Article

Recycling of Blast Furnace and Coal Slags in Aided Phytostabilisation of Soils Highly Polluted with Heavy Metals

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Abstract: (1) Background: The growing demand for developing new methods of degraded land remediation is linked to the need to improve the soil environment, including post-industrial soils. Biological methods such as the aided phytostabilisation technique are the most common methods applied to achieve effective remediation. This study aimed to determine the technical potential of methods using novel or yet not used soil amendments, such as blast furnace slag (BFS) and coal slag (CS), with Dactylis glomerata L. as a test plant. (2) Methods: The experiment was conducted on post-industrial area soil with high concentrations of Cu (761 mg/kg), Cd (23.9 mg/kg), Pb (13,539 mg/kg) and Zn (8683 mg/kg). The heavy metal content in roots and the above-ground parts of plants and soil was determined by flame atomic absorption spectrometry. (3) Results: The addition of BFS to the soil was the most effective in increasing Dactylis glomerata L. biomass yield. The Cu, Cd, Pb, and Zn concentrations were higher in the roots than in the above-ground parts of the plants. BFS and CS induced a considerable increase in soil pH, compared to the control treatment. The addition of BFS also produced the greatest significant decrease in the Pb content in soil following the phytostabilisation process. (4) Conclusions: In view of the above, the use of BFS in the aided phytostabilisation in soils contaminated with high levels of Cu, Cd, Pb, and Zn can be recommended for larger-scale in situ projects.

Keywords: soil contamination; metal immobilisation; soil amendments; Dactylis glomerata L.

1. Introduction

Contamination of soil in various post-industrial areas has attracted increasing interest for many years [1–4]. Research has been particularly focused on soil concentrations of heavy metals (HMs), which are regarded as a particular hazard to human health [5]. The negative effects of heavy metals are not immediate and are sometimes not visible for several years. The widespread pressure for investments has caused a shortage of land for residential buildings as well as reserve land for greenery. This reserve could be provided by degraded and contaminated land in post-industrial areas because, apart from the obvious need for their remediation, their incorporation in the public area system is also important.
Although they provide an investment reserve, degraded land is also beginning to play an important role in a system of green infrastructure in towns, performing a range of ecosystem services [5,6]. Kowarik [7] showed that degraded land becomes naturally active when left for free vegetation growth. The new plant associations composed of native and foreign species, called a ‘novel urban ecosystem’, are fully functional areas in a town’s natural system, providing important support in improving the residents’ living conditions [8]. However, degraded and contaminated land is usually used by residents of nearby areas as informal recreation areas. Given this situation, measures have to be taken to apply nature-based solutions (NBS) to remediate sites polluted by heavy metals [9]. Such solutions undoubtedly include the aided phytostabilisation technique, which reduces heavy metal mobility, decreasing their potential downward migration in the soil profile [10]. Apart from high contamination levels, soil in post-industrial areas is characterised by difficult living conditions for plants [11].

For this reason, it is reasonable to use various soil amendments (aided phytostabilisation), improving the soil’s physical, chemical, and biological properties, stimulating, in turn, the greenery growth in an area [12]. Many types of soil amendments described in the literature can be used successfully in the aided phytostabilisation technique [13]. However, new, cheap, and effective materials are still being sought to aid the phytostabilisation of soils contaminated by heavy metals. This study presents an evaluation of novel materials not yet applied in aided phytostabilisation, i.e., two types of slag: blast furnace slag and coal slag.

Anthropogenic aggregates such as blast furnace slag and coal slag are valuable and necessary for economic and social development, which is why they must be produced, used, and disposed of following the principles of sustainable development [14]. Although recycled aggregates in the construction sector are important from an environmental perspective, aggregate management and recycling can also be a burden. The production of recycled aggregates increases with the construction sector growth, and its management is becoming increasingly difficult. The estimated annual demand for aggregates in Europe now amounts to 3 billion tonnes [15]. The production of secondary aggregates, comprising recycled and reused aggregates, amounts to 327 million tonnes, accounting for 10.6% of their total production in the EU [16]. Managing recycled aggregates also has important consequences for the environment, especially concerning their transport and storage. However, if this waste is properly recycled and reintroduced to the value chain, it can provide added value, both in the economic and environmental aspects [17].

