Utilisation of Ochreous Sludge as a Soil Amendment

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Abstract

A study of the possible utilisation of ochreous sludge from iron-rich minewater and groundwater in artificial soil formulations was carried out. Ochre from the drying beds and wetland at the Woolley Mine Water Treatment System and from a natural analogue at the Silwood Park Wetland, was placed in growth cells in various proportions with local waste materials, particularly coal spoil and stabilised sewage sludge. In one set of experiments, twenty-four cells of dimensions approximately 0.7 m x 0.7 m x 0.4 m depth were employed in an exposed location, each fitted with a discharge pipe at the base. They were sown with typical restoration plants, e.g., red fescue, rye grass and white clover. Smaller scale laboratory trials were also sown in pots using Silwood wetland sludge in combination with local sand and decayed wood detritus.

Plant growth was vigorous in the field in coal shale and compost mixtures, both with and without added ochre. During the second season growth was reduced, but less so in ternary mixtures - possibly because of reduced nutrient leaching by beneficial retention of phosphate on ochre. Water percolating through the beds contained insignificant concentrations of iron and heavy metals, i.e., iron was not reduced and mobilised from ochre. In the laboratory, shorter-term experiments showed that growth

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was similarly vigorous in widely varying mixtures of ochre, sand and organic matter artificially watered with nutrients. A plot of total biomass against iron content did not show a significant correlation with iron content in the range 0-5% Fe.

Introduction

Earlier papers (Dudeney et al., 2003; Demin and Dudeney, 2003) provided geographical estimates of ochreous sludge accumulations at Coal Authority treatment operations current in 2001 and made preliminary recommendations on strategies for sludge management to replace landfill. The data were obtained partly from site visits and independent analyses of a range of discharges and treatment systems. Archival data were also employed. Based on the Coal Authority plants operating at that time in England, Scotland and Wales, and a number of uncontrolled discharges to rivers, some 2.8×10^4 tonnes per annum of sludge (15% solids basis) of quite widely varying composition was likely to be generated over the coming decades – much of it in sedimentation lagoons ahead of wetland filtration. A new estimate of 2.5×10^4 t/a has recently been made (Neville and Hancock, unpublished) taking account of 24 operating plants and 7 discharges awaiting development of treatment systems. Of course, changes in flow patterns underground and construction of new treatment systems (currently six per year) will alter the ochre produced and new data will be needed periodically as such systems are installed.

A conclusion of the earlier work was that compositional and geographical variations may preclude added value applications for ochre. For instance, although ochre can form the basis of a technically successful ferric sulphate coagulant, producers do not wish to risk quality assurance through use of ochre and relevant water/wastewater utilities have not prioritised use of ochre-derived coagulants. Dried ochre might provide a useful adsorbent for phosphate in agricultural run-off (Heal, et al. 2003), with final disposal perhaps as a slow-release fertilizer in artificial soil systems but this possibility has yet to be fully established. A further conclusion was that lower-value bulk applications (in combination with other materials) could be targeted more intensively, such as in the civil engineering construction sector, e.g., ochre in bricks, cement and ceramics, for which there is considerable precedent; and in brown-field site restoration, e.g., ochre in artificial soil formulations, which is a relatively new approach. Thus, some interest was shown at the time in lagoon sludge as an iron-balancing component in cement (Blue Circle Cement/Lafarge) and also as a component of ceramic formulations (Ferguson Wild & Co). However, there was no apparent market for less well-defined ochre deposits, particularly those composed of wetland sludge, even though they often contain substantial proportions of carbon and plant nutrients, and might be useful components in artificial soils.

The present paper describes ongoing work aimed at establishing the possible application of ochreous sludge in artificial soil formulations. Particular issues are minimisation of undue leaching of iron (and associated trace heavy metals) from the sludge into the environment, e.g., into plant tissues or groundwater, particularly by reductive processes, and maximisation of the beneficial retention of nutrients on ochre, particularly the retention of phosphate in a form available to plants, but not lost to groundwater. Thus, phosphate is well known to adsorb onto iron hydroxyoxides (such as ochre), but, as ochre reactivity can be very variable, is not well established as a slow release fertiliser. The mode of formation of ferrous iron from sulphide oxidation in pyrite underground (Spotts and Dollhopf, 1992) - and also from microbiologically-induced reduction of ochreous materials (Johnson and Bridge, 2002) – and the toxic effects of dissolved ferrous iron have been reported, but less is understood about the tendency of iron to become substantially reduced and mobilised in soil media.

