THE POTENTIAL OF IN SITU AMENDMENTS TO IMPROVE ALDER ESTABLISHMENT IN POTS OF METALLIFEROUS MINE WASTE[1]

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Abstract: Several inherent characteristics of metalliferous mine wastes inhibit the establishment of vegetation: nutrient deficiency, acidity, poor water retention, metal toxicity, instability, and poor soil structure. They may be overcome by in situ amendments. Most reclamation programs in west Cornwall, UK, involve extensive tree-planting schemes. Alder species are usually a major component of such schemes because of their N-fixing capability. A pot experiment (based on a quarter replicate of a full 2^7 factorial design) was established at the Camborne School of Mines, to investigate the effects of seven amendments (and their interactions) on the growth of common alder (*Alnus glutinosa* (L.) Gaertner). The amendments were ammonium nitrate, mineral phosphate and potash fertilizers, calcareous sea sand, dewatered sewage sludge, diatomaceous earth, and a superexpanding polyacrylamide gel polymer. The waste was analyzed for total and ammonium acetate-extractable metals and nutrients, pH, and total exchangeable bases at the outset of the experiment, and was found to contain 0.3% As and very low concentrations of the major nutrients. Tree heights and survival were recorded monthly for two growing seasons. Leaf, stem, and root dry weights were recorded after harvesting, from which various resource allocation ratios were calculated. Sewage was the best amendment, followed by diatomaceous earth. The N, P, and K fertilizers, sand, and gel had little effect, being deleterious in some cases. Of the interactions, the traditional mineral phosphate-calcareous sand treatment was ineffective. The importance of soil structure improvement by organic material was apparent.

Introduction

Cornwall contains approximately 3,200 ha of derelict mined land (DOE 1991). Most of this has resulted from centuries of intense metalliferous mining activity. When this land is reclaimed, trees are major constituents of most schemes. Once established, tree root systems are strong and very extensive, stabilizing the soil at depth. Soil organisms proliferate in the rhizosphere secreting soil-binding mucous substances, thus imparting the all-important "crumb texture" to soils. This improves aeration and drainage and reduces compaction (Cherfas, 1992).

An in situ amendment can be described as a material that is intimately mixed with the mine waste in order to improve the substrate for plant growth (Mitchell 1991a). The most common are lime, fertilizers, and organic wastes. The main problems with metalliferous mine wastes are their low nutrient concentrations, their poor soil structure and water retention, and the presence of heavy metals which any amendment would need to render unavailable to plants. This can be done in situ by either complexation or precipitation. The application of lime and/or phosphate fertilizers reduces the solubilities of some heavy metals and metalloids. Organic molecules in sewage sludges can also complex heavy metals. Mitchell and Atkinson (1991) have investigated the metal-complexing properties of ion-exchange minerals. Mitchell (1991b) has discussed their relevance to field reclamation practices. The possibility of using one of Mitchell's promising amendments and a superexpanding polyacrylamide gel, in combination with the more conventional amendments, has been investigated in this work.

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This experiment investigated the effects of seven amendments on the growth of common alder (*Alnus glutinosa* (L.) Gaertner) in metalliferous mine waste. Common alder was chosen owing to its importance in reclamation schemes. N, P, and K fertilizers provided essential plant macronutrients (P may also render some metals insoluble). Calcareous sea sand was used as a locally available lime source to increase the pH and therefore decrease the availability of some metals (as used in local reclamation schemes). Dewatered sewage sludge has many useful properties: it is a slow-release nutrient source; and its organic content retains water, improves the soil texture, and may complex metals (Byrom and Bradshaw 1991). Diatomaceous earth is believed to sequester metals as insoluble silicates (Mitchell and Atkinson 1991). Superexpanding gel polymers are used in horticulture primarily as water-retaining agents (Johnson 1984). They also sequester metals in solution (Mitchell 1991a). A factorial experiment was designed to investigate the potential of established and novel amendments, both individually and interactively with other amendments.

Methods

Collection and Preparation of Waste Samples

Approximately 4 t of mine waste was collected from United Downs, west Cornwall, UK (grid ref: SW7441). The site is composed of several abandoned copper mines that formed the highly productive United Mines group in the 19th Century. Waste from the top 30 cm of an area of the site was sieved through a 25 mm stainless steel mesh over a skip in order to extract the minus 25 mm material. The waste was mixed within the skip during collection and after emptying. Five 5 kg grab samples were taken at intervals and prepared for analysis.

