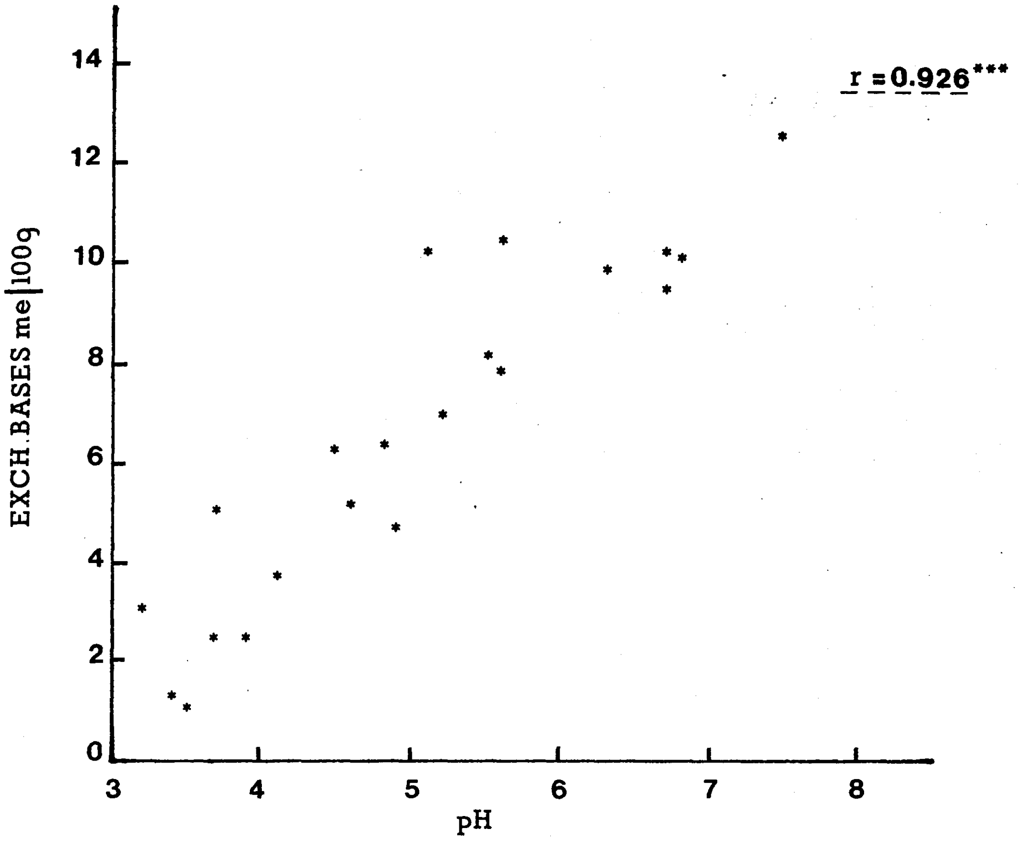


Figure 4.8. Relationship between spoil pH and exchangeable bases.

extractable aluminium (r = -0.608**, Fig. 4.10) and oxalate extractable manganese (r = 0.450*, Fig. 4.11). The correlation between exchangeable bases and pH indicates that, although the CEC of the spoil is affected greatly by the amorphous aluminium fraction, the level of bases actually held on the exchange complex is governed primarily by spoil pH; taking into account the level of oxalate extractable aluminium the partial correlation coefficient between pH and exchangeable bases is 0.880***.

This result indicates that although the aluminium oxide fraction occupies a significant proportion of the exchange sites, the negative charge on the exchange complex is strongly influenced by pH, lower pH giving a lower level of exchangeable Ca^{2+}, Mg^{2+}, K^+ and Na^+, these cations occupying the sites which are not involved in aluminium oxide adsorption.

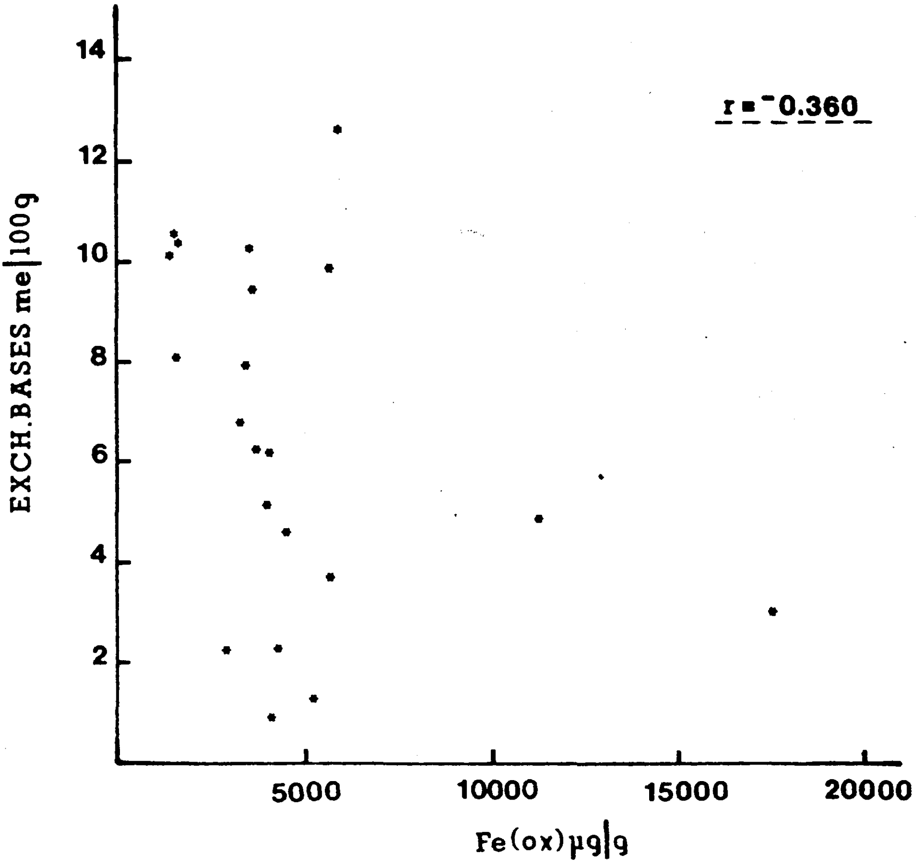


Figure 4.9. Relationship between oxalate extractable iron and exchangeable bases.

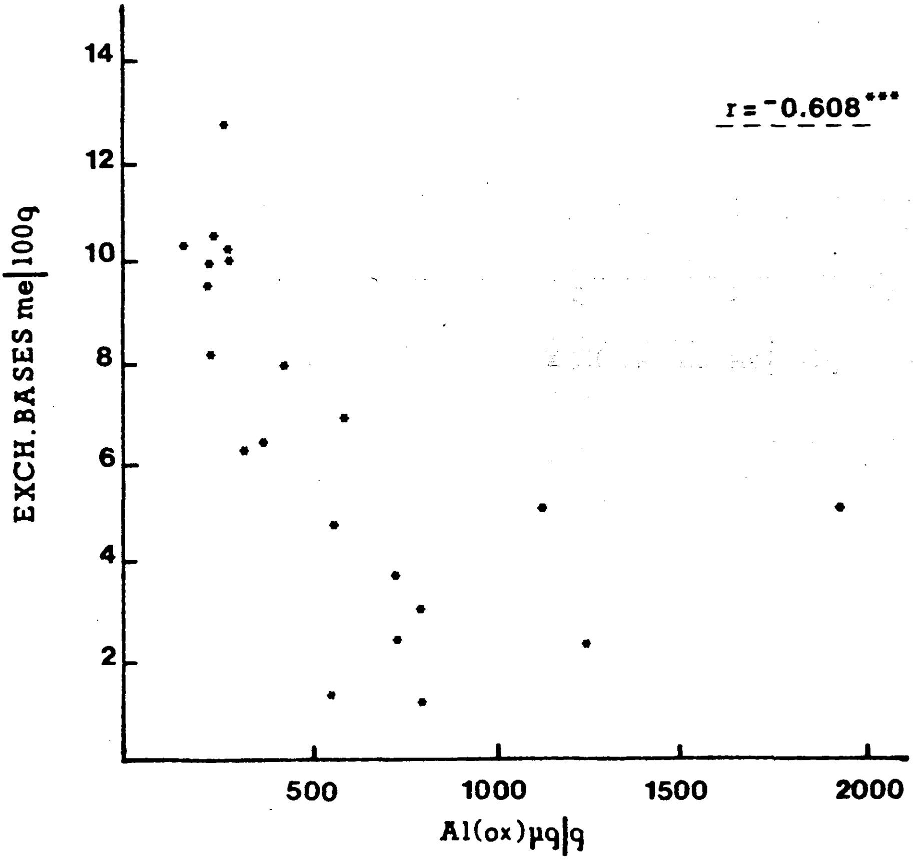


Figure 4.10. Relationship between oxalate extractable aluminium and exchangeable bases.

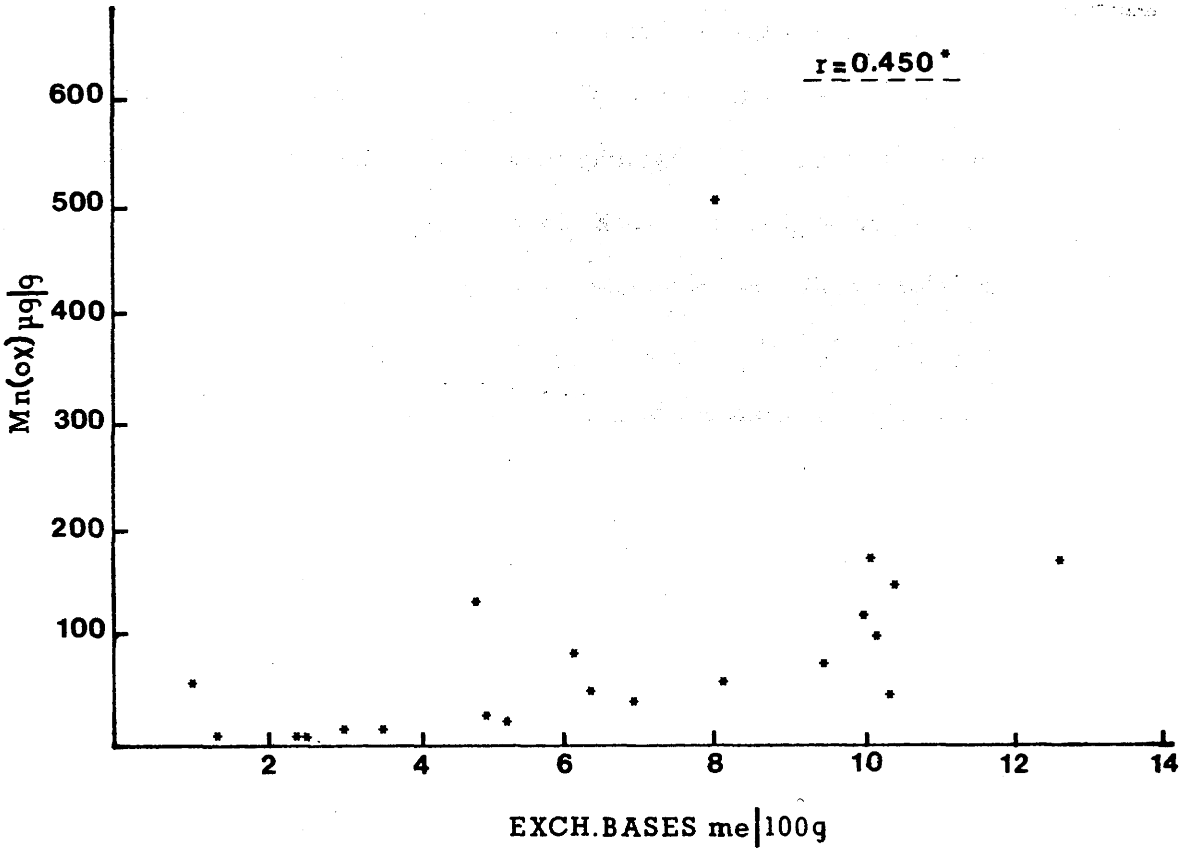


Figure 4.11. Relationship between oxalate extractable manganese and exchangeable bases.