The paper is also associated with potential re-use of coal spoil and composted sewage sludge in the restoration of brown-field sites such as forestry land and open amenities - not primarily with agricultural use. In respect of coal spoil, a huge quantity is available in principle, even though much has already been subject to surface restoration (DETR, 1999). Some 60-70% of weathered spoil normally occurs at fine particle sizes, suitable in principle for application as a soil matrix. The remainder is at coarser sizes and, as such, represents a substantial resource of engineering fill, particularly if previously hardened through historical spontaneous combustion - and especially if the accompanying fine sized materials could also be marketed. With regard to sewage sludge, application as a soil conditioner and source of nutrients has been used extensively in agriculture, but the water industry is seeking new sustainable outlets for this product of wastewater treatment due to pressures on the other routes of sludge management, such as landfill and incineration. Application of sewage sludge has also been widely used to restore growth on coal tips, and this material has therefore considerable potential as the organic matter (OM) source in the formulation of artificial soils from coal mining wastes and residues.

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Because of the need to satisfy increasingly demanding environmental regulations, the result of artificial soil formulation must be an identifiable and beneficial product and not merely a combined use of mixed waste materials, which happen to function together as a soil. In other words, the blending of different waste materials is not intended to facilitate the disposal of any particular material but each must provide a beneficial attribute to the end product, which is improved as a result of optimal blending together of the various component wastes. Thus, it is one objective to provide a soil cover where none currently exists, maybe as a result of former industrial activity, and another to demonstrate that the combination of bulk components forms a new commodity, i.e., a new growing medium having particular technical and environmental attractions.

Of course, artificial soil does not, in general, represent a new concept. Synthetic composts and hydroponic growing media have been studied and employed for many years (e.g., Rowell, 1994). In particular, various recent and older studies have sought to employ different bulk wastes in soil as the closure of many European coal mines has led to increasing requirements for restoration and rehabilitation of derelict land arising from surface installations and waste tips. Emphasis has been placed on agriculture and forestry since they are the most common land uses for reclaimed colliery spoil (Hester and Harrison, 1997). As sufficient soil was often not available, different materials have been used on top of the spoil as growing media: e.g., bottom ash and lime (Tedesco et al., 1999); arbuscular-mycorrhizal fungi (AMF) and composted sewage sludge (Thorne et al., 1998; Kleber et al. 2000); sewage sludge and limestone (Joost et al, 1987) and pulverized refuse fines (PRF) (Chu and Bradshaw, 1996). Thus, useful soil substitutes have been produced from otherwise pernicious wastes. The present work is viewed as an extension of those studies to the present circumstances, i.e., relatively iron-enriched media, to make the best possible use of bulk materials that would otherwise be waste. It is also a development based on preliminary studies carried out at the Imperial College Silwood Park Lake and Wetland (an analogue of the Coal Authority mine water wetlands), ochreous sludge from which was found to support plant growth when used in mixtures with local sand and wood waste (Dudeney et al., 2004).

Methods and materials

The main approach adopted has been to prepare, plant and monitor a representative range of small plots (Figure 1), modelling those likely to be needed in a full-scale restoration scheme. The plots were established at the Coal Authority's Woolley Mine Water Treatment Site, where suitable access, security, working space (about 40 m^2 for growth trials) and materials were available. Lagoon ochre (about one tonne) - previously recovered from the Woolley drying beds - was stored in a 3 m^3 tank on the site. Additionally, about 0.5 tonne wetland sludge was transported from the Imperial College Silwood Park Field Station (Carlile and Dudeney, 2000) and a similar quantity of sludge was excavated from the inlet end of the Woolley Wetland. At Silwood Park, the sludge was pumped from the lake and dewatered in 200 l capacity sand/gravel-drained containers (Dudeney et al., 2004). Some 12 tonnes of coal spoil (mainly black shale containing a small proportion of red shale from historical spontaneous combustion) were transported by the MED company, currently developing the Woolley site, from the tip at the SW perimeter of the site. Some 0.5 tonne dewatered composted sewage sludge ('treated conditioned sewage sludge', TCSS) was provided by Yorkshire Water Services from its Calder Vale Sewage Treatment Works.