The five samples were dried in an oven at 40°C to constant weight, and then sieved in stainless steel sieves to extract the minus 2 mm fraction. Approximately 200 g of this was riffled out using a stainless steel riffle, and about 20 g of this material was milled to a floury texture in a tungsten carbide tema mill.

Chemical Analysis

Weakly held metals were extracted using molar ammonium acetate adjusted to the pH of the waste (3.7) with glacial acetic acid. The procedure followed was that of Grimshaw (1989). The concentrations of Al, Ca, Cd, Cu, Fe, K, Mg, Mn, Na, Pb, and Zn were measured by atomic absorption spectrophotometry (AAS). The total exchangeable bases (TEB) were calculated by summation of the Ca, K, Mg, and Na concentrations in milliequivalents to derive a measure of the waste's effective cation exchange capacity (Grimshaw 1989). Waste pH was measured using the paste method of Hesse (1971).

The nitric-perchloric acid digestion method of Hesse (1971) and AAS were used to determine the "total" concentrations of each element, except As, which was measured by hydride generation. Total P was quantified colorimetrically using the molybdenum blue method of Grimshaw et al. (1989) with the additional sodium metabisulfite step of Olsen and Dean (1965) in order to reduce interference from arsenic acid and ferric iron.

Total N was extracted and measured using the semi-micro Kjeldahl digestion and distillation methods of Grimshaw et al. (1989). The Markham steam distillation apparatus was used (Markham 1942). Mineralizable N was measured using the method of Grimshaw et al. (1989). Total S was measured on a LECO HF10 Induction Furnace coupled with a sulfur titrater. Appropriate quality control standards were used throughout.

Preparation and Maintenance of the Experimental Treatments

The experiment was designed as a quarter replicate of a 2^7 factorial design with four replicates per treatment. Two-year-old (1+1) alder trees were planted in 10 L plastic pots at a rate of one tree per pot, in approximately 9 L of waste. One or more of the amendments was mixed with the waste according to the experimental design at the rates given in table 1. Addition rates were calculated according to volume (10 L) or weight (12 kg dry weight per pot), depending on the amendment. Fertilizer addition rates were obtained from

Williamson et al. (1982). Calcareous sea sand was added after measuring the pH and calculating the acid-neutralizing capacity (ANC) of the waste and its future acid generation potential (i.e., iron pyrite content) using the methods given in Williamson et al. (1982). The pH was 3.7, the ANC was 0.6%, and the pyrite content was 0.1%. It was calculated that 420 g of sand (at 40% calcium carbonate) per pot would be sufficient to neutralize the present and potential acidity. The application rates for diatomaceous earth and gel were more subjective owing to the novelty of this use. A 5% w/w rate has been used in work with zeolites as nutrient storage materials, and this was suggested as a suitable rate for diatomaceous earth (A. Moffat, Forestry Commission, UK, personal communication). The gel is usually applied at a rate of 0.1% w/v. An addition rate of 0.3% w/v was used after consultation with the manufacturers. The N, P, and K treatments received a maintenance top dressing of half the initial rate at the start of the second season (Williamson et al. 1982). The treatments were arranged in four randomized blocks and placed in an outside enclosure. Fungal infections were controlled with a systemic fungicide. During dry periods the trees were watered with tap water in the early morning or late afternoon.

Measuring and Harvesting Regimes

The experiment ran for almost two growing seasons. Monthly height measurements were taken to the nearest 0.5 cm during the growing season. Average heights and height increments from the start, from the four replicates of each treatment, were calculated. Tree deaths were taken into account during statistical analyses. The trees were harvested by removing the stems at soil level and grouping the replicates for analysis. This allowed the calculation of treatment means, which reduced the statistical effect of dead trees and the time required for further analysis. Roots were removed from the waste and washed. The root replicates were grouped as for the shoots. The trees were dried in a warm room at 33°C. When dry, the various parts were weighed. Biomass ratios were calculated from these results.

