

# REMEDIATION OF ACID GENERATING COLLIERY SPOIL USING STEEL SLAG – CASE STUDIES

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**Abstract:** One of the legacies of the coal mining industry is the existence of numerous colliery spoil mounds. Run-off waters from some of these mounds result in oxidation of sulphur compounds causing pH to drop to perhaps as low as 2.5. At this pH, mobility for metals increases and it results in destruction of both flora and fauna. In order to reduce acidity, a number of solutions have been investigated with varying degree of success. A recent study to reduce acidity in spoil run-off water included the use of Basic Oxygen Steel slag. Its slow release of lime resulted in longer term remediation compared with other techniques. In addition to this, steel slag contains elements which are essential for plant growth and can be regarded as a weak fertiliser. This was substantiated in two field trials, which had the aim of not only remediating acidity from two different types of colliery spoils, but also to develop a composition that supports grass growth. The objectives were achieved at both sites and some of the results of over 5000 chemical tests conducted during these studies are reported in this paper.

*Key words:* colliery spoil, steel slag, limestone quarry fines, remediation of acidity

## 1. INTRODUCTION

According to the British Geological survey report for the Environment Agency (Cameron et al. [1]) indicated that in 1996, about 22 000 ha were classified as colliery spoil waste. It is estimated that there is now only about 2000 ha of land areas covered with colliery spoil waste. Nonetheless, the remaining sites to lesser or greater extent, and depending on their location, continue to generate acid run-off due to the presence of pyrite. The European Waste Catalogue (Environment Agency [4]) codes this colliery spoil materials as EWC 01 04 07; i.e., Waste containing dangerous substances from physical and chemical processing of non metalliferous minerals with a hazardous mirror entry (Palumbo-Roe et al. [9]). These tips are regarded as

being potentially dangerous and should be monitored or remediated if the risk becomes unacceptable. One of the colliery spoil test sites was originally populated with a commercial tree plantation comprising pines. However, due to a coincidence of removal of trees from a large section of the area and the company going bankrupt, this section did not get replanted with the consequence of acid water run-off and resulting landscape could not self heal and showed excessive erosion. This was resulting in ongoing issue of generation of acidic run-off water and destruction of flora and fauna. If this area could be vegetated with grasses to enable a formation of a stable bio-system, then with time it was anticipated that trees would get established in the area naturally. Thus in the first instance it was to remediate the barren spoil areas to enable grasses to propagate. It was envisaged that the initial treatment

should be deep enough for self established trees to propagate. A number of treatments are available, but many deal with run-off water rather than deal with the problem at source. BOS slag, from the steel making industry, was examined as it dealt with the problem at source, i.e., prevented generation of acidity by raising its pH.

Typically, about 150 kg of Basic Oxygen Steel (BOS) slag is produced as a by-product for every ton of steel (MPA [8]). About 60% of the total of steel production in the world is undertaken using the Basic Oxygen steelmaking process (Stubbles [12]). Based on the World steel production of 1.64 billion tons in 2014 (World Steel Association [13]) annual production BOS is about 140 million tons. It is currently used as road-stone aggregate (main use), in agriculture as a liming agent, manufacture of both rock-wool insulation and cement amongst other minor applications.

A typical chemical analysis on processed BOS slag aggregate is: 42–44% CaO, 27–31% Fe<sub>2</sub>O<sub>3</sub>, 10–12% SiO<sub>2</sub>, 5–6% MgO, 3–4% Mn<sub>3</sub>O<sub>4</sub>, 1–2% P<sub>2</sub>O<sub>5</sub>, 1–2% Al<sub>2</sub>O<sub>3</sub>, 0.5% TiO<sub>2</sub>. BOS slag is usually dicalcium silicate (bredigite) (2CaO.SiO<sub>2</sub>), containing other elements up to 1–2% (e.g., Mn, Fe, Mg), tricalcium silicate (3CaO.SiO<sub>2</sub>), free lime (CaO), wustite (FeO), calcium ferrite (also dicalcium ferrite and calcium aluminoferrite), and minor amounts of periclase (MgO) and magnesiowustite (solid solution of FeO and MgO) (QPA, 2009). It can be seen that in addition to oxides and compounds of iron, aluminium and manganese, BOS slag contains calcium silicates.

Freshly produced slag can expand due to hydration of lime. However, it has been shown that BOS has the unusual characteristic of slowly releasing lime (Poh [10]) and other trace elements over long periods when in contact with soil. This slow release mechanism can continue over many months, making it an attractive alternative to using ground limestone for farmers. This property makes BOS slag an effective soil conditioner and taking account of the trace minerals present in slag, makes it a weak fertilizer. Both these facets have led to it being evaluated as a component in a mixture for remediating colliery spoil sites that are prone to generating acidity with pH as low as 2.7. Preliminary findings of the study were reported by Robinson et al. [11]. The aim of the study was to use BOS slag to raise the pH level to between 6 and 8 to enable vegetation to flourish and the ecological system to become self sustaining and stable. Work reported herein comprises final findings of the study which comprised construction and monitoring of trials at two colliery spoil sites in the

UK, together with extensive laboratory investigations at both the Universities of Birmingham and Harper Adams. Findings of some of the over 5000 tests conducted are reported in this paper.