The influence of the oxalate-extractable manganese fraction on the level of exchangeable bases contrasts with that of the iron and aluminium oxides, in that there is a positive correlation between the manganese oxides and exchangeable cations. Anderson et al. (1973), studying the adsorption of silver on poorly crystalline synthesized manganese oxides, found a surface exchange reaction between Ag^+ and Mn^2, K^+, Na^+ on the negatively charged oxide surface. Other workers have identified the negative charge on manganese oxides and their capacity to hold cations. Childs and Leslie (1977) found that Co, Ni and Zn showed a significant positive relationship with manganese concretions in a yellow-grey earth soil in New Zealand, while Kimber (1982) found a similar relationship with Co and Ni, and to a lesser extent Cu and Zn, in Scottish colliery spoil. The results in Table 4.5 indicate by the positive correlation between oxalate extractable manganese and exchangeable bases that the amorphous manganese oxide surface hold cations in an exchangeable form, and will therefore contribute towards the cation exchange capacity of the spoil. Figure 4.11 shows the variation in the level of exchangeable bases with oxalate-extractable Mn ($r = 0.450^*$). As this shows, one sample (SF22) has a particularly high Mn content (507 $\mu g/g$), and does not follow the pattern shown by the other samples. By not including SF22 in the correlation analysis of these two parameters the resulting correlation coefficient is 0.746^{***}, significant at the 0.1% level. It appears from this, therefore, that the relationship between the amorphous manganese fraction and exchangeable bases breaks down if the oxalate-extractable manganese level is exceptionally high.

Table 4.6. Level of exchangeable Ca^{2+}, Mg^{2+}, K^{+} and Na^{+} in a range of spoil samples

Sample	Exchangeable bases (me/100g)			
	Ca	Mg	K	Na
SF 5	7.73	1.98	0.36	0.27
10	1.53	0.64	0.16	0.15
12	0.31	0.18	0.16	0.35
17	10.17	1.68	0.31	0.43
22	5.30	1.90	0.35	0.35
WS 2	4.02	2.14	0.42	0.35
3	1.15	1.89	0.20	0.22
5	2.58	1.50	0.27	0.42
7	5.88	2.96	0.37	0.23
10	3.49	2.20	0.30	0.33
LG 6	1.30	0.44	0.34	0.33
10	0.47	0.30	0.17	0.39
SB 4	3.68	0.44	0.36	0.44
8	3.25	0.85	0.61	0.41
12	5.62	3.62	0.69	0.44
15	5.97	3.06	0.61	0.43
21	1.64	0.96	0.10	0.28
DK 2	4.85	2.41	0.43	0.43
4	4.21	5.17	0.30	0.32
8	5.96	3.02	0.49	0.42
10	3.32	2.06	0.31	0.43

Table 4.7. Average CEC, exchangeable base level and percentage base saturation of spoil samples

pH range	CEC (me/100g)	Exch. bases (me/100g)	Base saturation %
3-4	17.6	2.5	14.0
4-5	14.1	5.2	38.2
5-6	15.1	8.7	57.7
6-7	14.1	9.9	71.0
> 7	13.1	12.6	96.1

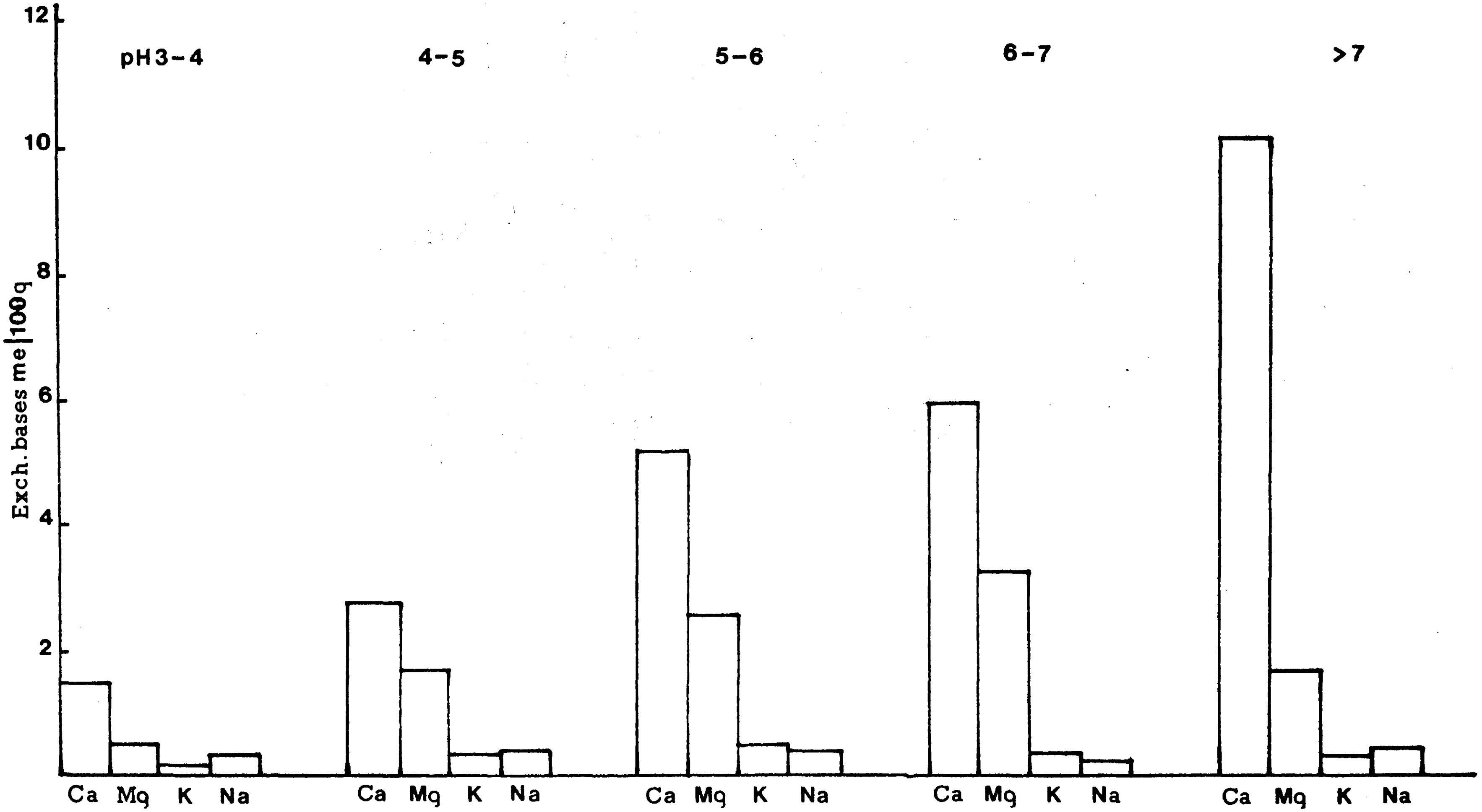


Figure 4.12. Data split into pH categories showing the levels of exchangeable Ca^{2+}, Mg^{2+}, K^{+} and Na^{+}.

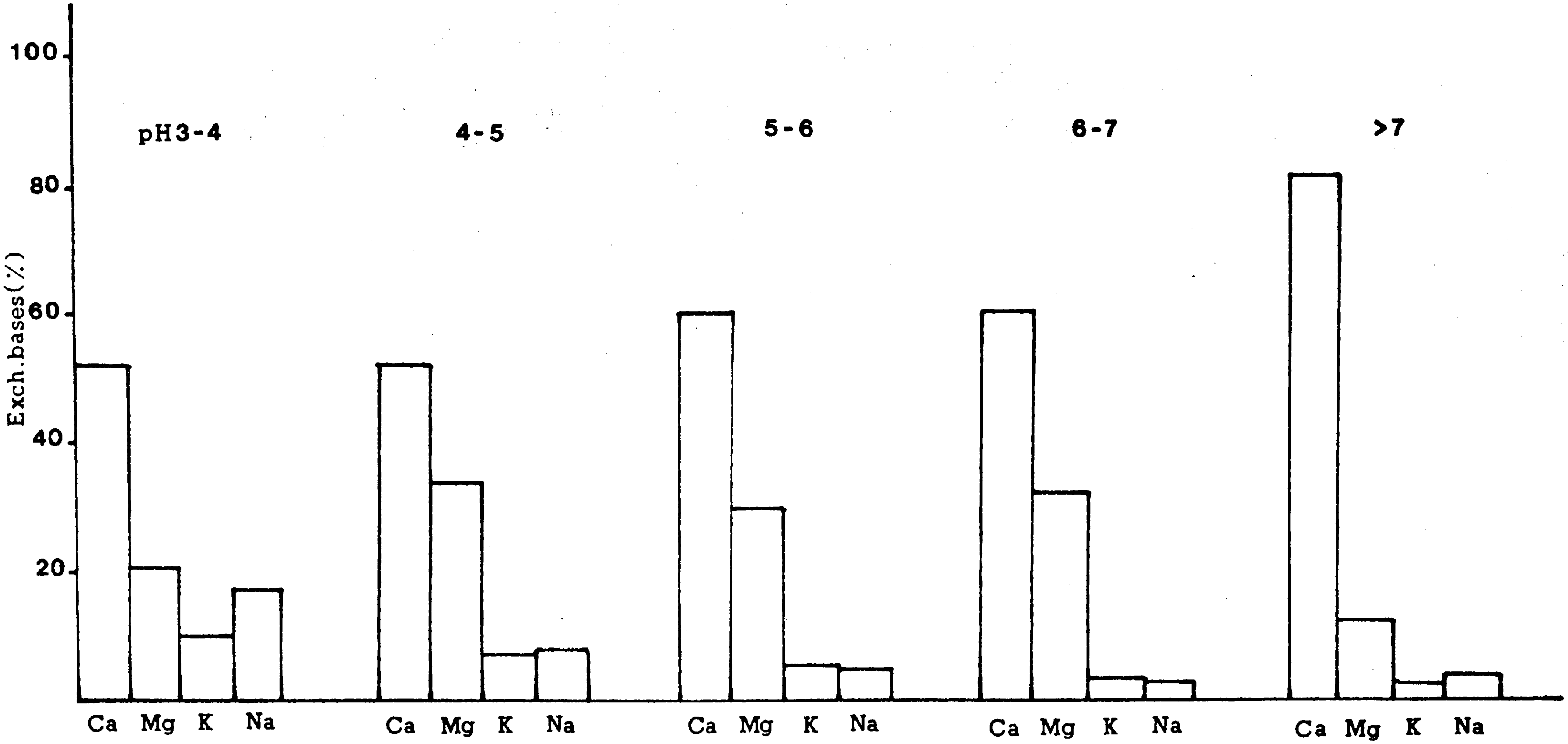


Figure 4.13. Levels of exchangeable Ca^{2+}, Mg^{2+}, K^{+} and Na^{+} expressed as a percentage of the total exchangeable base content.

The percentage base saturation of the spoil is significantly correlated with spoil pH ($r = 0.950$), indicating that as pH decreases H^+ replaces the adsorbed bases, with lower exchangeable base level resulting in a decrease in base saturation.

Table 4.6 shows the level of exchangeable Ca^{2+}, Mg^{2+}, K^+, and Na^+ in the exchangeable base pool.

In Fig. 4.12 this data is split into groups based on sample pH, and this shows that with increasing pH the amount of Ca^{2+} on the exchange complex increases, Mg^{2+} increases up to pH 7, while K^+ and Na^+ remain relatively constant. The proportion of the total exchangeable bases represented by each cation (shown as a percentage in Fig. 4.13) shows a marked consistency, particularly in the pH range 4-7, which suggests that as pH increases and more Ca^{2+} and Mg^{2+} ions are adsorbed (Fig. 4.12) they are taken up in equivalent amounts. The average CEC, exchangeable base level and percentage base saturation of the pH divisions are shown in Table 4.7. As these results show, the CEC remains fairly constant throughout the range, whereas the level of exchangeable bases and percentage base saturation increase with pH, this being a result of the increased adsorption of Ca^{2+} and Mg^{2+} at higher pH.