Referring to Figure 1, a group of 24 rectangular cells of nominal dimensions 0.7x0.7x0.45 m depth (approximate capacity 0.3 tonne or 0.22 m^3, surface area 0.48 m^2) were constructed by ECS (maintenance contractors to the Coal Authority) in April 2003, using 'breeze' blocks. The location was an existing concrete pad at the northern end of the cascade/concrete lagoon compound. To make best use of the pad, Cells 1 and 2 were of slightly reduced surface area (0.35 m^2). The cells were each lined with plastic sheet and provided with a 2.5 cm deep pea gravel drainage layer, a 50 mm diameter drainage pipe and sample bottles to collect leachate.

Eighteen of the cells (Cells 1-10, 13-14, 17-18 and 21-24) were filled by hand in Spring 2003 with designed mixtures of the coal spoil, ochre and/or stabilised sewage sludge in the volume proportions shown in Figure 1. For example, for Cell 9, ten buckets (about 150 l) of shale, 2 buckets of ochre and 2 buckets of compost, were mixed by coning and quartering. Shale chunks greater than about 2 cm, particularly of red shale, were discarded (but screening was not employed). The mixtures were transferred to the cells and lightly compacted in place. After the cells had been filled, the pipe outlets were protected by bunds and aluminium sheeting. Cells 11-

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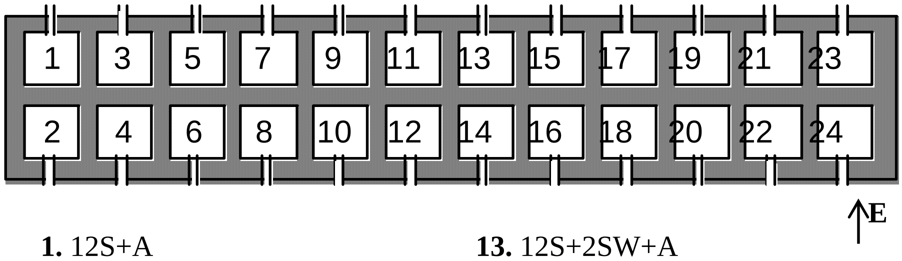
12, 15-16 and 19-20, were similarly filled in early Spring 2004, after allowing a season for the Woolley wetland ochre to weather before use.



1. 12S+A
2. 12S+BCD
3. 10S+10 cm layer 2S+2C+A
4. 10S+10 cm layer 2S+1C+ +1WDB+BCD
5. 12S+2WDB+A
6. 12S+2WDB+BCD
7. 10S+4WDB+A
8. 9S+5WDB+BCD
9. 10S+2WDB+2C+A
10. 10S+2WDB+2C+BCD
11. 10S+2WDB+2C+A (4/04)
12. 10S+2WDB+2C+BCD (4/04)
13. 12S+2SW+A
14. 12S+2SW+BCD
15. 10S+2WW+2C+A (4/04)
16. 10S+2WW+2C+BCD (4/04)
17. 9S+2SW+3C+A
18. 9S+2SW+3C+BCD
19. 12S+2WW +A (4/04)
20. 12S+2WW+BCD (4/04)
21. 9S+5C+A
22. 7S+7C+BCD
23. 11S+3C+A
24. 11S+3C+BCD

Figure 1. Disposition and contents of Woolley artificial soil cells, established in Spring 2003 (except where shown for 2004): **S**, shale; **WDB**, Woolley drying bed ochre; **SW**, Silwood wetland ochre; **WW**, Woolley wetland ochre; **A**, perennial ryegrass; **BCD**, equal numbers mixture of red fescue, timothy grass and white clover (bucket measure 15 l, e.g., 12S refers to 180 l shale).