Under the Waste Protocols project (Environment Agency [3]) investigations are underway to identify uses for steel slag, since it has limited application and large quantities are stockpiled. It is estimated that up to 470,000 tons may be used and diverted from landfills. Thus, importance of using steel slag in land remediation cannot be underestimated.

## 2. MATERIALS

BOS slag, limestone dust and compost were used in the investigation. This slag was obtained from the Corus steel works plant at Teesside, UK. Typical properties of slag used are shown in Table 1 (Jessic [5]). It had slightly lower than expected calcium oxide, but generally its properties were within the accepted range for typical steel slag.

Table 1. Properties of BOS slag from Teesport (Jessic [5])

Determinand	Percentage of sample	Typical properties of steel slag
Silicon as SiO <sub>2</sub>	16.29	11–18%
Calcium as CaO	38.80	44–50%
Aluminium as Al <sub>2</sub> O <sub>3</sub>	1.28	1–4%
Magnesium as MgO	8.15	1–8%
Titanium as TiO <sub>2</sub>	0.61	–
Phosphorus as P <sub>2</sub> O <sub>5</sub>	0.63	–
Manganese as MnO	3.44	–
Sodium as Na <sub>2</sub> O	0.42	–
Potassium as K <sub>2</sub> O	–	–
Total Sulphur	0.08	–
Total Iron as Fe	21.34	14–19%

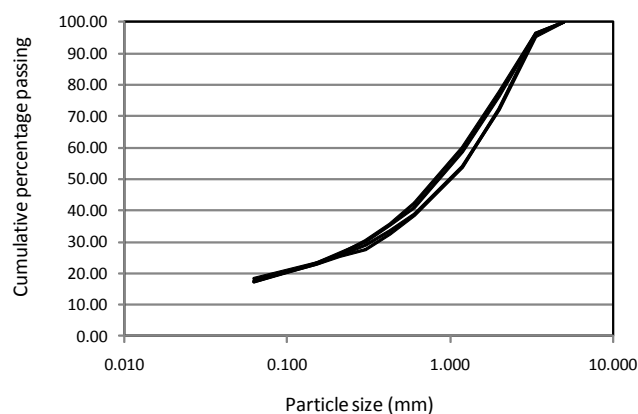


Fig. 1. Particle size distribution of limestone fines from the Sandside quarry

Limestone was obtained from Tarmac's Sandside quarry. Particle size distribution of this material ranged from 5 mm to dust with up to about 18% material finer than 60 mm; it is a well graded material and its range of particle content is shown in Fig. 1.

### 3. TRIAL SITES

Two trial sites (Car Wood and Derwent) were used in the study undertaken; both were located in Cumbria, UK. They were chosen as they contained colliery spoils with different characteristics. Each was treated in a different way to simulate the perceived end uses.

#### 3.1. CAR WOOD

Coal was produced from a mine at Car Wood (located near Aspatria, Cumbria, UK) between 1860 and 1930. After cessation of mining the site was originally vegetated with deciduous and coniferous trees (for commercial forestry) and other vegetation, to fit in with the surrounding woodland. During various changes of ownership, part of the wooded area was cleared for re-vegetation in mid 1970s, which never took place. Over time, run-off water from the barren areas had begun to kill the vegetation on the site and the surrounding watercourses had become polluted. During a survey of the polluted water in 2002, pH values as low as 2.7 were recorded and total iron concentrations of up to 597,000  $\mu\text{g/l}$  were measured (Law [6]).

Spoil was sampled in order to characterise it in terms of its chemistry and both moisture content and particle size distribution. Investigation was conducted to a depth 0.75 m as this was the maximum depth to which the spoil was to be treated. The moisture content and particle size distribution determinations are shown in Figs. 2 and 3, respectively. Moisture content was highest near the surface. This was not surprising as the spoil contained between 25% to 30% material that was finer than 0.060 mm.

After extensive laboratory trials at both the University of Birmingham and the Harper Adams University, which also included glass house growing trials, it was decided to conduct a field trial comprising BOS slag, limestone (zero to 4 mm) and compost were used to ameliorate acidity and provide the necessary fortification to support sustainable grass growth in material which had laid barren for two decades.

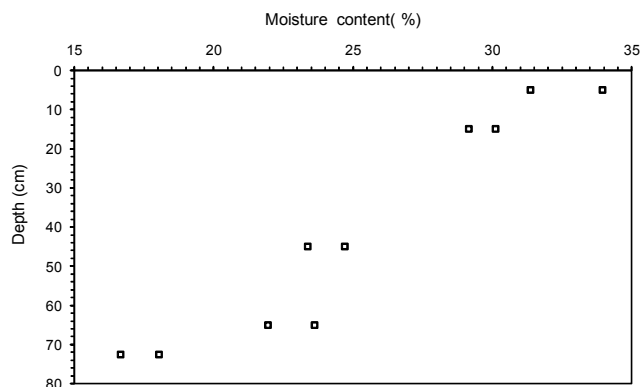


Fig. 2. Relationship between moisture and depth within the proposed trial area at Car Wood