4.10 Summary

The negatively charged amorphous iron and aluminium oxide fractions are closely associated with cation exchange sites in colliery spoil, particularly the aluminium fraction, its adsorption masking the pH-dependent character of the exchange complex. Cation exchange capacity and exchangeable base data indicate that aluminium oxides occupy exchange sites and thereby reduce the effective cation exchange capacity of the spoil. The influence of the manganese oxides contrasts with that of the iron and aluminium fractions in that the cation exchange capacity and level of exchangeable bases are directly related to the manganese oxide content, i.e. cations are exchangeably adsorbed by the manganese fraction.

The level of bases held on the exchange sites is pH-dependent, this being mainly a reflection of the increased adsorption of the dominant Ca^{2+} and Mg^{2+} ions with pH. However, the relative proportions of Ca^{2+}, Mg^{2+}, K^+ and Na^+ on the exchange complex varies little with pH in the range pH 4-7.

These results emphasise the influence of the amorphous oxides of Fe, Aℓ and Mn on spoil properties, their effects having been highlighted by other workers with regard to adsorption-precipitation reactions; the iron and aluminium fractions having an important role in phosphate adsorption, and the manganese fraction having the capacity to adsorb trace metals.

CHAPTER 5

THE USE OF A SOIL COVER IN COLLIERY SPOIL RECLAMATION

5.1 INTRODUCTION

While it is recognised that colliery spoil is a poor growth medium, comparing poorly with an arable soil, most workers agree that it is not essential to use a soil cover in the reclamation of spoil, provided that careful spoil management practices are followed (Bradshaw et al. 1973, Doubleday 1973, Gemmell 1973).

In order to assess the success of reclamation schemes with and without the use of a soil cover, 13 sites were visited in Central Scotland over a three year period. At each site a visual assessment was made of the quality of the vegetation cover and its species composition, with a number of profiles being dug to allow examination of the nature and depth of soil cover (if present), rooting depth, and type of spoil material, with samples taken at various depths for laboratory analysis. The number of pits varied from site to site, depending on size and topography and was sufficient to give a true representation of the site as a whole. Four of the sites studied comprised of areas which had a soil cover and also part which received no soil, thus giving a comparison of the two systems side by side, with observations and samples taken in both areas.

5.2 Site Observations

5.2.1 Ground cover

The density of the surface cover of vegetation was estimated at each site and assigned a score on a scale shown in Table 5.1.

Table 5.1. Assessment of surface vegetation cover

Cover Score	Comments
1	Very poor ground cover, few growing plants, much of the surface bare
2	Poor cover, weak plant growth
3	Good cover, bare surface material still evident
4	Very good cover, good growth, little bare surface visible
5	Excellent growth, no surface material visible

5.2.2 Soil cover

If a soil cover was present its average depth over the site was determined.

5.2.3 Root growth

The density of the root population down each profile was estimated and described on the following scale:

Very abundant
Abundant
Common
Few
None

Each profile was thus divided into a number of "horizons", either on the basis of type of material or by the density of rooting, and a sample of each horizon was taken.

5.3 Laboratory Analysis

Each sample was air-dried in a force draught oven at 32°C.

5.3.1 pH

10g of air-dried material was shaken on a wrist shaker with 25 ml deionized water for 20 mins and the pH of the suspension determined.

5.3.2 Moisture content

20 g of sample was placed in a weighed porcelain crucible and dried at 105°C. The weight loss on drying was expressed as a % of the air-dried sample weight.

5.3.3 Soil texture

The texture of all soil samples was determined by mechanical analysis using ultrasonic vibration as the method of dispersion (Edwards and Bremner 1967).

5.4 Results

Each site was judged by three main criteria:

1. Vegetation quality and growth
2. Ground cover
3. Root growth

Details of each site are given in Appendix II which shows the location of each bing, the date of completion of site work, the seed mixture used at reclamation and a list of the species growing in 1979/80, together with an assessment of ground cover. A typical profile of each site is given in Table 5.4.

5.4.1 Vegetation quality

In most cases grass and clover growth is better when a soil cover is used, showing a healthier sward which does not suffer as severely during dry weather. Although no measurements of plant yield were carried out, there is a visible difference between the condition of the swards on sites reclaimed under both the soiled and non-soiled systems, and discussion with the landowners confirmed higher yield when a soil cover is used. Despite the higher yield under the soiled system it is still low when compared to a normal arable soil, yield reduction estimations ranging from 30-50% (personal communications) when comparing soiled sites with adjacent farmland (See Chapter 2, Plate 3). This indicates that the underlying spoil still has considerable influence on the plants growing on a reclaimed soiled site when a thin soil cover is used (See 5.5.3).

5.4.2 Ground cover

One of the main criteria used to assess the success of a site is the density on the surface of the vegetation cover. Sites such as those described in Appendix II which have an agricultural after-use are ultimately judged on the basis of yield for hay or silage production, or by the stocking rate which the land can support. It is therefore of little value if the ground cover is poor, since yield is governed significantly by the number of successful plants per unit area.

In most cases, sites having both a soiled and non-soiled area show better ground cover on the soiled part. The exception is Castlehill which has a comparable ground cover on both parts, due to the poor structure of the clay loam soil layer which becomes waterlogged during heavy rain, thereby causing poor root growth and a sparse cover, together with the fact that the unsoiled area had a higher proportion of legumes in the seed mixture.

5.4.3 Root growth

(a) Rooting on soiled sites

A study of root growth on sites with a soil cover shows that plant roots are not restricted to the soil layer, but can be found to a depth of 25 cm when a soil cover of only 10 cm is used. There is, in most cases, however, a marked reduction in root density on crossing the interface, indicating that roots tend to remain in the more favourable soil layer in preference to occupying the underlying spoil material. Observations made on sites having a variable depth of soil show that there is a relationship between the thickness of the soil layer and depth of root penetration, with a thicker soil cover generally inducing deeper rooting (Table 5.2).

(b) Rooting on unsoiled sites

A study of rooting depth on unsoiled sites shows that roots can penetrate to considerable depth, with deepest rooting being found on spoils having a loose, open structure, i.e. Bank, Annandale, North Lodge, where roots are found at a depth exceeding that found on any of the soiled sites (Table 5.3).

In both cases root nodulation of legumes was observed, indicating that nitrogen fixation occurs on both types of sites.

Table 5.4 shows a "typical" profile from each of the sites studied, based on observations made in a number of pits at each site with root density, pH and moisture content data.

5.5 Discussion

Vegetation quality and cover density are generally better when a soil cover is used due to the fact that the chemical, physical and biological properties of soil are on the whole more favourable for plant growth than the conditions in colliery spoil. A point of note when examining profiles on soiled sites was the total absence of worm activity in the spoil material although the soil layer often supported a significant worm population.

Table 5.2. The relationship between soil layer thickness and rooting depth

Site name	Soil thickness (cm)	Depth of rooting (cm)
Enterkine	9	9
	12	12
Seaforth	8	15
	15	28
	20	31
Dumbreck	10	15^{+} *
	16	28
Stane	6	20
	12	34
Castlehill	7	19
	10	26
Devon	4	12
	10	18
Jenny Gray	11	22
	15	25^{+}
Lochore Meadows Phase II	6	18
	13	15
	19	19^{+}
Lochore Meadows Phase IV	6	18
	8	22
	10	22^{+}
	14	26
Lochore Meadows Phase VI	7	20
	10	18
	11	20

* 15^{+} indicates the total depth of the profile, with roots found throughout, and possibly penetrating deeper.

Table 5.3. Depth of rooting on unsoiled sites (maximum depth at which roots were found)

Site name	Root depth (cm)
Bank	40
Annandale	39
Dumbreck	19
North Lodge	43
Stane	20
Castlehill	21

Table 5.4. Typical profile from each site

SITE	Soil type	Depth (cm)	Type of material	Roots	pH	Moisture content (%)
ENTERKINE	Sandy Clay Loam	0-2	Plant litter			
		2-6	Soil	V. Abundant	4.5	17.0
		6-10	"	Common	4.3	15.2
		10+	Coal waste	None	2.9	9.8
SEAFORTH	Clay Loam	0-2	Plant litter			
		2-4	Soil	V. Abundant	5.8	21.0
		4-8	"	Abundant	5.7	16.0
		8-15	Coal waste	Few	6.7	5.9
		15+	" "	None	6.8	5.1
BANK	Silty Clay Loam	0-5	Soil	V. Abundant	4.7	22.0
		5-18	Coal waste	Abundant	3.6	29.0
		18-31	" "	Few	4.2	10.4
		31+	" "	None	4.7	10.0
	None	0-2	Coal waste	V. Abundant	5.1	7.5
		2-18	" "	Abundant	5.2	9.7
		18-25	" "	Common	3.6	7.9
		25-34	" "	Few	4.2	8.3
		34+	" "	None	4.2	7.0
ANNANDALE	None	0-12	Coal waste	Common	5.4	15.9
		12-25	" "	Few	6.9	12.5
		25+	" "	None	7.0	11.5

Table 5.4 (contd.)

SITE	Soil type	Depth (cm)	Type of material	Roots	pH	Moisture content (%)
DUMBRECK	Loam	0-2	Plant litter			
		2-8	Soil	Abundant	5.4	21.0
		8-15	"	Common	5.2	13.3
		15-20	Coal waste	Few	6.4	9.8
		20+	" "	None	6.4	9.3
	None	0-1	Plant litter			
		1-10	Coal waste	Common	6.4	9.7
		10-19	" "	Few	6.2	7.0
		19+	" "	None	6.2	7.1
NORTH LODGE	None	0-2	Plant litter			
		2-17	Coal waste	V. Abundant	7.5	8.4
		17-38	" "	Common	7.7	7.6
		38+	" "	None	8.2	7.4
STANE	Sandy Loam	0-3	Soil	V. Abundant	7.0	18.1
		3-10	"	Abundant	6.8	25.2
		10-19	Coal waste	Abundant	7.3	9.6
		19-30	" "	Few	6.6	7.7
		30+	" "	None	6.6	7.0
	None	0-4	Coal waste	Abundant	7.3	9.0
		4-20	" "	Common	7.4	8.7
		20-32	" "	Few	7.1	9.6
		32+	" "	None	7.1	7.9

Table 5.4 (contd.)

SITE	Soil type	Depth (cm)	Type of material	Roots	pH	Moisture content (%)
CASTLEHILL	Clay Loam	0-10	Soil	Abundant	6.5	16.8
		10-20	Coal waste	Common	8.3	5.0
		20-26	" "	Few	8.4	4.8
		26+	" "	None	8.4	5.1
	None	0-1	Plant litter			
		1-20	Coal waste	Common	7.2	9.7
		20-25	" "	Few	7.0	5.8
		25+	" "	None	7.1	5.1
DEVON	Loam	0-8	Soil	V. Abundant	6.8	20.0
		8-13	Coal waste	Abundant	7.2	14.1
		13-20	" "	Few	7.4	11.4
		20+	" "	None	7.4	10.6
JENNY GRAY	Clay Loam	0-3	Soil	V. Abundant	6.1	14.7
		3-11	"	Abundant	5.8	9.7
		11-15	Coal waste	Abundant	6.1	8.3
		15-25	" "	Few	6.1	9.9
		25+	" "	None	6.4	8.9
LOCHORE MEADOWS II	Sandy Clay Loam	0-2	Soil	V. Abundant	6.2	26.0
		2-11	"	Abundant	6.0	11.5
		11-13	"	Few	5.6	8.5
		13-15	Coal waste	Few	6.4	6.5
		15+	" "	None	6.4	6.9

Table 5.4 (contd.)

SITE	Soil type	Depth (cm)	Type of material	Roots	pH	Moisture content (%)
LOCHORE MEADOWS IV	Sandy Clay Loam	0-5	Soil	V. Abundant	6.2	21.6
		5-8	Soil	Abundant	6.6	21.0
		8-17	Coal waste	Common	6.2	15.9
		17-26	" "	Few	6.3	11.6
		26+	" "	None	6.2	9.0
LOCHORE MEADOWS VI	Sandy Clay Loam	0-6	Soil	V. Abundant	7.5	17.8
		6-11	"	Abundant	6.8	14.8
		11-20	Coal waste	Few	5.5	13.2
		20+	" "	None	5.4	9.6

5.5.1 Conditions in soil v spoil

5.5.1.1 Cation exchange capacity

In general soil has a higher cation exchange capacity than spoil i.e. has a greater ability to hold nutrient cations such as Ca^{2+}, Mg^{2+} and K^+ in a form available to plants. A typical colliery spoil has a cation exchange capacity of $<$ 20 me/100g (Chapter 4) compared to an average value of 25-30 me/100g for a productive silt or clay loam soil (Bradshaw and Chadwick 1980), thus a soil is able to maintain a nutrient supply to plants, giving a higher plant survival rate. Fertilizer applied to a soil has a longer persistence in the root zone than that added to bare spoil, since the spoil's lower cation holding capacity is less able to prevent leaching losses. In order to support a vegetation cover on a spoil, comparable to that of a "normal" arable soil which has associated organic matter and faunal activity, it is necessary to apply more regular dressings of fertilizer, notably nitrogen and phosphorus, to maintain an adequate nutrient supply.