Statistical Analyses

Growth results were analyzed using the factorial design and analysis capability of the MINITAB release 8.2 computer package. As the factorial design was not orthogonal, adjusted means were automatically calculated for each factor and the first-order interactions. A factor or interaction has a positive or negative effect if the adjusted mean of all the treatments containing the factor is respectively higher or lower than the adjusted mean of the treatments without the factor. The factorial design chosen incorporated the treatment containing no amendments. Second and higher order interactions were omitted in order to release sufficient degrees of freedom for calculation of the t and F statistics. The effects values for these higher order interactions were noted and were found to be small and probably insignificant.

Table 1. Amendment application rates used in the experiment, and the equivalent field application rates.

		Application rate (dry weights)	
Amendment	Factor	g/pot	t/ha[1]
Ammonium nitrate	N	25	-
Mineral phosphate	P	100	-
Sulfate of potash	K	20	-
Calcareous sea sand	s	420	67
Superexpanding polymer	g	30	4.8
Dewatered sewage sludge	w	450	72
Diatomaceous earth	d	600	95

[1] Calculated as t/ha to 22 cm depth (equivalent to the depth of the pots).

"-" denotes that there are no equivalent field application rates for the fertilizers.

Results and Discussion

Mine Waste Properties

Mine waste was removed from an area of Ericaceous vegetation common over much of the United Downs site. It consists predominantly of waste rock that is very deficient in all the major plant nutrients and is moderately toxic (As 0.3%) with a pH of 3.7 (table 2). The TEB of 0.42 meq/100g is very low thereby exacerbating the leaching caused by the area's high annual rainfall.

Effects of Individual Amendments on Tree Performance

The three fertilizers showed no significant growth enhancing abilities and were deleterious in some cases (table 3). Ammonium nitrate fertilizer has an acidity equivalent to the neutralizing ability of 60 g $CaCO_3$ per 100 g fertilizer (Williamson et al. 1982). This is caused by oxidation of the NH_4^+ producing NO_3^- and H^+. It is known that the solubilities of many minerals increase at low pHs. The nutrient effect of N applied in this form may be irrelevant due to its high solubility, despite the maintenance top dressings. Also the natural N-fixing ability of alder could account for a large proportion of the plant's N requirements.

Mineral phosphate is a slow-release phosphate fertilizer that is widely used in reclamation schemes in west Cornwall. It was a major contributor to the poor survival of some treatments, although it had negligible effects on other properties (table 3). In highly acidic soils, phosphates may become fixed as insoluble iron and aluminium phosphates (Williamson et al. 1982). This effect would be compounded by the high Fe and Al concentrations of the waste (table 2). Phosphates also combine with Ca and Mg which would further reduce their availability and further depress the already low buffering capacity of the waste. There is evidence that excessive soil phosphate may displace arsenate ions into the soil solution, thus increasing the plant availability of As (O'Neil 1990).

Table 2. Selected chemical properties of the mine waste used in the experiment (mean, standard deviation (S.D.) and range. Note: *n*=5 unless otherwise stated).

	Total extractable[1], %			Ammonium acetate[2], µg/g		
Element	Mean	S.D.	Range	Mean	S.D.	Range
Al	5.6	0.27	5.30-5.80	277	14.5	264-298
As	0.30	0.01	0.30-0.32	-	-	-
Ca	0.04	0.02	0.02-0.05	61 (*n*=4)	43	0.00-96.0
Cu	0.05 (*n*=4)	0.002	0.04-0.05	21	6.1	15.0-30.0
Fe	8.4	0.09	8.30-8.50	1,980	236	1600-2200
K	0.36	0.08	0.26-0.48	1.3	1.5	0.00-3.80
Mg	0.66	0.02	0.64-0.68	6.7 (*n*=4)	1.5	5.60-8.60
N	0.06	0.01	0.04-0.07	-	-	-
P	43.0*	30.0	15-95	-	-	-
Other properties						
Mineralizable N (mg/100g)				0.94 (*n*=10)	0.63	0.00-1.64
pH				3.70	0.05	3.70-3.80
TEB (meq/100g)				0.42 (*n*=4)	0.23	0.08-0.60

ND = not detectable. TEB = total exchangeable bases. "-" = element not analyzed for.