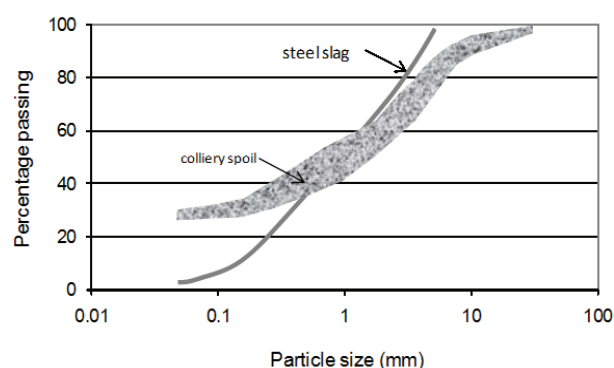


Fig. 3. Particle size distribution of colliery spoil from Car Wood and BOS slag

#### 3.2. DERWENT FOREST

The site is approximately 425 hectares and is located about 500 m from River Derwent, at its closest position. River Derwent forms part of a Special Area of Conservation. From about 1938 to 1992 the site formed part of an active ammunitions storage, inspection, repair and proofing facility under the ownership of the Ministry of Defence and was closed in December 1992.

Prior to 1938 the principal industrial usage of the site related to Buckhill Colliery and associated coal carbonation and gas storage facilities. There was large amount of spoil on the site. The site area contained about 300 buildings in various state of repair, which included 131 magazines. In some sections of the site, colliery spoil was used to construct blast bunds that were used to isolate magazines and munitions stores. In terms of the future site development, a number of schemes have existed, but the most favoured ones included returning the site to "natural" woodland which would require removal of many buildings and flattening of a number of blast bunds. This study was undertaken as there was some concern that the expo-

sure of spoil to moisture and oxygen (from air and water) may result in generation of acidity that may impact on the local ecosystem and River Derwent.

Historical information was reviewed in order to identify the area that was likely to contain mounds that were made using colliery spoil and in particular unburnt spoil. Trial pits were dug into the sides of embankments as shown in Fig. 4. Moisture content of the spoil ranged between about 17.5% to 19.8%, pH ranged between about 4.7 to 5.4 and loss on ignition ranged between 9.4% and 22.4%. The unburnt spoil was essentially in the medium sand to coarse gravel sized, see Fig. 5.

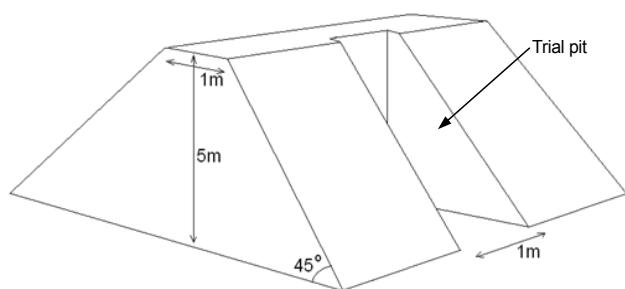


Fig. 4. Typical layout of trial pits in a blast mound at Derwent Forest

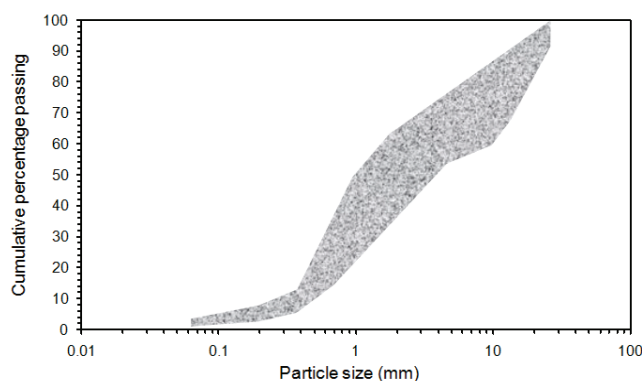


Fig. 5. Particle size distribution of un-burnt spoil from trial pits at Derwent Forest

## 4. LABORATORY INVESTIGATION AND RESULTS

### 4.1. LABORATORY INVESTIGATION

The study included assessment of change in pH with time for a range of compositions, measurements of conductivity and grass growing trials. Conductivity tests were conducted on both filtered and unfiltered eluate. Only a limited range of typical examples are presented in this paper.

Three sets of experiments were conducted at Harper Adams University College. These were accelerated plant growing trials and were designed to measure pH response, grass growth and effectiveness of phyto-remediator plants. Accelerated pH response trial was carried out using <1 mm BOS and limestone to see how quickly and for how long the pH of the glass-house trials could be raised. Phyto-remediator plants were investigated for their ability to remove heavy metals from the soil.

Specific treatments investigated were as follows:

#### *Accelerated pH response without leaching*

Both pH and electrical conductivity were measured in aerated and regularly wetted soils kept at 70 degrees centigrade over 8 weeks, accelerating lime reaction and sulphur oxidation.

5% limestone and 5% compost with varying steel slag rates, 2, 3, 4 and 5% steel slag

#### *Accelerated pH response with leaching*

As above (A) but kept at 50 degrees centigrade and leached with distilled water to simulate 10 years of leaching. Both soils and leachates were tested for pH and conductivity. A leaching water depth equal to the depth of the soil layer was used.