5.5.1.2 Water holding capacity

The water holding capacity of coarse-textured spoils is too low to maintain a constant supply of water in the root zone and may lead to drought stress, particularly in young plants with shallow rooting systems. The moisture content data in Table 5.4 show that, generally, the soil layer has a higher moisture content than underlying spoil. These figures cannot be used as a comparison between sites as sampling was carried out at different times and under different weather conditions, they are included simply as a comparison between profiles on each site and down each profile.

Decreasing moisture content with depth occurs on both soiled and non-soiled sites as a natural consequence of slow infiltration and a decrease in root density. However, the higher water holding capacity of the soil cover is evident, despite the fact that some spoils have a heavier texture than the soil layer above, e.g. Enterkine. In this case the main factor controlling water penetration is the compaction of the spoil before soil spreading, resulting in the soil layer becoming waterlogged during wet periods (Plate 9) which gives rise to poaching by grazing cattle.

5.5.1.3 Buffering capacity

The buffering capacity of a soil or spoil refers to its ability to resist a change in pH. This is related to the materials cation exchange capacity, since the presence of clay and organic matter increase cation exchange capacity and are also reservoirs of exchangeable hydrogen ions in equilibrium with the soil/spoil solution. Soil, which has a predominance of expanding clays such as bentonite in the clay fraction and may have a reserve of organic matter, will require more lime to effect a given change in pH than a spoil, which has a low organic matter content and more expanding clays such as illite. Coupled with this is the fact that a drop in pH of a limed soil is resisted by its buffering ability, while a limed spoil is subject to loss of lime through leaching, causing a decrease in pH. Production of hydrogen ions through biochemical change in the root zone or from the oxidation of iron pyrites will therefore result in a fall in spoil pH, while a soil cover is able to maintain a more constant pH in the upper part of the profile.

5.5.1.4 Acidity and soluble salts

Acid produced by the oxidation or iron pyrites reduces spoil pH and is injurious to plant roots, this problem being minimised by spoil compaction and covering with a soil layer. Acid attack of spoil material and water evaporation from the bare spoil surface leaves high salt concentrations in the root zone, which can result in the inability of the roots to extract sufficient water, causing wilting of the plants. Struthers (1964) found that plant establishment on colliery spoil was more successful if sown in the spring than in the autumn, the main factor being that winter rains washed out soluble salts from the root zone.

5.5.1.5 Surface temperature

Darker surfaces have a higher heat adsorption coefficient, resulting in uncovered sites having higher surface temperatures and evaporation rates than soiled sites (See Chapter 1) which can cause wilting during hot weather, while soil has a further advantage of retaining heat better than spoil during cooler weather. These factors together maintain a more constant temperature in the root zone of plants on soiled sites.

There are therefore many factors contributing towards the improved growth observed on soiled sites, and in summary it may be said that colliery spoil brings together many of the problems encountered in poor soils.

5.5.2 Root growth on soiled sites

Many workers have shown that fertilizer nitrogen and phosphorus together cause proliferation of plant roots in the fertilized zone, with an increase in the number of roots developing and greater branching of these roots (Duncan and Ohlrogge 1958, Russell 1973, Fitter and Bradshaw 1974, Robinson and Rorison 1985). The rate of growth of the primary roots is not significantly enhanced, but the increase in growth and branching of the smaller roots leads to an appreciable increase in the surface area of root tissue in contact with the soil and fertilizer, resulting in a higher rate of water and nutrient uptake. As a result a plant cover can be maintained adequately by this prolific root system exploiting a relatively small volume of soil. Since the soil used as a cover is fertilized at seeding, there are sufficient nutrients in this layer to induce beneficial root development and no need for roots to penetrate the underlying spoil in the early stages of growth, with the result that root density in the soil is considerably higher than that in the spoil.

Roots leaving a fertilized, well-structured soil layer cross the soil/spoil interface leaving a well-buffered and chemically favourable environment and enter a medium higher in soluble salts, exchangeable acidity and levels of trace metals, all of which act against root development and growth, e.g. on Enterkine very few roots were found in the spoil material because of low pH.

A factor which has great effect, causing a reduction in rooting across the interface is the physical nature of the spoil. Roots tend to stay in the soil layer simply because they are following the route which offers least resistance. Since the soil has a more friable structure than the compacted spoil, roots, on reaching the interface turn laterally and remain in the soil rather than continue to travel downwards. This factor alone discourages deeper rooting even when the spoil is not of particularly poor quality from the nutritional point of view and is free from toxic levels of trace metals. Evidence of the existence of a structural barrier can be seen clearly when a stone traversing the boundary is removed; roots are equally as abundant on both sides. This indicates that it is not simply a nutritional problem which is preventing or discouraging root penetration but a physical problem also, as roots find an easier route around a stone than they do moving directly into the compacted spoil. The greater depth attained by roots when a deeper soil layer is used is a result of a stronger rooting system developing in the soil before crossing the interface, thereby increasing the penetrating power of the roots.

5.5.3 Reclamation using a soil cover

If a soil cover is used, the normal practice after stone removal (stones greater than 10 cm diameter) is first to compact the spoil to give a firm, level surface before soil spreading. The advantages of compaction are, firstly, to provide a compact layer less liable to subsidence and secondly, to effectively seal the spoil from the atmosphere and expel most of the air, thus preventing, or at least slowing down, oxidation of iron pyrites which results in acid production (See Chapter 1). However, the fact that roots extend below the soil layer suggests that, with some improvement, the spoil could support a larger root population and thereby a vegetation cover less dependent on the soil layer alone.

It may be asked whether it is desirable to encourage deeper rooting, or if plants can be supported adequately by the soil layer alone, while keeping the influence of the spoil removed as far as possible. However, the ultimate aim of bringing disused land back into productive use can only be achieved by taking the spoil into account and considering the soil cover simply as an aid to reclamation and not as a means of burying the waste problem. If the layer of soil used is sufficiently thick and is of suitable quality, then it will effectively remove the influence of the spoil from the plants completely. This would not restrict the use of the reclaimed land to grass production alone, but would give land which could support high yielding arable species, and there would be little danger that such land would suffer regression through the influence of the spoil. Power et al. (1981) studying five crops (alfalfa, crested wheat-grass, native warm-season grasses and spring wheat) grown on varying thicknesses of suboil and topsoil over sodic mine spoil in North Dakota, found increases in crop yield with increasing soil layer thickness up to 75-120 cm, with greatest yield occurring when 20 cm of topsoil was placed over 55-110 cm of subsoil. However, due to short supply and high cost, the thickness of the soil layer normally varies from only 2.5 cm-15 cm and it is therefore impossible to ignore the spoil effects. If the vegetation cover could tap only those nutrient reserves in this narrow soil band it would require careful management and high fertilizer inputs to maintain a grass cover over a number of seasons, while continuous cropping would lead to downgrading of the soil structure and reduced crop yield.

Plant roots open up and improve the structure of soil and spoil, creating channels by which air and water can enter and through which gaseous exchange takes place. Roots grow only under aerobic conditions, which require the free entry of oxygen and the free passage of respiratory products away from the root surface, therefore, expulsion of air from the spoil and covering with a soil layer will reduce its suitability for root growth. Certain practices could be adopted when reclaiming spoil with a soil cover which would, in the long-term, create a deeper cultivation layer and a deeper root zone, thus approaching a situation more akin to a soil profile than simply a soil covered waste. If this could be achieved a crop rotation system could be introduced to continuously upgrade the land.

Some of the recommendations outlined in the following sections may be slightly more costly initially than the standard reclamation techniques in use. However, the long-term result would be the creation of land which is more self-sustaining, and not requiring periodic heavy applications of fertilizer which characterise many reclaimed sites.

5.6 Recommended Practices to Encourage Deeper Rooting when a Soil Cover is Used

5.6.1 Retaining an open structure of the spoil

Since plant roots cross the soil/spoil interface, provided there are no fundamental chemical or physical factors preventing them, root penetration would be aided by not compacting the spoil surface before soil spreading. Since the cost of reclamation is governed to a large extent by the number of times machines are put on the site, this practice would in fact reduce the cost of each project, while encouraging a slight degree of soil/spoil integration, making root penetration easier.

5.6.2 Addition of lime to the spoil

If the spoil is acid, by the incorporation of lime into the top 10-15 cm at a rate sufficient to raise the pH to 6.2-6.5 before soil spreading, rooting would be encourages in the following ways:

(a) As root growth is reduced under acid conditions, raising spoil pH would in itself encourage root growth. An example of the prevention of root growth by spoil acidity is shown by the almost complete absence of roots below the soil layer on Enterkine (spoil pH 2.2-3.1).

(b) By incorporating lime using a disc cultivator, heavy spoil structure would be improved.

(c) Lime influences the structure of heavy-textured spoils, bringing together clay particles as floccs or loose aggregates and reducing bulk density.

(d) By raising the spoil pH the availability of trace metals would be reduced, thus protecting the plants from possible trace metal toxicity, and the phosphate fixing capacity would also be reduced.

5.6.3 Addition of lime and fertilizer to the spoil

Addition of a small amount of compound fertilizer with the lime would give all the advantages of liming, and would give the plant cover a further aid by supplying it with a readily-available source of nutrients. Rooting is encouraged by fertilization of the root zone, and although the nutrients supplied would be used up rapidly (due to their ready availability), the more vigorous and extensive root system would exploit a larger volume of spoil. Once the roots had become established and used up the added fertilizer, the spoil may not be capable of supporting such a prolific root system due to its low nutrient status. However, even with a high proportion of the root increment due to fertilization dying back, structure would be improved and nutrients from the decomposition of the roots would be added.

5.6.4 Addition of organic matter to the spoil

Addition of an organic amendment such as farmyard manure, sewage sludge, peat or fresh plant residues would improve spoil structure of heavy-textured spoils, and increase the water holding capacity of free-draining spoil, with the breakdown of the organic matter supplying nutrients. Addition of a light dressing of lime with the organic matter would aid breakdown in acid spoils. Organic matter performs many functions in the soil which may be summarised as follows (Donahue *et al.* 1971):

(a) Plasticity and cohesion of heavy, clayey soil is reduced and drainage is improved.

(b) Water holding capacity of coarse, loose soil is increased.

(c) Granulation is encouraged.

(d) Cation exchange capacity is increased.

(e) Upon decomposition, organic matter supplies nutrients.

(f) Long continued use of high levels of manure can so improve soil structure that germination and seedling growth are greatly improved in difficult years.

(g) Microbial activity is encouraged, resulting in the production of microbial gums which improve the structure of coarse soils.

(h) Breakdown products such as citrates and oxalates combine with iron and aluminium, releasing adsorbed phosphate and reducing phosphate adsorption.

Since most spoils are low in mineralisable organic matter the benefits of organic matter addition will be greater than those gained in a soil.

5.7 Reclamation Without the Use of a Soil Cover

When there is no source of soil which can be economically used as a cover, the spoil material must be treated as a "soil" requiring careful management and additions of lime and fertilizer. The normal procedure adopted is to take representative samples for analysis in order to determine lime and fertilizer rates after regrading of the surface is complete. However, if samples were taken before regrading, many sites could be reclaimed more successfully by identifying unsuitable material for burial, and retaining weathered spoil for surface spreading, thus giving a greater chance of plant survival.

Incorporation of organic matter in the top 10-15 cm of the spoil would improve spoil conditions as described in section 5.6.4. Organic materials which could be used include farmyard manure, poultry manure, sewage sludge, peat and pulverised wood bark.