Meanwhile, from reducing CO$_2$ emissions, which is one of the global priorities for the coming years, it is essential to use sustainable materials in every aspect of the economy. Slags are materials with vast application possibilities in the construction industry. They are used as a construction binder for concrete. This property of slag is significant because the process of cement production is characterised by high emissions and energy and material intensity [18]. The efficient use of cement and concrete products and their reuse as construction waste at the end of a project’s life impact the sustainability of concrete. As does the design of concrete mixes, which directly impact the thermal efficiency of buildings. The thermal inertia of concretes means that smartly designed modern concrete buildings can use up to 75% less energy over their entire life cycle. Traditionally, limestone has been used in cement production. Limestone is widely available, but over 60% of our industry’s CO$_2$ emissions are due to the conversion of limestone to lime, called decarbonisation. Limestone substitution is necessary to produce low-carbon concrete. Materials such as coal or lignite ash, blast furnace slag, aerated concrete meal, and demolition waste fractions are already decarbonated. This means that they can substitute for limestone, thus avoiding CO$_2$ emissions from their conversion to lime during the production process. By-products of the energy sector (fly ash and slag) and the steel industry (blast furnace slag) are used as geopolymers and concrete to produce alkali-activated cements [19,20].

After furnace smelting, liquid blast furnace slag (BFS) is transported in cisterns to dumps. The cooling process results in the crystallisation of many minerals, mainly silicates
and aluminium silicates of calcium and magnesium. With a tonne of the final product, approximately 200–400 kg of liquid slag is often produced. The bulk density of blast furnace slag is lower than that of natural aggregates [21]. It has insulation properties, good fire resistance, and thermal strength. Its absorption capacity is greater than that of natural aggregates, which is a consequence of its higher porosity. However, it has mainly closed porosity, with no connections between pores and fine material structures around them. The BFS chemical composition depends on that of the raw materials. Its main components, SiO$_2$, Al$_2$O$_3$, CaO, and MgO, account for 95% of the whole [22]. The other components include Fe$_2$O$_3$ and Mn$_2$O$_7$. Coal slag (CS) is a type of waste produced by heating plants in the combustion of fine coal. Power plants usually use fine coal furnaces or cyclone furnaces. Ash collected from fine coal furnaces consists of fly ash and boiler slag. Burnt slag is red and large amounts of fine aggregates and hard sinters are produced in coal combustion [23].

This paper presents the results of a pot experiment with novel soil amendments, i.e., blast furnace slag (BFC) and coal slag (CS) used in aided phytostabilisation of heavy metal contaminated soils. The effectiveness of heavy metal immobilisation was evaluated by determining the biomass yield and chemical composition of roots and above-ground parts and the selected physicochemical properties of soil, both before and after the experiment.

2. Materials and Methods

2.1. Soil Sampling Site

Experimental soil samples were collected from land located in northeastern Poland, where scrap metal had been stored directly on the ground for several dozen years. In consequence, the Cu, Cd, Pb, and Zn concentrations in the soil considerably exceeded the norms valid in Poland [24]. The soil was collected by the methodology described by Radziemska et al. [25]. All soil samples were carefully transferred to clean polyethylene bags before being transported to the laboratory where they were air-dried at room temperature and sifted through a 2 mm sieve and stored in a refrigerator at 4 °C.

2.2. Amendments

Blast furnace slag (BFS) and coal slag (CS) were used in the experiment. Both materials have a similar, alkaline pH. The aeration level of BFS is twice that of CS. BFS contains considerable concentrations of Si, Al, and Ca, whereas CS consists mainly of C, followed by Al and Si (Table 1). The presence of these elements shows that alkaline silicate bonds are formed in the studied slags. Considering a much higher Si and Na content in BFS, it is much more capable of forming silicate polymer bonds. BFS is highly porous, which can be observed in images taken with an electron microscope (SEM) (Figure 1a). Pores in CS are smaller, but their surface is rough (Figure 1b).