Following 1-2 weeks settling time, the filled cells were watered (5 l each of tap water), furrowed on a grid pattern to 5-10 cm depth, carefully broadcast with seeds (donated by DLF Perrifields Seed Merchants), gently raked, and covered with plastic sheet (to reduce desiccation during germination). Odd-numbered cells were sown with perennial ryegrass (*Lolium perenne*) and even-numbered cells with a recommended restoration mixture of red fescue (*Festuca rubra*), timothy grass (*Phleum pratense*) and white clover (*Trifolium repens*). Ryegrass (containing approximately 590-600 seeds/g) was sown at the recommended rate of 3.2 g/m^2 per plot, i.e., 1.12 g, 665-700 seeds in Cell 1 and 1.54 g or 910-920 seeds in Cells 3, 5, 7, 9, 13, 17, 21 and 23. To provide similar numbers of seeds overall, and equal plant proportions in the mixed cultivations, red fescue, timothy grass and clover – containing approximately 1050, 1865 and 1300 seeds/g, respectively - were sown at 0.23, 0.13 and 0.18 g/cell or roughly 200 seeds of each type in Cell 2, and similarly for Cells 4, 6, 8, 10, 14, 18, 22 and 24. After germination, (about 2 weeks) the plastic sheets were removed and plants were left to develop without interference. In particular, they were not watered except by natural rainfall.

The supporting (laboratory) approach was similar to that adopted previously (Dudeney et al., 2004) and used complementary materials to those employed in the Woolley trials. Exact replication of the Woolley setup was not possible as the materials had to be weathered for a year prior to use in order to make handling more amenable. Thus, 16 x 12 cm diameter (400 cm^3) plastic pots (in replicates of five) were filled with selected mixtures of dewatered Silwood Park wetland sludge, sand and wood detritus (as OM) both from the surrounding land. They were sown with the same seed types in similar ratios to those outlined above, and, after germination, were sustained with water from the Silwood Lake (which was known to provide sufficient dissolved nutrients for normal growth). Ambient laboratory conditions were employed, with daylight simulation using a time-regulated strip-lighting system. After 10 weeks growth, shoots and roots were recovered from selected pots, separated, washed, dried at 105˚C, calcined at 600˚C, leached with aqua regia and analysed by ICP-AES spectroscopy.

Results and discussion

Fieldwork

The cells were designed to give a two-spit depth for sustainable rooting and fines retention and a large enough surface area for at least 500 plants to develop in each without undue crowding. Twenty-four cells was the maximum that could be accommodated at this scale, so a pragmatic (rather than a fully statistical) matrix of conditions was employed. The nominal proportions of the materials used and their compositions are shown in Figure 1 and Table 1, respectively. From the figure, four types of mix (shale alone, shale plus ochre, shale plus compost, and shale plus ochre plus compost) can be discerned, in conjunction with (but not fully independently

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with respect to) three types of ochre and two types of sowings. In two cells the main growing medium was restricted to the upper 25% of the full depth. These choices made were based largely on practicality and previous experience. On the basis of occasional weighing to check the simple volumetric procedures employed, actions were normally reproducible within approximately ±5%, although adverse weather caused additional problems in metering and sowing. In particular, the settled volume of artificial soil was greater when cells were filled in wet weather. The materials employed gave a near-neutral reaction (pH 6.6-7.6) and all contained suitably low concentrations of heavy metals (e.g., Zn 0.13-0.44, Pb 0.02-0.03, As <0.01 g/kg on a dry mass basis). Acid leachable iron concentrations for shale (S), Woolley drying bed ochre (WDB), Woolley wetland ochre (WW), Silwood wetland ochre (SW) were respectively, 20, 574, 209 and 487 g/kg. The TCSS contained total N 2.9%, C/N ratio 15:1, P (as P_2O_5) 3.1% and K (as K_2O) 0.25%, and thus functioned as a typical compost. The other materials contained relatively insignificant levels of available N, P and K. SW and WW (but not WDB) had substantial organic matter, e.g., SW, approximately 10% OM. All the materials exhibited a large LOI variously from combined water, OM and oxidation. As the materials were substantially variable in composition, more detailed characterisation was not warranted.