5.7.1 Animal wastes and sewage sludge

Farmyard manure contains about 75% moisture, and on average approximately half the nitrogen, almost all of the phosphorus and about 40% of the potassium are found in the solid part. However, the advantages of the solid form are offset by the ready availability of the nutrients in the liquid fraction giving slurry approximately the same nutritional value as the solid form.

Poultry manure is richer in nitrogen than most other bulky organic manures and the nitrogen is more readily available. It is usually applied mixed with bedding litter such as straw or wood shavings giving a friable product with approximately 30% moisture content.

Digested sewage sludge may be used either in a liquid or cake form. Liquid sludge contains 2-5% by weight of dry solids and has the advantage over the cake form (20-35% dry solids) in that the nutrients in the liquid are in a readily available form. Quality of sewage sludge varies greatly depending on the source, the main problem being the presence of high levels of trace metals in samples from industrial areas.

Each of these materials may therefore be applied as a liquid by spraying or in a solid form by a dung spreader, the liquid form supplying readily available nutrients and little organic matter, with the solid form adding organic matter with slower release of nutrients.

5.7.2 Peat and woodwastes

The content of plant nutrients and their availability in peat, wood shavings and wood bark are extremely low, and for this reason they may be considered solely as a source of organic matter, having a physical rather than a chemical effect. Incorporation of 10-20 tonnes/ha to a depth of 10-15 cm would improve structure but have a negligible effect on nutrient status. The carbon:nitrogen ratio of wood bark can vary from 40:1 to 200:1 and therefore little breakdown would occur in the spoil due to lack of nitrogen. Spoils with a heavy texture would benefit by a reduction in bulk density. However, the effect on coarse textured spoils would be to aggravate the problem of low water holding capacity, since these materials are high in lignin and cellulose and do not wet easily. In order to increase their wetability and to make them more effective as spoil amendments, these materials could first be composted before spoil incorporation.

From the point of view of adding organic matter and plant nutrients, compost and fresh organic residues are equally effective. However, breaking down of resistant organic materials has two main advantages, due to the action of bacteria and fungi in the compost heap. Firstly, plant cell wall material is broken down, releasing nutrients and retaining them in an available form until the compost is added to the spoil, and secondly, the friable residue left after cell wall destruction has a much higher water holding capacity. Composting of wood wastes would release virtually no plant nutrients, but the physical properties would be improved, and breakdown in the spoil would be more rapid provided that spoil conditions were favourable. A well decomposed compost could be made by mixing farmyard manure or poultry manure with a wood waste, active decay organisms supplied by the manure breaking down the organic matter in the presence of nitrogen, while the addition of lime to maintain a near neutral pH of the pile would also aid breakdown.

Since it is not always possible, due to bad weather or ground conditions, to utilize an organic amendment as it becomes available, composting offers a way of storing these materials until they can be used, while producing a more effective spoil conditioner during storage.

5.8 Changes in Species Composition of the Sward

Appendix II shows the seed mixture used on each site at reclamation and a list of the species found growing in 1979/80. No attempt was made to estimate the percentage composition of the sward, and this list simply shows those species which have survived or have invaded naturally. Any site, or part of a site, which required re-seeding since the first sward establishment was replanted with basically the same seed mixture as used initially, this being confirmed by the landowners, the only exception being Lochore Meadows Phase II, Inchgall Farm, where part of the site was sown with kale for strip-grazing by cattle - this scheme is discussed in Appendix II.

Table 5.5 lists the species used on all sites and shows the number of sites on which each has died out or appeared in the sward.

Table 5.5 shows that perennial ryegrass (Lolium perenne), timothy (Phleum pratense) and smooth-stalked meadow grass (Poa pratensis) are the most successful grass species planted, and although the survival rate of common bent (Agrostis tenuis) is low, it has naturally invaded both soiled and non-soiled sites. Cocksfoot (Dactylis glomerata), red fescue (Festuca rubra) and meadow grass (Festuca pratensis) survive best on soiled sites, while crested dog's-tail (Cynosurus cristatus) and rough-stalked meadow grass (Poa trivialis) are more successful on the non-soiled

Table 5.5. Number of sites on which each plant species was planted and found in the sward in 1979/80

Species	No. of sites planted	No. of sites where species died out	No. of sites where species invaded naturally	Comments
Perennial ryegrass (Lolium perenne)	13	2	-	Most commonly used species, survives on most sites
Timothy (Phleum pratense)	11	-	2	Survives on all sites, invades 2 naturally
Smooth-stalked meadow grass (Poa pratensis)	4	-	1	Survives on all sites, invades 1 soiled site
Common bent (Agrostis tenuis)	3	2	7	Naturally invades 7 sites
Cocksfoot (Dactylis glomerata)	7	4	1	Dies out on non-soiled site, invades 1 soiled site
Red fescue (Festuca rubra)	8	3	-	Survives best on soiled sites
Meadow fescue (Festuca pratensis)	5	2	-	Dies out on non-soiled site
Crested dogs' tail (Cynosurus cristatus)	5	4	-	Dies out on 4/5 soiled sites, survives on non-soiled site
Rough-stalked meadow grass (Poa trivialis)	4	3	-	Dies out on all soiled sites, survives on non-soiled sites
Brown top (Agrostis stolonifera)	4	4	-	Dies out on all sites
White clover (Trifolium repens)	7	-	4	Survives on all sites, invades 4 (3 soiled sites)
Red clover (Trifolium pratense)	6	2	2	Naturally invades 2 soiled sites
Birdsfoot trefoil (Lotus corniculatus)	1	1	-	Dies out on both soiled and non-soiled part of site
Alsike clover (Trifolium hybridum)	4	4	-	Dies out on all sites
Yorkshire fog (Holcus lanatus)	-	-	9	Not sown, naturally invades both types of site
Sweet vernal-grass (Anthoxanthum odoratum)	-	-	2	Not sown, naturally invades 2 soiled sites

sites. Brown top (Agrostis stolonifera) dies out on all sites. Of the legume species planted, white clover (Trifolium repens) and red clover (Trifolium pratense) survive well and invade naturally, particularly on soiled sites, while birdsfoot trefoil (Lotus corniculatus) and alsike clover (Trifolium hybridum) do not survive.

Of particular note is the natural invasion of Yorkshire fog (Holcus lanatus) and sweet vernal-grass (Anthoxanthum odoratum), particularly the former which comes in on both soiled and non-soiled sites. Yorkshire fog (Holcus lanatus) is a common, perennial grass found on rough grassland and waste land, including colliery spoil (Bradshaw and Chadwick 1980), but is not regarded as suitable for grazing. However, in view of its capacity to naturally invade colliery spoil its use in seed mixtures as a means of establishing an initial cover must be considered important.

5.9 Summary

Before commencing work on earthmoving it is essential that spoil samples from each area of the site are analysed, whether or not a soil cover is used. Laboratory analysis, together with a visual assessment of the site before regrading will identify areas which may present a problem for plant growth, such as:

(a) Very acid material

(b) Spoil which has a particularly high iron pyrites content

(c) Areas which have a structural problem which could hinder plant growth, e.g. fines or coal washings from settling lagoons.

(d) Areas of rubble or debris.

By identifying such areas, allowance can be made at the planning stage to ensure that this material is buried, to remove its influence from the plants, while material suitable for plant growth should be removed for re-spreading as a surface material, or if a soil cover is used, as material immediately below the soil layer.

Based on the evidence collected from 13 sites in the Central Scottish Coalfield, it can be concluded that the use of a soil cover gives better ground cover and plant growth than treatment of the bare spoil. However, on some soiled sites, if consideration had been given to the spoil at reclamation, better results could have been achieved. For example, the pyritic waste on Bank and Enterkine is affecting the soil cover, making liming and re-seeding necessary. Liming of the spoil before soil spreading would slow down the rate of pyrite oxidation and maintain a higher pH of the spoil, thus reducing the damage to the soil layer. Applying a top dressing of lime to the soil to repair damaged areas has little effect on the underlying spoil which may continue to oxidise and produce acid for many years. This example illustrates the importance of spoil analysis if a soil cover is used and shows the influence that spoil can have on plant growth.

The depth of rooting on soiled sites is related to the thickness of the soil layer, but root penetration is discouraged by the physical and chemical properties of the spoil. In order to encourage deeper rooting, which leads to a reduced dependence of the vegetation cover on the soil layer and give a cover less prone to drought, certain practices are outlined which require little extra expense, and the effects of which would be long lasting.

If there is no soil available, the alternative practice used in the past has been the amendment of the spoil with lime and fertilizer, with little attention given to its organic content. Since the addition of organic matter to soil is a standard practice in an arable system, due to an understanding of its beneficial effects on soil conditions and plant growth, and as the level of mineralisable organic matter in spoil is far lower than that of cultivated topsoil, it must be asked why this aspect of spoil management has been neglected for so long. There are a number of cheaply available organic materials which would ameliorate both the physical and chemical properties of the spoil, improving spoil conditions and yield.

When reclaiming without the use of soil, material which is removed for use as a surface layer could be improved greatly during storage through the use of lime, fertilizer, organic matter or compost. Spreading the amended spoil as a 15 cm surface layer over limed, uncompacted spoil would give the grass and clover considerable help at establishment, inducing better root growth and giving an improved surface cover which would protect the site from damage through run-off and poaching.

APPENDIX I

The following is a listing of pH, Tamm-extractable Fe, Aℓ, Mn, NH_4F-extractable phosphate and phosphate adsorption capacity of colliery spoil samples from 6 sites in the Central Scottish coalfield.

SITE - SAINT FLANNANS (SF)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
SF 1	8.0	1780	400	41	3.7	6.7
2	8.0	1800	308	39	3.0	6.4
3	3.7	5110	403	36	3.1	13.4
4	8.4	2130	321	27	4.3	7.1
5	6.7	1560	151	46	10.3	6.7
6	5.0	3510	351	51	10.2	8.7
7	4.5	6150	615	12	1.8	11.7
8	3.6	6100	679	5	1.9	11.2
9	3.6	5560	699	5	2.0	10.6
10	3.7	4395	725	7	4.1	9.0
11	3.8	5240	900	151	4.6	9.9
12	3.5	4190	800	56	4.5	8.8
13	3.7	4760	825	88	4.4	9.5
14	3.8	4590	800	82	4.8	8.7
15	3.9	4560	865	512	3.9	10.0
16	3.7	4220	880	76	6.4	8.9
17	7.5	5780	275	172	1.1	6.3
18	7.2	2840	900	230	7.3	6.8
19	8.2	2485	505	322	2.3	6.6
20	8.2	2855	510	371	2.9	7.5
21	7.6	2065	280	448	2.2	6.4
22	5.6	3290	425	507	8.0	9.4

SITE - WATERSIDE (WS)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
WS 1	5.7	1950	267	15	4.5	5.0
2	5.2	3160	584	43	13.3	7.8
3	4.1	5590	723	17	17.2	8.0
4	4.5	3520	514	22	10.9	7.5
5	4.9	4480	564	131	15.0	8.0
6	5.5	3480	404	134	17.0	8.6
7	6.7	3570	230	74	8.7	7.1
8	4.8	3960	408	68	15.1	7.5
9	5.1	3870	329	101	12.1	7.1
10	4.8	3580	383	51	12.6	6.5

SITE - LOGANLEE (LG)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
LG 1	3.1	3540	775	2	3.8	11.7
2	2.9	6360	535	1	0.2	13.7
3	3.6	4350	1590	5	4.8	12.3
4	3.5	3200	1840	3	4.9	12.8
5	3.8	2970	1610	4	3.3	11.5
6	3.9	3030	1240	8	4.0	10.8
7	3.2	6760	75	3	0.5	13.1
8	3.2	9230	524	6	0.1	13.9
9	3.5	5320	712	7	5.4	10.8
10	3.4	5140	550	5	4.1	11.9

SITE - LARKHALL (LH)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
LH 1	3.4	2800	380	3	n.d.*	13.4
2	3.4	3100	440	5	n.d.	12.8
3	3.9	2600	790	23	n.d.	12.7
4	3.7	2800	710	26	1.4	12.3
5	3.7	4100	1650	49	3.1	12.6
6	3.5	4400	940	16	0.6	12.9
7	3.2	3400	720	8	n.d.	13.2
8	3.7	3500	1010	24	9.9	11.4
9	3.2	5700	560	11	2.5	12.9
10	5.5	1040	422	225	12.2	5.1