[1] Total extractable = nitric-perchloric acid-extractable for all elements, except N and S (see text). Units expressed as percentages unless marked "*", where units are µg/g.

[2] Ammonium acetate-extractable metals expressed as µg/g unless otherwise stated.

Table 3. Effects and significances of the various factors and interactions on several plant growth properties[1].

		Growth property						Resource allocation				
Factor[2]	Survivorship	Height	Increment	Leaf DW	Stem DW	Root DW	Total DW	RSR	LWR	StWR	ShWR	RWR
N	- ***	- N.S.	- N.S.	- *	- N.S.	- *	- N.S.	- N.S.	- *	- N.S.	- N.S.	- N.S.
P	- ***	- N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.
K	- *	- N.S.	- N.S.	- *	- N.S.	- *	- *	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.
s	- ***	- *	- N.S.	+ N.S.	- **	- ***	- **	- **	+ ***	- N.S.	+ N.S	- *
g	- *	- N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
w	+ *	+ *	+ ***	+ ***	+ ***	+ **	+ ***	- N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.
d	- N.S.	+ N.S.	+ N.S.	+ *	+ *	+ N.S.	+ *	- *	- N.S.	+ N.S.	+ N.S.	- N.S.
N*P	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ *	+ N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	+ N.S.
N*K	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.
N*s	- *	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.
N*g	+ N.S.	+ N.S.	+ N.S.	+ *	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.
N*w	+ N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.
N*d	- *	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.
P*K	- N.S.	- *	- N.S.	- N.S.	- *	- *	- *	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.
P*s	- N.S.	- *	- **	- *	- **	- *	- *	- N.S.	- N.S	- N.S.	- N.S.	- N.S.
P*g	- N.S.	+ N.S.	+ N.S.	+ *	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
P*w	+ ***	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
P*d	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	+ N.S.	- N.S.	- N.S	- N.S.	- N.S.
K*s	+ N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	- N.S.	+ N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- *
K*g	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
K*w	+ *	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
K*d	+ N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.
s*g	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.	+ N.S.
s*w	- N.S.	+ N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	+ N.S.	+ N.S.	+ N.S.
s*d	- N.S.	- N.S.	+ N.S.	+ N.S.	- N.S.	- N.S.	+ N.S.	- N.S.	- N.S.	- N.S.	- N.S.	- N.S.

DW = dry weight, RSR = root/shoot ratio, LWR = leaf weight ratio, StWR = stem WR, ShWR = shoot WR, RWR = root weight ratio.

[1] Effect: + = positive effect, - = negative effect. Significance: * = $P \leq 0.05$, ** = $P \leq 0.01$, *** = $P \leq 0.005$, N.S. = not significant.

[2] See table 1 for identification of factors.

Sulfate of potash negatively affected survival and biomass (table 3). Height and stem dry weights were not affected, which suggests that the trees were preferentially allocating resources to poorer quality stem material. This could have been aggravated by the shortage of some other nutrient, e.g. P. In soils with a low cation exchange capacity (TEB in this case), potassium is rapidly leached (Williamson et al. 1982)

Calcareous sea sand was the most deleterious amendment (table 3), despite its use as a liming agent in local reclamation schemes! Its poor performance may be explained by referring to the soil chemistry of As. Arsenic occurs in soils in various oxyanionic forms and shares a similar soil chemistry to P. There is evidence to suggest that the availability of As increases as the soil pH rises from pH 7 to 9 (O'Neil 1990).

The sand had the greatest effect on resource allocation of any amendment. The root weight ratio (RWR) was significantly decreased ($P \leq 0.05$), leading to a lower overall root-shoot ratio (RSR) ($P \leq 0.01$); at the same time there was a highly significant increase in leaf weight ratio (LWR) ($P \leq 0.005$). When taken together with the other results, it seems that the sand caused a higher allocation towards the aerial parts by increasing shoot biomass without increasing height (see height and increment measurements, table 3), perhaps as a response to a stressful environment. The stem weight ratio (StWR) was not significantly affected, although stem and root dry weights were, which suggests that the actual biomass per branch was relatively low because most resource allocation was going into leaf production.