Figure 2 indicates qualitatively the general success of germination and growth of ryegrass, red fescue, timothy grass and white clover (together with incidental tomatoes, poppies and general weeds) in the first year under the exposed conditions typically experienced at Woolley, provided compost was present (Cells 3-4, 9-10, 17-18, 21-24). Germination also occurred satisfactorily in shale alone (Cells 1-2) and shale plus ochre alone (Cells 5-8, 13-14). However, hard surface caking in dry weather with shale alone (arising from self-adherence effects of contained clays) prevented the emergence of seedlings. The presence of ochre reduced caking. Subsequent growth in the absence of added compost was generally poor but healthy during the first season, although larger proportions of ochre (Cells 7-8) caused plant stems and leaves to become chlorotic (yellow). As expected insufficient nutrient was available in shale or ochre to support sustained growth. Nonetheless, growth in the presence of Silwood wetland ochre (which contains plant detritus) was maintained into the second season. A third season of observation will be necessary to substantiate this effect of wetland ochre.

Further examination revealed, not surprisingly, that use of more compost increased plant growth (Cells 21-24) and that a moderate decrease in vigour occurred during the second year as nutrients were consumed or leached out. Observation of Cells 3 and 4 (shale plus compost and shale plus compost plus ochre, respectively), also suggested a significantly positive effect of ochre. Thus, luxuriant growth in both cells in the first year was replaced by relatively strong growth in Cell 4 in the second year (Figure 3), despite less compost being present in this cell. This could be explained as the ochre preventing a loss of phosphate during drainage. However, as several variables changed at once, e.g., different plant species were grown, careful substantiation will be necessary. All grasses flowered during the second season (excluding those cells planted this year) confirming the success of all four varieties of seed.

Rooting was observed via 10 cm diameter holes augered to the full depth of the artificial soil and covered. After several months, removal of the covers indicated healthy rooting to near the full depth in presence of compost, but much shallower penetration in shale and ochre alone. Quantitative comparison of top growth and root runs was not practical owing to the presence of ruderal species and grass cropping by rabbits. However, laboratory experiments gave information on this matter (see below).

Leachate emerging from the base of the cells after heavy rain was usually clear and water-white, except when compost was used - in which case it was pale yellow in colour as a result of humate content. Dissolved iron was below 0.5 mg/l and particulate iron was minor or absent. The water was normally hard with large concentrations of calcium, sodium and magnesium, but insignificant quantities of heavy metals. Dissolved organics were often close to 50 mg/l but as expected, concentrations decreased with time, and were substantially reduced from their initial values during the second season.

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Figure 2. Growth of plants: general view, left; Cells 9/10, right. From top down: 5/6, 9/7, 5/8, 15/9.

Figure 3. Luxuriant growth of grass, tomatoes, etc., in Cell 3 (foreground) and Cell 4 (background) in 2003, and reduced growth (but relatively strong in Cell 4) in 2004.

Laboratory work

Precise scientific substantiation of comparative growth characteristics is evidently difficult in the field. A series of analogous laboratory experiments on a smaller, more controllable, scale are therefore being used to provide supporting results, particularly with respect to the relationship between shoot and root growth and between the total biomass produced and the proportion of iron added to the artificial soil mixture (Table 1, Figure 4). Table 1 indicates seed germination viability ranging from 50-70%. Timothy grass was difficult to discern from other grasses during early growth stages and was only positively identified during the second season of the Woolley trials by structural and vegetative differences not evident during the 10-week growth trial at Silwood Park. Shoot/root mass ratios were variable in the range 4-9, without any clearly discernable pattern, but nevertheless indicative of healthy early-stage plant development (Rowell, 1994) before root growth was fully established. Figure 4 shows a relationship between total biomass produced and iron content.