SITE - STONEYBURN (SB)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
SB 1	5.6	2050	390	62	16.2	4.0
2	4.4	4050	550	40	8.2	8.5
3	4.6	3650	750	41	16.6	8.3
4	3.7	11300	1930	28	3.1	12.0
5	3.9	13300	180	7	3.7	12.9
6	3.0	14000	430	8	1.7	12.3
7	3.9	5650	1300	10	4.8	10.9
8	4.6	3950	1160	26	9.6	10.6
9	4.6	3900	520	31	7.5	8.7
10	5.0	3600	1560	27	12.2	10.4
11	5.2	2100	640	56	19.3	7.2
12	5.6	1450	240	151	7.6	6.6
13	3.2	4100	270	3	1.6	12.2
14	3.7	1950	490	3	2.8	11.8
15	5.1	3350	280	102	7.0	8.6
16	5.2	2300	440	69	12.2	6.6
17	5.5	2000	390	60	11.0	13.9
18	5.3	2600	610	41	16.7	8.7
19	4.5	3750	1210	56	10.9	11.7
20	5.3	3100	620	58	11.2	8.9
21	3.2	17500	790	12	0.9	14.1
22	3.0	11700	580	21	2.1	12.4
23	3.4	15500	600	14	5.8	14.6
24	3.6	10700	730	21	3.8	11.9

SITE - DYKEHEAD (DK)

SAMPLE	pH	Ammonium oxalate extractable (μg/g)			Extractable P (μg/g)	P-adsorption (mg/100g)
		Fe	Aℓ	Mn		
DK 1	7.6	1985	340	150	7.0	6.5
2	5.5	1445	230	59	5.7	4.1
3	6.7	1620	274	179	10.8	5.9
4	6.8	1440	285	175	6.2	6.4
5	4.6	900	321	90	3.1	6.8
6	4.8	1380	194	77	6.6	5.3
7	5.5	2520	212	125	6.8	5.0
8	6.3	5650	232	122	0.6	6.1
9	6.7	2540	151	114	0.5	4.7
10	4.5	4100	315	82	6.9	6.7

APPENDIX II - DETAILS OF SITES SURVEYED IN CHAPTER 5

SITE NAME ; Enterkine

LOCATION : Mossblown Farm, Annbank, Strathclyde

DATE OF COMPLETION : July 1975

SOIL COVER : 7 - 15 cm

SOIL TYPE : Sandy clay loam

FERTILIZER TREATMENT : 510 kg/ha of 20:10:10 compound fertilizer
380 kg/ha of superslag (0:16:0)

LIMING RATE : 2.5 tonnes/ha

SEEDING RATE : 70 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	35
Red fescue (*Festuca rubra*)	23
Timothy (*Phleum pratense*)	20
Smooth-stalked meadow grass (*Poa pratensis*)	10
Brown top (*Agrostis stolonifera*)	5
White clover (*Trifolium repens*)	7

SPECIES PRESENT 1979/80

Perennial ryegrass (Lolium perenne)

Red fescue (Festuca rubra)

Timothy (Phleum pratense)

Smooth-stalked meadow grass (Poa pratensis)

Yorkshire fog (Holcus lanatus)

Common bent (Agrostis tenuis)

Sweet vernal (Anthoxanthum odoratum)

White clover (Trifolium repens)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-2	PLANT LITTER			
2-6	SOIL	VERY ABUNDANT	4.5	17.0
6-10	"	COMMON	4.3	15.2
10+	COAL WASTE	NONE	2.9	9.8

USE: Cattle grazing with some grass cut for hay and silage

COVER ASSESSMENT: 4 (Plate 7)

COMMENTS:

Although the vegetation cover is generally good, there are some bare patches due to the oxidation of pockets of pyritic material causing a reduction in pH and the emergence of iron-enriched water at the surface (Plate 8). These bare patches are due to uneven spreading

PLATE 7 - ENTERKINE - Surface

PLATE 8 - ENTERKINE - Evidence of pyrite oxidation in underlying spoil affecting soil cover.

of the soil layer or poaching by animals, resulting in exposure of the acid coal waste, and can be treated by additions of lime and fertilizer and re-seeding. Lime should not be added as a top dressing but should be incorporated into the soil layer. The very acid nature of the coal waste is clearly influencing the fairly shallow soil layer, bare patches having surface pH values of 2.3-2.8 compared to 4.8-5.3 on adjacent, vegetated areas. Rooting is confined solely to the soil layer. This site is an example of where liming of the spoil before soil spreading would have been beneficial.

Periods of heavy rain cause saturation of the soil layer which leads to poaching by animals around gates and troughs and to some movement of the soil layer on the slopes (Plate 9). Poaching during the winter of 1979 was severe but damaged areas recovered well the following year. Water draining through the soil layer does not penetrate the underlying spoil but flows down-slope along the soil/spoil interface and can be seen emerging at an exposed bank causing erosion of the bank (Plate 10) and contamination of a stream running along the edge of the site.

The liming rate used on this site was very low and the spoil is evidently pyritic, causing damage to the soil layer, resulting in destruction of the vegetation cover.

PLATE 9 - ENTERKINE - Damage to soil layer.

PLATE 10 - ENTERKINE - Eroded bank showing saturated soil layer.

SITE NAME : Seaforth

LOCATION : New Cumnock, Strathclyde

DATE OF COMPLETION : August 1978

SOIL COVER : 8 - 20 cm

SOIL TYPE : Clay loam

FERTILIZER TREATMENT : 510 kg/ha of 20:10:10 compound fertilizer

: 380 kg/ha superslag (0:16:0)

LIMING RATE : 3.5 tonnes/ha

SEEDING RATE : 65 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	30
Red fescue (*Festuca rubra*)	20
Timothy (*Phleum pratense*)	20
Rough-stalked meadow grass (*Poa trivialis*)	10
Smooth-stalked meadow grass(*Poa pratensis*)	7
Brown top (*Agrostis stolonifera*)	5
Red clover (*Trifolium pratense*)	4
White cover (*Trifolium repens*)	4

SPECIES PRESENT 1979/80

Perennial ryegrass (Lolium perenne)

Red fescue (Festuca rubra)

Timothy (Phleum pratense)

Smooth-stalked meadow grass (Poa pratensis)

Yorkshire fog (Holcus lanatus)

White clover (Trifolium repens)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-2	PLANT LITTER			
2-4	SOIL	VERY ABUNDANT	5.8	21.0
4-8	"	ABUNDANT	5.7	16.0
8-15	COAL WASTE	FEW	6.7	5.9
15+	" "	NONE	6.8	5.1

USE: Cattle grazing

COVER ASSESSMENT: 4

COMMENTS:

Generally the growth of grass and clover is good (Plate 11) with good surface cover. The regraded surface forms a series of low ridges with drainage channels between them. The top and slopes of the ridges are well-drained and support good growth, but during wet weather the lower areas become waterlogged and the drainage channels fill with

PLATE 11 - SEAFORTH - General view.

PLATE 12 - SEAFORTH - Waterlogging in low-lying areas.

water due to the impermeability of the spoil (Plate 12). As a result of this, and the heavy texture of the soil, damage due to poaching occurs. Although the underlying spoil is very compacted, with low moisture content, roots are found to 31 cm.

SITE NAME : Bank

LOCATION : New Cumnock, Strathclyde

DATE OF COMPLETION : 1975

SOIL COVER : NONE

SOIL TYPE : -

FERTILIZER TREATMENT : 380 kg/ha of 20:10:10 compound fertilizer
380 kg/ha superslag (0:16:0)

LIMING RATE : NIL

SEEDING RATE : 90 kg/ ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	30
Red fescue (*Festuca rubra*)	20
Timothy (*Phleum pratense*)	20
Smooth-stalked meadow grass (*Poa pratensis*)	7.5
Rough-stalked meadow grass (*Poa trivialis*)	7.5
Brown top (*Agrostis stolonifera*)	5
Red clover (*Trifolium pratense*)	5
White clover (*Trifolium repens*)	5

SPECIES PRESENT 1979/80

Red fescue (Festuca rubra)

Timothy (Phleum pratense)

Smooth-stalked meadow grass (Poa pratensis)

Common bent (Agrostis tenuis)

Yorkshire fog (Holcus lanatus)

Red clover (Trifolium pratense)

White cover (Trifolium repens)

PROFILE:

DEPTH (cm)	MATERIAL		ROOTS	pH	MOISTURE CONTENT (%)
0-2	COAL	WASTE	VERY ABUNDANT	5.1	7.5
2-18	"	"	ABUNDANT	5.2	9.7
18-25	"	"	COMMON	3.6	7.9
25-34	"	"	FEW	4.2	8.3
34+	"	"	NONE	4.2	7.0

USE: Cattle grazing

COVER ASSESSMENT: 2 (Plate 13)

COMMENTS:

This site received no lime at reclamation and seed was applied by hydroseeding; seed is sprayed on the surface with a mulch in a thixotropic gel with no physical incorporation into the spoil.

PLATE 13 - BANK - Surface (no soil).

PLATE 14 - BANK - Damage to soil layer and vegetation cover due to pyrite oxidation in underlying spoil.

There are a lot of coal, shale and rock fragments in the spoil giving good drainage properties and an open structure for root penetration (maximum depth 40 cm). However, despite a high seeding rate and low stocking rate ground cover is poor, possibly due to the stoney, coarse surface and low survival rate of germinated seed due to the seeding method used. There are some bare patches due to localised acid generation, pH values as low as pH 3.3 being found, with adjacent vegetated areas having pH 4.0-4.6. There is poor clover growth on much of the site due to spoil acidity. Bare slopes have no protection from run-off and erosion gullies have developed.

A small part of this site received a 5 cm cover of silt loam soil with the same seed mixture, application method, seeding and fertilizer treatments as the unsoiled area.

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-5	SOIL	VERY ABUNDANT	4.7	22.0
5-18	COAL WASTE	ABUNDANT	3.6	29.0
18-31	" "	FEW	4.2	10.4
31+	" "	NONE	4.7	10.0

USE: Cattle grazing

COVER ASSESSMENT: 3

COMMENTS:

At reclamation the pH of the soil was 7.4 and no lime was applied. However the acidic spoil has caused a fall in the pH of the soil layer. As on the unsoiled area clover growth is poor and the grass cover has been killed in many parts due to the acid conditions (Plate 14). The moisture content of the upper layers is higher than on the unsoiled area resulting generally in better plant survival and ground cover.

SITE NAME : Annandale

LOCATION : Kilmarnock, Strathclyde

DATE OF COMPLETION : May 1974

SOIL COVER : NONE

SOIL TYPE : -

FERTILIZER TREATMENT : 380 kg/ha of 25:10:10 compound fertilizer
510 kg/ha superslag (0:16:0)

LIMING RATE : NONE

SEEDING RATE : 90 kg/ha

SEED MIXTURE :

Species	% by weight
Brown top (*Agrostis stolonifera*)	30
Perennial ryegrass (*Lolium perenne*)	25
Red fescue (*Festuca rubra*)	20
Rough-stalked meadow grass (*Poa trivialis*)	17
Red clover (*Trifolium pratense*)	8

SPECIES PRESENT 1979/80

Perennial ryegrass (*Lolium perenne*)

Rough-stalked meadow grass (*Poa trivialis*)

Timothy (*Phleum pratense*)

Common bent (*Agrostis tenuis*)

Yorkshire fog (*Holcus lanatus*)

White clover (*Trifolium repens*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-12	COAL WASTE	COMMON	5.4	15.9
12-25	" "	FEW	6.9	12.5
25+	" "	NONE	7.0	11.1

USE: Grassland

COVER ASSESSMENT: 3

COMMENTS:

Vegetation cover is sparse with moss covering much of the surface where grass and clover are absent, indicating moist surface conditions over prolonged periods. The material over the entire site is fine washings or tailings with few stones or coal fragments. The grass cover on the south facing slope and on top of the main mound of the site did not survive the first summer. The bare patches were re-seeded in September of that year but the vegetation subsequently died. Samples of the coal waste taken from the bare patches in 1979/80 had pH values in the range 2.4-2.7 indicating acid generation in the spoil. At that time the site had received no lime or fertilizer treatment since 1974, hence the failure of re-seeded areas.