#### *“Fine tuning” accelerated pH response without leaching*

Fresh soils were treated with lime, steel slag and compost in an accelerated pH response trial, again at 70 degrees without leaching. pH of each mix was measured.

#### *Glasshouse trial pH and conductivity checks*

The soils from the first grass growth trial were measured at intervals to check the relationship between ambient temperature versus the accelerated pH response trials.

#### *Grass growth trials*

A mixture of two grass species were used namely Tall fescue (*Festuca arundinacea*) and Red top or Creeping bent (*Agrostis stolonifera*). These were chosen for their soil tolerance, durability and ground cover.

### 4.2. RESULTS OF THE LABORATORY STUDY

Laboratory investigations for change in pH with addition of slag are shown in Fig. 6. Results showed

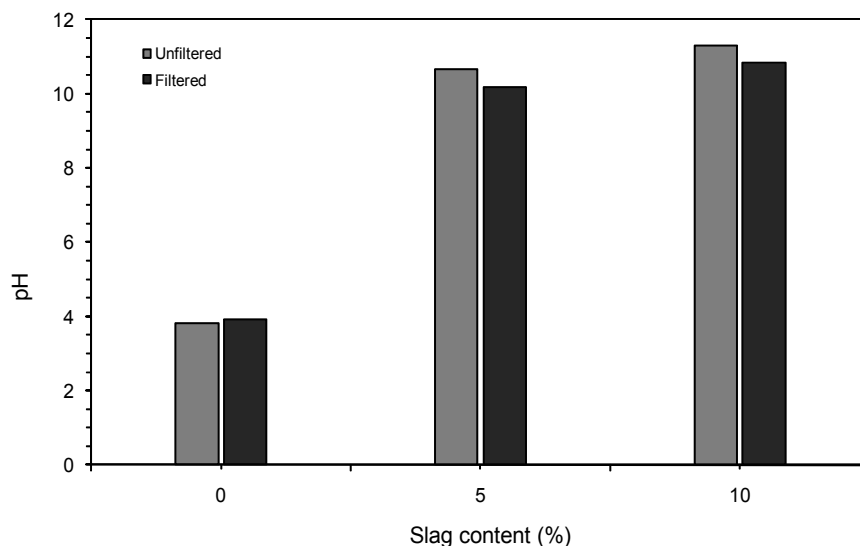


Fig. 6. pH of leachate for 0%, 5% and 10% BOS slag

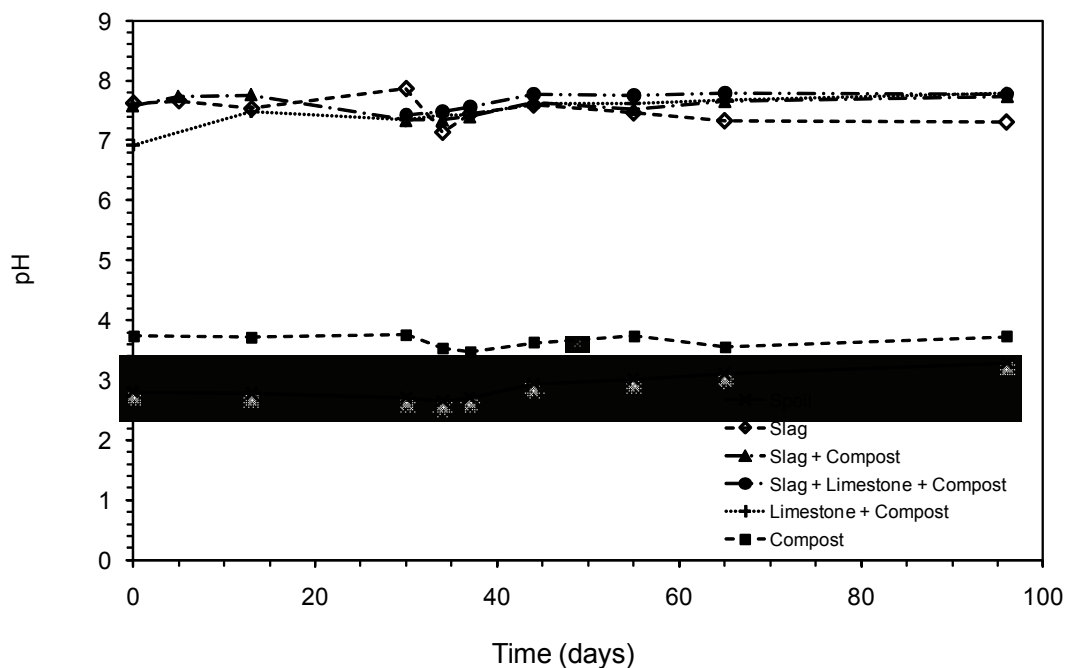


Fig. 7. Results of pH testing in laboratory trials on Car Wood spoil (slag, compost and limestone were added at up to 5% by dry weight of spoil)

that both filtered and unfiltered eluate exhibited similar behaviour and that less than 5% slag was required to raise the pH to over 8 for the Car Wood spoil. For the Derwent Forest site lesser quantity of slag was required to raise the pH to above the target value of 7 to 7.5. Time related relationship of various additives and change in pH is shown in Fig. 7.