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Table 1. Root and shoot biomass from artificial soil formulations

Sludge (%)	Sand (%)	OM (%)	Viability (%)	Shoot (g)	Root (g)	S/R Ratio
1.0	-	-	70	0.36	0.041	8.8
0.4	0.6	-	61	0.23	0.032	7.2
0.2	0.8	-	56	0.16	0.043	3.7
-	1.0	-	56	0.29	0.047	6.1
0.6	-	0.4	68	0.25	0.035	7.1
0.3	-	0.7	62	0.18	0.065	2.8
-	-	1.0	66	0.22	0.062	3.5
0.3	0.4	0.3	63	0.18	0.055	3.3
0.5	0.3	0.2	70	0.21	0.037	5.7
0.2	0.6	0.2	73	0.26	0.058	4.5
0.3	0.3	0.4	62	0.23	0.055	4.2
-	0.6	0.4	53	0.35	0.067	5.2
0.1	0.9	-	51	0.23	0.036	6.4
0.1	0.8	0.1	60	0.26	0.051	5.1
-	0.9	0.1	50	0.23	0.029	7.9
				0.24	0.051	4.7

Despite an evident scatter in the results (Figure 4), the total biomass appears to be independent of percentage iron and no significant correlation, positive or negative, was found. Similar scattered correlations (not shown here) were observed between iron-in-roots and iron-in-shoots. A marginal negative correlation (also not shown) was seen between phosphate-in-shoots and available iron, thus possibly a marginal tendency for iron-in-ochre to retain phosphate at the expense of biomass.

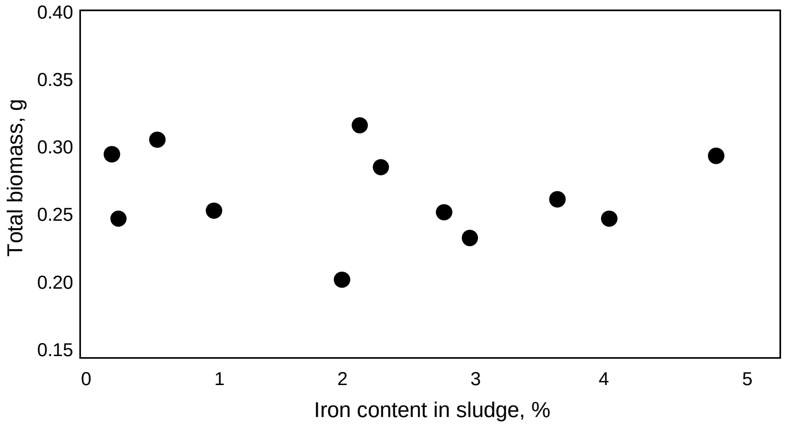

Figure 4. Total biomass as a function of iron content in artificial soil from sand, wood waste and Silwood wetland sludge

Conclusion

The results of field work during 1-2 seasons have confirmed qualitatively that typical restoration plants, ryegrass, red fescue, timothy grass and clover germinate and grow well in artificial soil formulations containing a typical coal shale base, composted sewage sludge and ochre in quite widely varying volume proportions, such as 5:1:1 and 2:1:1, respectively. Initial evidence suggests that the presence of ochre enhanced growth in the second season, possibly by retention of phosphate, which would otherwise have leached from the compost. Iron was not reduced and mobilised from the top 0.4 m of such a soil and leachates collected did not contain toxic concentrations of metals or anions. Convincing evidence has not been found of a significant soil conditioning effect, similar to compost, from organic matter contained in wetland sludge. Precise comparisons being difficult on a field scale, laboratory experiments were employed to determine the effects of iron on plant growth in widely varying mixtures of sand, wood detritus and ochre sludge - used in conjunction with the same plant species as in the field experiments and nutrient-rich lake water. No positive or negative impact was observed for iron content on biomass produced but this may be as the experiment only extended for the duration of the germination period. Experimental work is continuing to determine longer-term responses in the field generated in the second and third seasons, and to place the laboratory work on a firmer statistically-sound basis on a 400

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cm^3 scale at Imperial College and a 10 l scale at the Bradley laboratories of Yorkshire Water Services.

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