SITE NAME : Dumbreck

LOCATION : Queenzieburn, Strathclyde

DATE OF COMPLETION : 1974

SOIL COVER : NONE

SOIL TYPE : -

FERTILIZER TREATMENT : 380 kg/ha SAI lawn food No.4 (9.5:9.5:13.2)

LIMING RATE : NONE

SEEDING RATE : 90 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	20
Red fescue (*Festuca rubra*)	15
Common bent (*Agrostis tenuis*)	10
Crested dogs'-tail (*Cynosurus cristatus*)	10
Rough-stalked meadow grass (*Poa trivialis*)	10
Smooth-stalked meadow grass (*Poa pratensis*)	10
Meadow fescue (*Festuca pratensis*)	5
Cocksfoot (*Dactylis glomerata*)	5
White clover (*Trifolium repens*)	10
Birdsfoot trefoil (*Lotus corniculatus*)	5

SPECIES PRESENT 1979/80

Perennial ryegrass (Lolium perenne)

Common bent (Agrostis tenuis)

Crested dogs'-tail (Cynosurus cristatus)

Smooth-stalked meadow grass (Poa pratensis)

White clover (Trifolium repens)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-1	PLANT LITTER			
1-10	COAL WASTE	COMMON	6.4	9.7
10-19	" "	FEW	6.2	7.0
19+	" "	NONE	6.2	7.1

USE: Sheep grazing

COVER ASSESSMENT: 3 (Plate 15)

COMMENTS:

The quality of the vegetation (grass and clover) is poor and in many parts mosses and lichens have taken over from the grasses. The vegetation mat is not well anchored in the spoil as the roots are restricted mainly to the surface layers. This may be due to the combination of the seeding method (hydroseeding) which leaves the seed on the surface at sowing and the compacted, stoney nature of the spoil. Due to the lack of deep rooting the vegetation suffers from a shortage of water during dry weather, while heavy rain leads to saturation of the surface litter and moss cover, which has resulted in erosion gullies on some slopes.

There is a general nutrient deficiency on this site, particularly of nitrogen, shown by the yellow colour of the grass.

Part of the site received a 12-16 cm cover of loam soil with liming rate, fertilizer treatment and seeding rate the same as the unsoiled part. The seed mixture used was essentially the same as the main part of the site, with a higher proportion of fescues and bent grasses and a reduction in the proportion of ryegrass and meadow grasses.

SPECIES PRESENT 1979/80

Red fescue (Festuca rubra)
Common bent (Agrostis tenuis)
Smooth-stalked meadow grass (Poa pratensis)
Cocksfoot (Dactylis glomerata)
Timothy (Phleum pratense)
Yorkshire fog (Holcus lanatus)
Sweet vernal (Anthoxanthum odoratum)
White clover (Trifolium repens)

PLATE 15 - DUMBRECK - Surface (no soil).

PLATE 16 - DUMBRECK - Profile showing accumulation of plant litter.

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-2	PLANT LITTER			
2-8	SOIL	ABUNDANT	5.4	21.0
8-15	"	COMMON	5.2	13.3
15-20	COAL WASTE	FEW	6.4	9.8
20+	" "	NONE	6.4	9.3

USE: None

COVER ASSESSMENT: 5

COMMENTS:

This part of the site has been fenced off to prevent sheep grazing and some trees have been planted. Lack of grazing or cutting has resulted in grass 60-80 cm tall and an accumulation of 2-4 cm of plant litter on the surface (Plate 16). The soil used is a well-structured loam and earthworms are common in the soil layer. The well established vegetation cover is deep rooted (maximum depth 28 cm) and shows no ill-effect during dry weather. There is little chance that this part of the site will suffer regression due to nutritional or physical factors.

SITE NAME : North Lodge

LOCATION : Motherwell, Strathclyde

DATE OF COMPLETION : 1977

SOIL COVER : NONE

SOIL TYPE : -

FERTILIZER TREATMENT : 450 kg/ha of 20:10:10 compound fertilizer

LIMING RATE : 10 tonnes/ha

SEEDING RATE : 60 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	43
Red fescue (*Festuca rubra*)	37
Common bent (*Agrostis tenuis*)	10
Timothy (*Phleum pratense*)	10

SPECIES PRESENT 1979/80

Perennial ryegrass (*Lolium perenne*)

Red fescue (*Festuca rubra*)

Timothy (*Phleum pratense*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-2	PLANT LITTER			
2-17	COAL WASTE	VERY ABUNDANT	7.5	8.4
17-38	" "	COMMON	7.7	7.6
38+	" "	NONE	8.2	7.4

USE: Cattle grazing

COVER ASSESSMENT: 4 (Plate 17)

COMMENTS:

The grass on this site is well established despite the absence of legumes in the seed mixture. There is a well-developed root system, the maximum rooting depth being 43 cm, and there is no evidence of acid generation. The surface is extremely hard and the underlying material is very difficult to dig due to compaction. Drainage, however, is good and no water lies on the surface after heavy rain. During extended dry periods the vegetation cover has a poor appearance due to shortage of water.

A small area of this site received a different seed mixture and was used for tree planting. Seeding rate, lime and fertilizer treatments were the same as the main part of the site.

PLATE 17 - NORTH LODGE - Surface.

PLATE 18 - NORTH LODGE - General view showing vigorous clover growth.

Seed mixture in tree planting area

Species	% by weight
Red fescue (*Festuca rubra*)	65
Common bent (*Agrostis tenuis*)	8
Smooth-stalked meadow grass (*Poa pratensis*)	7
Cocksfoot (*Dactylis glomerata*)	7
White clover (*Trifolium repens*)	8
Red clover (*Trifolium pratense*)	5

COVER ASSESSMENT: 3

One season after seeding, during which grass height was cut to 7.5 cm as required, the following tree species were planted 1.5 m. apart in rows, with 1.5 m. between rows.

Norway maple (*Acer platanoides*)
Alder (*Alnus glutinosa*)
Willow (*Salix caprea*)
Birch (*Betula pendula*)

All trees were 60-90 cm tall with an established root system at planting. Each tree received 60g of a compound granular fertilizer lightly worked in at planting and a further two applications in the first year. The grass in this area benefits from the inclusion of clover which is growing vigorously (Plate 18) although ground cover is poorer than on the main part of the site.

SITE NAME : Stane

LOCATION : Shotts, Strathclyde

DATE OF COMPLETION : 1976

SOIL COVER : 6 - 12 cm

SOIL TYPE : Sandy loam

FERTILIZER TREATMENT : 500 kg/ha of 12:24:12 compound fertilizer
250 kg/ha of triple superphosphate

LIMING RATE : 2.5 tonnes/ha

SEEDING RATE : 67 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	45
Cocksfoot (*Dactylis glomerata*)	20
Timothy (*Phleum pratense*)	10
Red fescue (*Festuca rubra*)	10
White clover (*Trifolium repens*)	10
Red clover (*Trifolium pratense*)	5

SPECIES PRESENT 1979/80

Perennial ryegrass (*Lolium perenne*)

Timothy (*Phleum pratense*)

Common bent (*Agrostis tenuis*)

Smooth-stalked meadow grass (*Poa pratensis*)

Yorkshire fog (*Holcus lanatus*)

White clover (*Trifolium repens*)

Red clover (*Trifolium pratense*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-3	SOIL	VERY ABUNDANT	7.0	18.1
3-10	"	ABUNDANT	6.8	25.2
10-19	COAL WASTE	"	7.3	9.6
19-30	" "	FEW	6.6	7.7
30+	" "	NONE	6.6	7.0

USE: Cattle grazing

COVER ASSESSMENT: 4

COMMENTS:

The well-structured sandy loam soil supports good grass and clover growth. The loose, friable underlying spoil is not a physical barrier to root penetration and roots are found at 34 cm, indicating a well-established vegetation cover which is unlikely to suffer regression in dry weather and deep rooting will minimise soil erosion. The pH of the spoil suggests that there are no problems of acid generation.

Due to lack of available soil, part of this site was reclaimed without a soil cover. Lime was added at 5 tonnes/ha and seed at 100 kg /ha using the same seed mixture and fertilizer treatment.

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-4	COAL WASTE	ABUNDANT	7.3	9.0
4-20	" "	COMMON	7.4	8.7
20-32	" "	FEW	7.1	9.6
32+	" "	NONE	7.1	7.9

USE: Cattle grazing

COVER ASSESSMENT: 3

COMMENTS:

Surface cover is not as good as on the soiled area although root penetration is satisfactory (20 cm). The lower waterholding capacity of the coal waste, compared to the soil layer on the main part of the site, causes drought stress during dry periods. There are no problems of acidity in the spoil.

SITE NAME :	Castlehill
LOCATION :	Carluke, Strathclyde
DATE OF COMPLETION :	1976
SOIL COVER :	8 - 10 cm
SOIL TYPE :	Clay loam
FERTILIZER TREATMENT :	350 kg /ha SAI turf food T.F.4 granular fertilizer (9.5:9.5:13.2)
LIMING RATE :	NONE
SEEDING RATE :	65 kg /ha
SEED MIXTURE :	

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	31
Red fescue (*Festuca rubra*)	22
Common bent (*Agrostis tenuis*)	20
Timothy (*Phleum pratense*)	12
Red clover (*Trifolium pratense*)	9
White clover (*Trifolium repens*)	6

SPECIES PRESENT 1979/80

Perennial ryegrass (*Lolium perenne*)
Red fescue (*Festuca rubra*)
Timothy (*Phleum pratense*)
Cocksfoot (*Dactylis glomerata*)
Yorkshire fog (*Holcus lanatus*)
Red clover (*Trifolium pratense*)
White clover (*Trifolium repens*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-10	SOIL	ABUNDANT	6.5	16.8
10-20	COAL WASTE	COMMON	8.3	5.0
20-26	" "	FEW	8.4	4.8
26+	" "	NONE	8.4	5.1

USE: Grassland

COVER ASSESSMENT: 3

COMMENTS:

The heavy texture of the soil used on this site causes problems during periods of heavy rainfall and over the winter. The high water-holding capacity, together with the lack of slope on parts of the site, lead to waterlogging and bare patches (Plate 19). Growth on slopes is better due to improved drainage, with a tighter vegetation mat (Plate 20) and good ground cover. Clover growth is very good and there are no

PLATE 19 - CASTLEHILL - Surface (soil).

PLATE 20 - CASTLEHILL - Profile.

problems of acid generation.

Part of this site received no soil cover and was used for tree planting. Seeding rate and fertilizer treatment were the same as for the solid area.

SEED MIXTURE:

Species	% by weight
Red fescue (*Festuca rubra*)	31
Fine-leaved sheep's fescue (*Festuca tenuifolia*)	31
Common bent (*Agrostis tenuis*)	9
White clover (*Trifolium repens*)	21
Red clover (*Trifolium pratense*)	8

16 tree species were planted in the same season as the grass was sown. All trees were 20-45 cm tall with an established root system. 10g of 20:10:5 slow-release compound fertilizer was placed in the planting hold and 45g of 18:6:12 fertilizer plus 1 kg of pulverised bark was placed around the tree base.

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-1	PLANT LITTER			
1-20	COAL WASTE	COMMON	7.2	9.7
20-25	" "	FEW	7.0	5.8
25+	" "	NONE	7.1	5.1

USE: Tree planting as a screen for the main part of the site

COVER ASSESSMENT: 3 (Plate 21)

COMMENTS:

The coal waste was extremely hard and difficult to dig below 25 cm although roots were found to this depth. There is an accumulation of plant litter on the surface and clover growth is good. There are no problems of acid generation and the vegetation cover is comparable to that on the soiled part. Localized addition of fertilizer around the tree bases and the inclusion of such a high percentage of legumes in the seed mixture no doubt contribute to the success of this area of the site.