Compost was used in order to raise the organic matter content in the spoil, improve nitrogen release,

buffer against pH changes and promote stable physical properties. However, while the compost contained some lime, the 5% by weight used only raised pH by <0.5 units. To lift the pH to the desired target range, either BOS slag or limestone dusts were used, singly or in combination. The laboratory study described above and accelerated grass growing trials led to the development of mix designs that were used in the field trials.

#### 4.3. CONSTRUCTION AND MONITORING OF TRIAL SITES

Coal mining spoil has specific chemical characteristics that require assessment for the safe and effective restoration and use of land and materials. Based on these, the following characteristics were assessed at intervals of time for the different plot treatments and at different depths at the sites, using the methods of Ministry of Agriculture, Food and Fisheries (MAFF [7]):

- Total and EDTA extractable lead, cadmium, zinc, copper and nickel,
- Soil pH,
- Carbon and nitrogen,
- Available phosphate, potassium and magnesium,
- Sulphur and sulfate,
- Exchangeable and easily reducible manganese.

In addition to the above heavy metal content in the leaf matter of grass was also measured.

Samples were taken for scanning electron microscopy and thin section preparation in order to understand spatial relationships more fully.

Soil tests were aimed at assessing heavy metals that are commonly present in coal spoil, the pH environment that influences heavy metal availability and toxicity and general soil quality for plant growth, soil fertility parameters such as nitrogen, phosphorous, potassium and magnesium, carbon that makes up organic matter that influences the physical quality of the soil in its aggregation and aeration and manganese which is a trace element affecting all plant types in deficiency and toxicity situations. Sulphur and sulphate contents were also measured because sulphate is associated with salinity and the sulphur level could be used to check potential acidity production and thereby substantiate the accelerated timing response trials.

For the Car wood site, in grassed areas, three samples were obtained from depths of 50 mm and from about 200 mm from each plot. Samples from each depth are then mixed such that two samples are available from each area. Thus for each round of testing 36 test samples will be taken. Samples were retrieved at day 0, 189, 374, and 554 days. In addition to this both pH and manganese levels were monitored at 445 days.

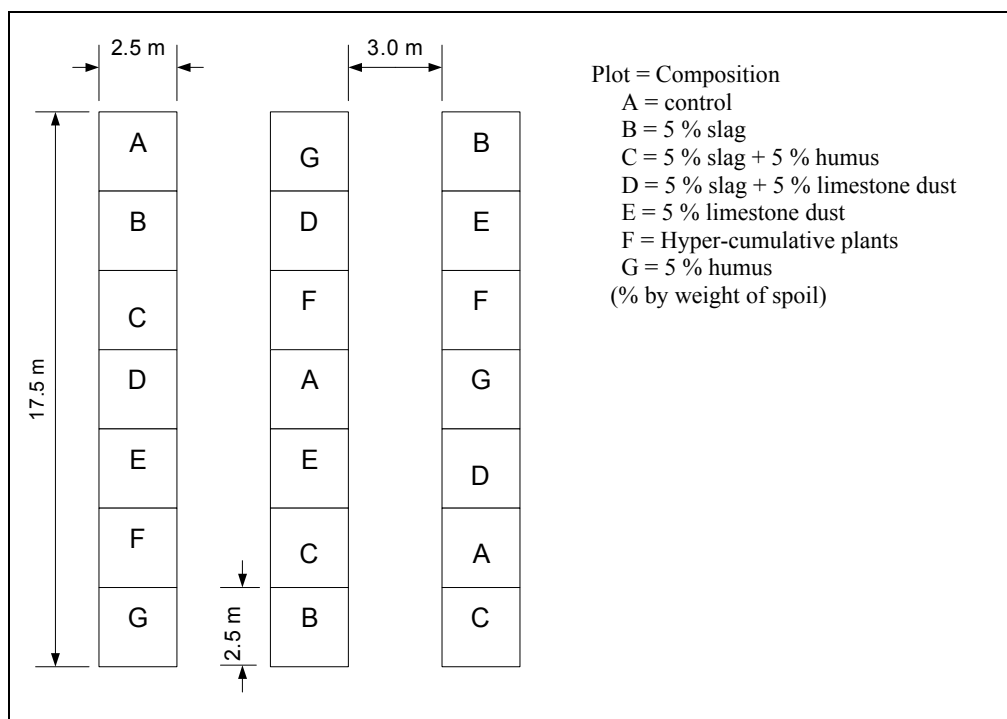


Fig. 8. Layout of plots of different mixes at Car Wood

Plot	A	B	C	D	E	F	G	H	I	J
Slag	—	2	4%	—	—	2%	4%	4%	—	—
Limestone	—	—	—	2%	4%	2%	4%	4%	—	—
Compost	2%	2%	2%	2%	2%	2%	4%	2%	4%	2%

For the field trial, seven mixes were repeated three times in twenty one plots in three rows. The resulting configuration is shown in Fig. 8. Plots were 2.5 m × 2.5 m and 0.75 m in depth (below existing ground level). All the excavated material was returned to the plot forming mounds of different heights.