Superexpanding gel is a potential soil structure improver. It can expand up to 400x its dry volume in pure water, releasing it gradually (Johnson 1984). There is also evidence that the gel sequesters metal ions from solution (Mitchell 1991a). The gel had a significant negative effect on survival ($P \leq 0.05$). It was observed accumulating at the surface of the waste where its ameliorating action is redundant.

The most promising in situ amendment identified in this study was dewatered sewage sludge. It was the only amendment to significantly improve survival ($P \leq 0.05$, table 3) and height increment ($P \leq 0.05$). It also significantly enhanced all aspects of the biomass (table 3). Dewatered sludge is produced by the compression of raw sewage to form a cake of approximately 30% solids by weight. Its main roles here were as a soil structure improver and a slow-release source of nutrients. Improvements in soil structure are caused by the presence of organic material, which expands when wet and contracts when dry thereby, reducing compaction (and therefore root damage), and improving air and water (and therefore nutrient) permeability. The organic fraction also acts as: a substrate for the growth of microorganisms, a water storage facility, and a complexing agent for heavy metals (Bradshaw and Chadwick 1980).

Diatomaceous earth is neither a major nutrient source nor a soil structure improver. However, it has a high ion removal capacity (Mitchell and Atkinson, 1991). Increasing the number of anionic exchange and/or removal sites in the waste should ameliorate the problem of competition between phosphate and arsenate ions for the otherwise scarce exchange sites. It significantly increased leaf and stem dry weights (table 3), thus having a significant effect on the RSR ($P \leq 0.05$, table 3). There was no effect on height or increment (table 3). This could be caused by an increase in the branching frequency, thereby increasing bushiness, but not necessarily affecting overall height growth.

Effects of Interactions

Of the first-order interactions, nitrogen-sand (N*s) and nitrogen-diatomaceous earth (N*d) had significant negative effects on survival ($P \leq 0.05$), whereas phosphorus-sewage (P*w) and potassium-sewage (K*w) had significant positive effects ($P \leq 0.005$ and $P \leq 0.05$ respectively, table 3). This suggests that there was a survival-promoting interaction between a nutrient amendment and a soil structure-enhancing amendment. The waste was highly deficient in both P and K (table 2), but both these factors had negative effects on growth when considered individually. The benefit of sewage in providing extra exchange sites, thus offset the toxicity effects of added P, and provided a top-up source of P for the increased plant demand due to K fertilization. Neither the N*s nor the N*d interaction contained a structural component; consequently there was an associated negative effect.

The phosphorus-potassium (P*K) and phosphorus-sand (P*s) interactions are the most prominent in table 3. Both had significantly negative effects on several properties. Neither contains a structure-improving amendment. A significant positive P*K interaction would be expected if potash were acting to produce poorer quality stems due to the shortage of P (for example). This was not the case, perhaps because the beneficial effects of P were being offset by an increase in As availability. However, the K*w interaction was positively significant for survival, and the negative effects on leaf, root and total dry weights have been eradicated (table 3). This suggests that the lower levels of P in sewage were enough to promote growth, but not enough to initiate As toxicity. The potentially toxic effects of any excess As release could have been reduced by the increase in exchange sites on organic molecules in the sewage. Both P and sand acted individually to increase As availability. Together they produced a synergistic effect, further worsening the situation, i.e. sand increased the solubility of As, while P increased the competition for the limited number of exchange sites in the soil.

Conclusions and Future Work

Dewatered sewage sludge was the most promising amendment. Its main benefit was that it improved the soil structure. Heavy metal sequestration, nutrient supply, and water retention were secondary-but nevertheless important-as far as alder performance was concerned. Sewage is also cheap and widely available. Traditional local amendments such as mineral phosphate and calcareous sea sand may have no positive effects on alder growth at best, and at worst may actually reduce alder performance. Of the novel amendments, the gel had little effect, but the diatomaceous earth was promising. The major drawback with both of these potential amendments is cost.

Further work is required to refine the optimal application rates in terms of tree performance and economics. Chemical analysis of the treatments is presently underway. A fieldwork program is required to test the trends outlined in this paper. A similar experiment is presently underway with sycamore (*Acer pseudoplatanus* L.) to test the modifying effects (if any) of the alder's N-fixing capability on the action of the amendments.

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