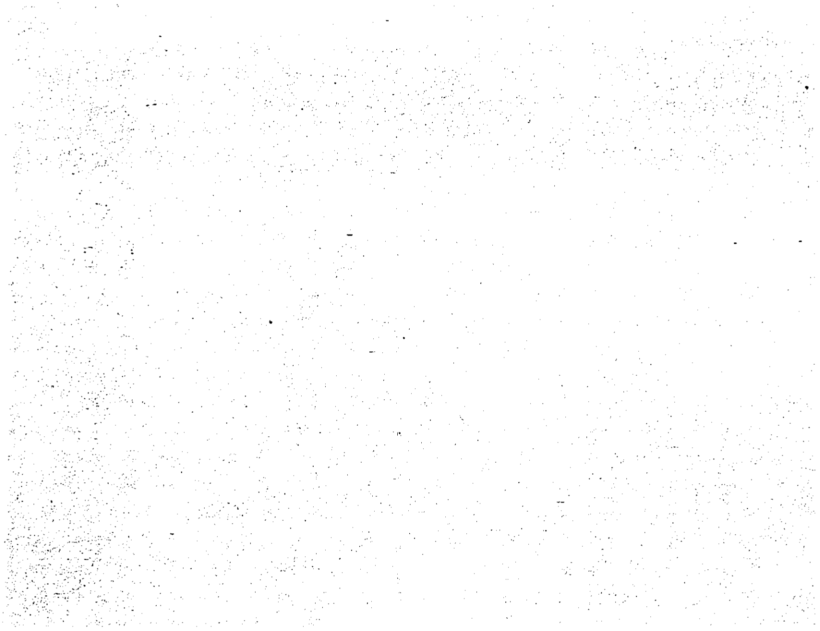


PLATE 21 - CASTLEHILL - Surface (no soil).

PLATE 22 - JENNY GRAY - General view.

SITE NAME : Devon

LOCATION : Fishcross, Central

DATE OF COMPLETION : 1976

SOIL COVER : 5 - 10 cm

SOIL TYPE : Loam

FERTILIZER TREATMENT : 375 kg/ha SAI plant food No. 4 (9.5:9.5:13.2)

LIMING RATE : 5 tonnes/ha

SEEDING RATE : 110 kg/ha

SEED MIXTURE :

Species	% by weight
Perennial ryegrass (*Lolium perenne*)	45
Cocksfoot (*Dactylis glomerata*)	25
Timothy (*Phleum pratense*)	15
Red clover (*Trifolium pratense*)	10
White clover (*Trifolium repens*)	5

SPECIES PRESENT 1979/80

Perennial ryegrass (*Lolium perenne*)
Timothy (*Phleum pratense*)
Common bent (*Agrostis tenuis*)
Yorkshire fog (*Holcus lanatus*)
Red clover (*Trifolium pratense*)
White clover (*Trifolium repens*)

USE: Grassland

COVER ASSESSMENT: 4

COMMENTS:

The grass and clover growth on this site is very good, both legume species surviving with well-developed root nodules. The well-structured loam soil is freely drained and supports good root growth. The site as a whole has sufficient slope to prevent waterlogging although there are severe erosion gullies in the soil layer on the lower slopes suggesting that the spoil is impermeable, further evidence of this being shown by the lateral growth of roots at the soil/spoil interface.

SITE NAME	:	Jenny Gray
LOCATION	:	Newfarm, Lochgelly, Fife
DATE OF COMPLETION	:	1965
SOIL COVER	:	11 - 15 cm
SOIL TYPE	:	Clay loam
FERTILIZER TREATMENT	:	1.3 tonnes/ha SAI plant food No. 3, supplying 100 units N, 250 Units P_2O_5, 150 units K_2O per hectare. Basic slag to supply 500 units P_2O_5 per hectare.
LIMING RATE	:	5 tonnes/ha
SEEDING RATE	:	49 kg/ha
SEED MIXTURE	:	

Species	% by weight
Meadow fescue (*Festuca pratensis*)	37
Timothy (*Phleum pratense*)	19
Cocksfoot (*Dactylis glomerata*)	19
Perennial ryegrass (*Lolium perenne*)	11
Crested dogs'-tail (*Cynosurus cristatus*)	2
Alsike clover (*Trifolium hybridum*)	12

SPECIES PRESENT 1979/80

Meadow fescue (Festuca pratensis)

Timothy (Phleum pratense)

Cocksfoot (Dactylis glomerata)

Perennial ryegrass (Lolium perenne)

Yorkshire fog (Holcus lanatus)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-3	SOIL	VERY ABUNDANT	6.1	14.7
3-11	"	ABUNDANT	5.8	9.7
11-15	COAL WASTE	"	6.1	8.3
15-25	" "	FEW	6.1	9.9
25+	" "	NONE	6.4	8.9

USE: Cattle grazing

COVER ASSESSMENT: 5

COMMENTS:

The pH of the coal waste is not a problem and there is little liklihood of acid generation. Surface cover and drainage are good (Plate 22) and there is little damage by stock, except at gates and troughs. Despite the fact that the coal waste is compacted and difficult to dig, roots are found to a depth of 25 cm$^+$, an indication that the vegetation is well-established. Even during dry weather the cover shows a healthy sward, being sustained by the strong roots which freely cross the soil / spoil interface, an indication that the underlying spoil is free from pyritic, acid generation.

SITE NAME : Lochore Meadows, Phase II

LOCATION : Inchgall farm, Lochgelly, Fife

DATE OF COMPLETION : 1970

SOIL COVER : 5 - 19 cm

SOIL TYPE : Sandy clay loam

FERTILIZER TREATMENT : 1.3 tonnes/ha SAI plant food No. 3, supplying 100 units N, 250 units P_2O_5, 150 units K_2O per hectare. Basic slag to supply 500 units P_2O_5 per hectare.

LIMING RATE : 5 tonnes/ha

SEEDING RATE : 49 kg/ha

SEED MIXTURE :

Species	% by weight
Meadow fescue (*Festuca pratensis*)	37
Timothy (*Phleum pratense*)	19
Cocksfoot (*Dactylis glomerata*)	19
Perennial ryegrass (*Lolium perenne*)	11
Crested dogs'-tail (*Cynosurus cristatus*)	2
Alsike clover (*Trifolium hybridum*)	12

PLATE 23 - LOCHORE MEADOWS PHASE II - Kale growth at 3½ weeks.

PLATE 24 - LOCHORE MEADOWS PHASE II - Strip-grazing of kale at 13 weeks.

SPECIES PRESENT 1979/80

Meadow fescue (*Festuca pratensis*)

Timothy (*Phleum pratense*)

Cocksfoot (*Dactylis glomerata*)

Perennial ryegrass (*Lolium perenne*)

White clover (*Trifolium repens*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-2	SOIL	VERY ABUNDANT	6.2	26.0
2-11	"	ABUNDANT	6.0	11.5
11-13	"	FEW	5.6	8.5
13-15	COAL WASTE	"	6.4	6.5
15+	" "	NONE	6.4	6.9

USE: Cattle grazing

COVER ASSESSMENT: 5

COMMENTS:

The depth of soil cover on this site varies considerably but overall the vegetation cover is very good. There is some poaching late in the season when the soil layer is wet due to compaction of the spoil, this also leading to erosion of the soil layer on slopes, forming channels in the spoil.

PLATE 25 - LOCHORE MEADOWS PHASE II - Soil layer damage due to strip-grazing of kale.

PLATE 26 - LOCHORE MEADOWS PHASE II - Re-seeded area the season following kale grazing.

Of particular note on this site is the growing of kale in an area with only 3-5 cm of soil cover. The grass cover was killed using a contact herbicide and the ground was harrowed, seeded by direct drilling and rolled. Plate 23 shows the kale growing after 3½ weeks and Plate 24 shows 13 weeks growth, at which stage it was strip-grazed. Damage of the surface layer by grazing stock was severe (Plate 25) but only affected the soil cover, with no mixing of the soil and spoil. In the spring following kale grazing the ground was re-seeded to pasture. Grass establishment was good (Plate 26), there being considerable benefit to the area from the cattle manure and kale residues. Nutrients from these sources tended to be retained in the soil layer as there was little leaching due to compaction of the spoil.

SITE NAME : Lochore Meadows, Phase IV

LOCATION : Hilton of Beath Farm, Kelty, Fife

DATE OF COMPLETION : 1974

SOIL COVER : 7 - 14 cm

SOIL TYPE : Sandy clay loam

FERTILIZER TREATMENT : 1.3 tonnes/ha SAI plant food No. 3, supplying 100 units N, 250 units P_2O_5, 150 units K_2O per hectare. Basic slag to supply 500 units P_2O_5 per hectare.

LIMING RATE : 5 tonnes/ha

SEEDING RATE : 49 kg/ha

SEED MIXTURE

Species	% by weight
Meadow fescue (Festuca pratensis)	37
Timothy (Phleum pratense)	19
Cocksfoot (Dactylis glomerata)	19
Perennial ryegrass (Lolium perenne)	11
Crested dogs'-tail (Cynosurus cristatus)	2
Alsike clover (Trifolium hybridum)	12

SPECIES PRESENT 1979/80

Meadow fescue (Festuca pratensis)
Timothy (Phleum pratense)
Cocksfoot (Dactylis glomerata)
Perennial ryegrass (Lolium perenne)
Common bent (Agrostis tenuis)
Red clover (Trifolium pratense)
White clover (Trifolium repens)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-5	SOIL	VERY ABUNDANT	6.2	21.6
5-8	"	ABUNDANT	6.6	21.0
8-17	COAL WASTE	COMMON	6.2	15.9
17-26	" "	FEW	6.3	11.6
26+	" "	NONE	6.2	9.0

USE: Cattle and sheep grazing, some grass cut for hay

COVER ASSESSMENT: 5

COMMENTS:

As the above profile description shows, there is no problem of acid generation on this site and together with the loose, ash-type waste found over most of this site, deep rooting is encouraged (maximum rooting depth 26 cm). The growth of grass and naturally

PLATE 27 - LOCHORE MEADOWS PHASE IV - Damage to soil layer caused by poaching.

PLATE 28 - LOCHORE MEADOWS PHASE IV - Area in Plate 27 after re-seeding.

invading red clover (Trifolium pratense) and white clover (Trifolium repens) is good and the site supports a fairly high stocking rate. There is some poaching from stock and vehicles when the soil layer is wet (Plate 27) but rolling and re-seeding restores damaged areas (Plate 28). The soil cover is a well-structured, freely-draining sandy clay loam over a loose spoil and therefore, despite the lack of slope over most of the site, waterlogging is not a major problem.

SITE NAME	: Lochore Meadows, Phase VI
LOCATION	: Hilton of Beath Farm, Kelty, Fife
DATE OF COMPLETION	: 1976
SOIL COVER	: 7 - 12 cm
SOIL TYPE	: Sandy clay loam
FERTILIZER TREATMENT	: 1.3 tonnes/ha SAI plant food No. 3, supplying 100 units N, 250 units P_2O_5, 150 units K_2O per hectare. Basic slag to supply 500 units P_2O_5 per hectare.
LIMING RATE	: 5 tonnes/ha
SEEDING RATE	: 49 kg/ha
SEED MIXTURE	:

Species	% by weight
Meadow fescue (*Festuca pratensis*)	37
Timothy (*Phleum pratense*)	19
Cocksfoot (*Dactylis glomerata*)	19
Perennial ryegrass (*Lolium perenne*)	11
Crested dogs'-tail (*Cynosurus cristatus*)	2
Alsike clovre (*Trifolium hybridum*)	12

SPECIES PRESENT 1979/80

Meadow fescue (*Festuca pratensis*)

Timothy (*Phleum pratense*)

Perennial ryegrass (*Lolium perenne*)

Crested dogs'-tail (*Cynosurus cristatus*)

Common bent (*Agrostis tenuis*)

Red clover (*Trifolium pratense*)

White clover (*Trifolium repens*)

PROFILE:

DEPTH (cm)	MATERIAL	ROOTS	pH	MOISTURE CONTENT (%)
0-6	SOIL	VERY ABUNDANT	7.5	17.8
6-11	"	ABUNDANT	6.8	14.8
11-20	COAL WASTE	FEW	5.5	13.2
20+	" "	NONE	5.4	9.6

USE: Sheep grazing

COVER ASSESSMENT: 4

COMMENTS:

Rooting on this site is not as good as on Phase IV because the coal waste has a heavier texture and has been well compacted before soil spreading. Being a more recent scheme there has been less time for the vegetation to become established. The vegetation cover, however, is good and there is adequate drainage due to the conformation of the site, resulting in little water impoundment and no damage by poaching. The original legume species have died out and have been replaced to a degree by natural invasion of red clover (*Trifolium pratense*) and white clover (*Trifolium repens*).

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