For the Car Wood site a backhoe excavator was then used to excavate this area to a depth of 0.75 m. The same plant was also used to mix the materials after which they were placed back into the excavation. A rotivator was also used to mix the components before the backhoe replaced it in test sections. About 15 mm of mulch was added to surface of all the test plots except the control area, where excavated soil was mixed and any clods were broken up before replacing it in the test area. Adjacent plots were separated by plastic sheet that extended the whole width of each plot in order to prevent cross contamination. A 50:50 mix (in terms of seed numbers) of Tall Fescue (*Festuca arundinacea*) and Creeping Bent (*Agrostis stolonifera*) seeds were planted on plots A, B, C, D, E and G. Tall fescue was chosen because of its ease of establishment, low maintenance and soil tolerance. Creeping bent was chosen due to its ease of establishment and attractive appearance and popularity, for example, in golf course grasses. Hyper-accumulator plants Thale cress (*Arabidopsis thaliana*) and Alyssum (*Alyssum argenteum*) were planted in plots F. These were chosen because of their ability to take up heavy metals. All areas except F were seeded in July at the time of construction of the trial. Plot F was seeded in September due to delayed delivery of seeds. All seed was applied at a rate of 50 g per m<sup>2</sup> and each of the plots was constructed in the same manner.

Since, at the Derwent Forest site, spoil was to be spread on existing ground, trial plots measuring 2.4 m × 2.4 m and 0.3 m deep were constructed. Each plot was separated from the adjacent area by a 12 mm thick sheet of ply wood measuring 2.4 m × 0.3 m deep. Material required for all the plots was excavated from one mound. The required amount of spoil for each plot was then removed from the mound and the corresponding amounts of additives were added and mixed using the backhoe excavator.

When all the plots were constructed, about 15 mm of mulch was applied to the top before seeding the plots to prevent crusting and initiate soil development, as for the Car Wood site. A safety fence, including a rabbit fence was erected around trial area and also separately around each of the trees. The layout of the plots is shown in Fig. 9.

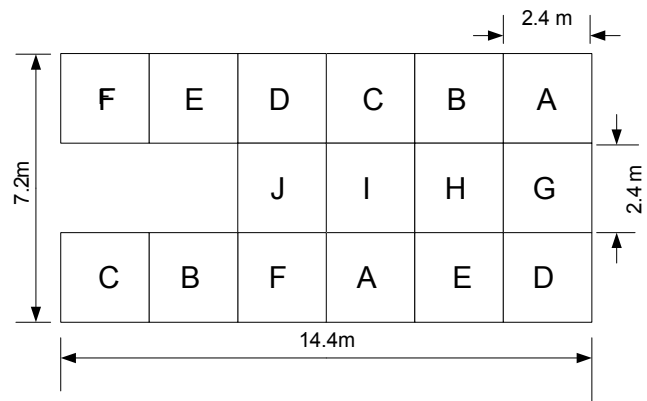


Fig. 9. Composition and layout of trial areas at Derwent Forest

## 5. RESULTS AND DISCUSSION

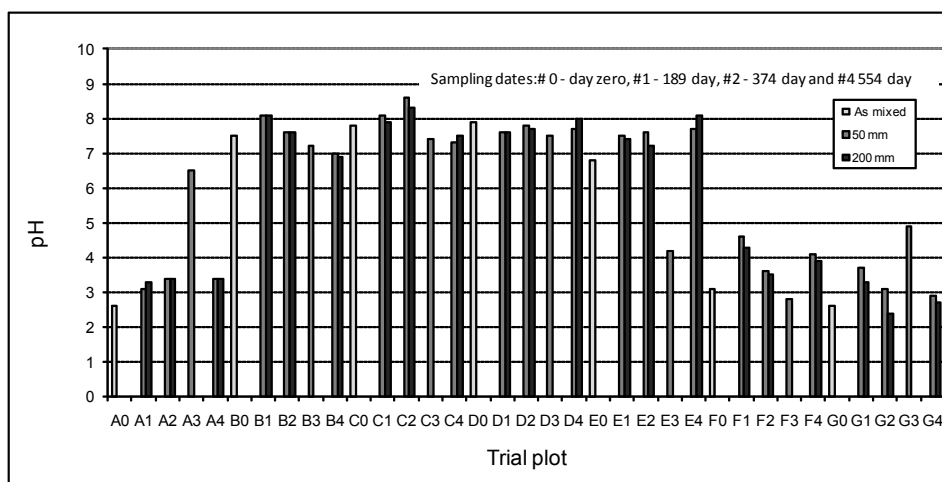
Results are plotted in terms of plot identification ranging from A to G for the Car Wood site and A to J for the Derwent Forest. The key to the composition of each site is shown in Figs. 8 and 9. Over 5000 chemical tests were conducted during this study and only key results are presented below.

Low acidity is related to the levels of sulphur and sulphate. pH levels show very acid conditions and this is higher near to the surface (above 1.5 metres in the profile) due to sulphur oxidation. The sulphur contents have been used to calculate the maximum theoretical lime requirement and these suggest a requirement for 11% to 12% limestone content for the Car Wood site and only about 3% for the Derwent Forest site.

Variations in pH with time for the various mixes examined in the field trial are shown in Figs. 10 and 11 for the Car wood and Derwent Forest sites, respectively. The Car wood results show that pH of between about 7 and 8 was maintained for with slag, slag and compost, slag, compost and limestone fines and each limestone fines and compost. Generally pH of about 3 to 4 was recorded for the untreated areas. The results clearly show that adding compost only (5%) does not affect the pH.

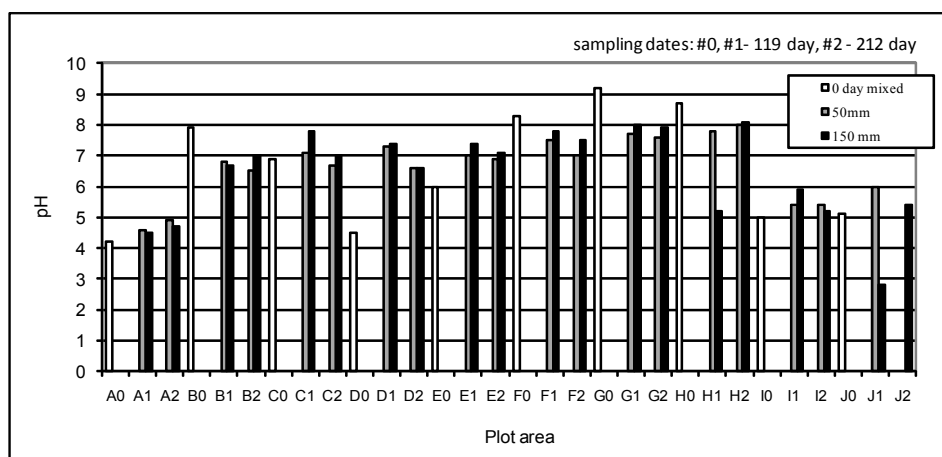
The Derwent Forest results show that the most appropriate mixture would contain about 2% lime, 2% limestone dust, 2% steel slag and 2% compost and that containing 4% lime, 4% limestone dust 4% steel slag and 2% compost.

Carbon contents were high for Car Wood, and most of the results were in the range 6.2 to 15.5%. Carbon/nitrogen ratios range between about 15 and 40. Results of carbon content and carbon/nitrogen ratios are shown in Fig. 12. Carbon content for Derwent Forest site was also high, in the range 5.8 to 11.5%. Carbon/



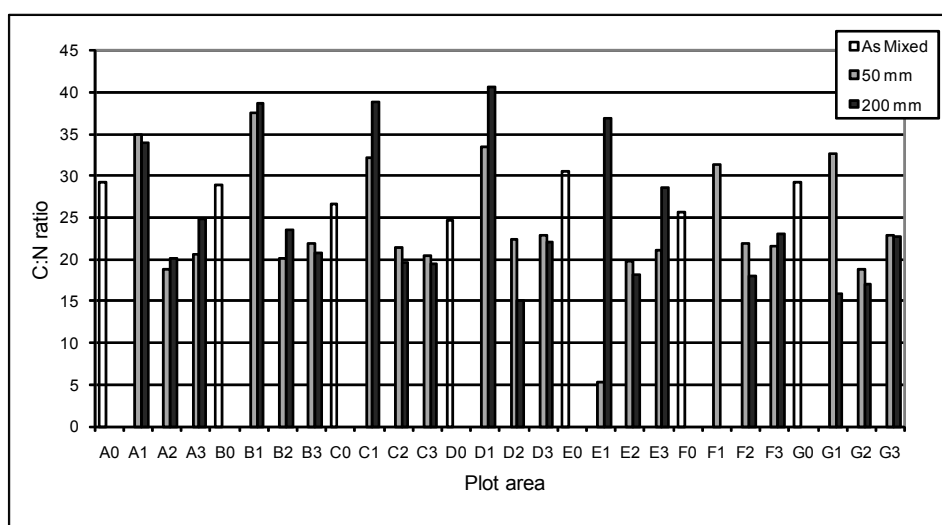
Refer to Fig. 8 for trial plot description

Fig. 10. Variation of pH with time for Car Wood



Refer to Fig. 9 for trial plot description

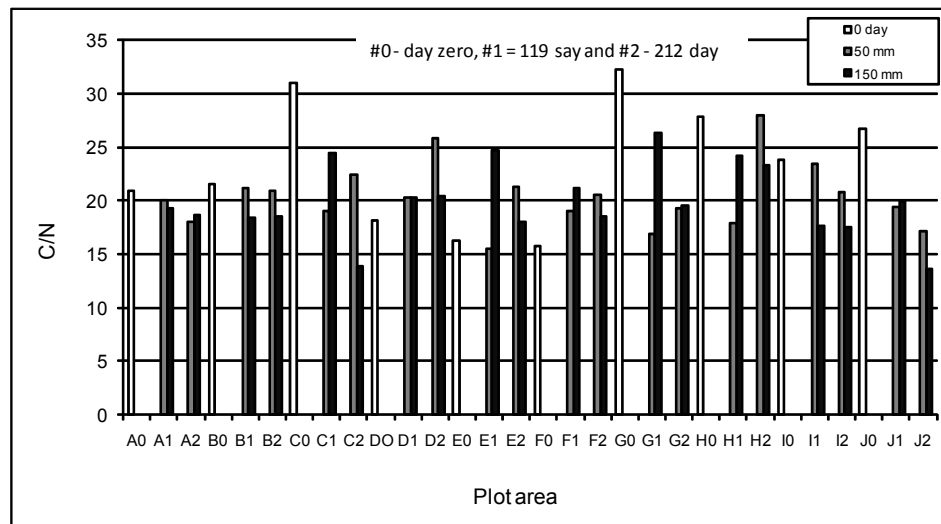
Fig. 11. Variation of the pH with time for Derwent Forest



Refer to Fig. 10b for trial plot description

Fig. 12. Carbon/nitrogen ratio for Car Wood





Refer to Fig. 11b for trial plot description

Fig. 13. Carbon/Nitrogen ratios for Derwent Forest

Table 2. Copper and cadmium levels in grass

Glasshouse treatment	Grass		Alyssum argenteum		Arabidopsis thaliana	
	Cu	Cd	Cu	Cd	Cu	Cd
Compost only	6.58	0.31	3.59	0.31	4.92	<0.10
Derwent Forest (Nil added)	4.67	0.34	ND	ND	ND	ND
Derwent Forest 2% BOS (and with +5% limestone and compost)	5.7	0.16	5.61	0.25	1.37	0.24

NB: Cu – copper: Cd – cadmium both  $\text{mg kg}^{-1}$  on a dry matter basis.

nitrogen ratios range between 17 and 33, see Fig. 13. Productive agricultural soils have C:N ratios of 10:1 and therefore the addition of compost and lime will encourage improvement in nitrogen release and “greening” of the vegetation.

Potassium, phosphorus and magnesium were satisfactory for supporting grass. Lead, nickel, cadmium and zinc were below the critical levels for total element content (MAFF [7]), although some cadmium values were at the critical level.

Copper was considered to be more serious at both Car Wood, where mean of about  $174 \text{ mg kg}^{-1}$  (range  $156 \text{ mg/kg}$  to  $202 \text{ mg/kg}$ ) and at Derwent Forest sites with mean of  $107.6 \text{ mg/kg}$  (range  $90$  to  $149 \text{ mg/kg}$ ). Soil Code (MAFF [7]) maximum is  $200 \text{ mg kg}^{-1}$  for a soil of pH greater than 7, where slurries are in use. The target pH at these sites would have to exceed 7 as for pH's of 6–7, the maximum is  $135 \text{ mg kg}^{-1}$ . The WRAP compost maximum is  $200 \text{ mg kg}^{-1}$ . While the means are below  $200 \text{ mg kg}^{-1}$ , it should be noted that 8 out of 48 readings over the period of measurement exceed  $200 \text{ mg kg}^{-1}$  at Car Wood. DEFRA [2] pub-

lished revised trigger values for copper at 100 ppm copper at which threshold, professional advice should be taken before applying more manures. The E.D.T.A extractable copper values are a measure of available copper. The values are in the range found on mixed farms namely less than 10 ppm, and not of concern as a toxic element.

In order to explain these findings, soil microscopy was used. This showed that the coal fragments are often discrete skeleton grains with a very low surface area. As a result, the available copper is low.

Levels of heavy metals in grass and other plants are shown in Table 2. Results support the choice of *Alyssum argenteum* for phytoremediation as it showed the highest uptake of copper with about 3 times that of grass. Cadmium levels (maximum  $0.34 \text{ mg/kg}$ ) were below the safe limits for animal consumption ( $20$  to  $160 \text{ mg/kg}$ ).

Findings of the laboratory trials and two case studies reported herein have led to the development of a prediction model that can be used to estimate the amount of additive(s) required for given soil types and drainage conditions.

## 6. CONCLUSIONS

The soils and plant tests have produced an integrated assessment of both Car Wood and Derwent Forest soils in which the findings generally agree well with each other and the literature. The “accelerated” oxidation and leaching experiments together with the microscopy all allow firm recommendations to be made and a “blueprint” for coal site remediation could be put forward.

If acidity develops in the longer term, which it will eventually, (normal rain is pH 6) the test results can be used to estimate surface lime and BOS applications.

The overriding conclusion is that limestone with BOS slag with compost is the best long term amelioration treatment to reduce acidity, start biological and biodiversity improvement and aid fertility for grassland or forestry canopies. The results suggest that stable products are generated in terms of pH and other elements.

Some spots of higher copper and cadmium levels were identified with total copper levels exceeding acceptable limits. Plant available copper was in line with soils on a typical mixed farm showing that the copper was “trapped” inside mineral particles and would not be a problem based on mixing of these soils and their treatment.

Potassium, phosphorus and magnesium were satisfactory for supporting grass and lead, nickel, cadmium and zinc are below the levels for total element content.

For both the sites grass was successfully grown. In particular it was demonstrated that sustainable vegetation could be grown on barren colliery spoil by adding steel slag, limestone dust and compost.

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