2.3. Greenhouse Experiment Setup

The greenhouse experiment was conducted in 5.0 kg polyethylene pots. Each was filled with a soil mixture with BFS or CS at 3.0% (w/w) and with unamended soil (0.0%, w/w, control) and mixed thoroughly. The pots were then kept in a dark room for two weeks to ensure stabilisation under natural conditions prior to planting. The experiment was then performed in quintuplicate, using 5 g of seeds per pot (experimental plant: Dactylis glomerata L. cv. Berta). The following conditions were maintained in the greenhouse: natural day/night conditions; during the day (14 h), the air temperature was 26 ± 3 °C, and ~10$^\circ$ lower (16 ± 2 °C) at night (10 h), with a relative humidity of 75 ± 5%. The plants were watered every second day with distilled water to 60% of the maximum water holding capacity of the soil. Soil moisture content for each pot was maintained at the field capacity every three days. After the experiment was completed (54 days), soil samples and the above-ground parts and roots of the experimental plant were collected from each pot.
Table 1. Physicochemical composition of blast furnace slag (BFS) and coal slag (CS) used in the experiment.

| Parameter | Unit   | BFS      | CS       |
|-----------|--------|----------|----------|
| Surface Area | m$^2$/kg | 315     | 450     |
| pH        | -      | 9.20    | 8.74    |
| Cu        | mg/kg  | 0.48    | 0.29    |
| Cd        | mg/kg  | 0.007   | 0.012   |
| Pb        | mg/kg  | 0.21    | 0.09    |
| Zn        | mg/kg  | 1.33    | 0.07    |
| C%        |       | 0.66    | 29.56   |
| O%        |       | 35.66   | 60.63   |
| Na%       |       | 4.78    | 0.59    |
| Mg%       |       | 2.65    | 0.73    |
| Al%       |       | 18.26   | 2.68    |
| Si%       |       | 23.52   | 2.55    |
| S%        |       | -       | 0.49    |
| Cl%       |       | -       | 0.26    |
| K%        |       | 1.50    | 0.29    |
| Ca%       |       | 8.45    | 0.97    |
| Ti%       |       | 1.53    | 0.20    |
| Fe%       |       | 2.74    | 1.05    |
| P%        |       | 0.25    | -       |

Figure 1. Scanning electron microscopy (SEM) image at 5000x magnification of the blast furnace slag (BFS) (a) and coal slag (CS) (b).

2.4. Soil Analyses

After the experiment, soil samples were taken from each pot, air-dried, sifted through a 2 mm mesh sieve, and then homogenised for analytical procedures. The soil pH was determined with an HI 221 (Orion, NY, USA) pH meter in a 1:2.5 water to soil extract. The total content of Cu, Cd, Pb, and Zn was determined by flame atomic absorption spectrometry (Varian spectrometer, Mulgrave, Australia, AA280FS), preceded by soil mineralisation in a mixture of HCl, HNO$_3$, and H$_2$O$_2$ in a MARSXpress microwave oven (CEM, Matthews, NC, USA). The quality of analyses was evaluated with a reference material (CRM 142 R), and the metal recovery rates ranged from 95% to 101%. Ultra-pure water (Milli-Q System, Kenilworth, NJ, USA) was used in the chemical analyses.
2.5. Determination of the Heavy Metal Contents in Plants

The above-ground parts and roots of the test plants were washed thoroughly with deionised water and dried after the experiment. The prepared plant material was then ground in an analytical mill (Retsch, ZM300, Hann, Germany) and the above-ground part and root samples were mineralised in concentrated nitric acid and hydrogen peroxide in a microwave oven (MARSXpress, CEM, Matthews, NC, USA). The concentrations of Cu, Cd, Pb, and Zn in the digestates were estimated by a Varian, AA28OFS (Varian, Mulgrave, Australia) flame atomic absorption spectrometry after dilution of the digestates with ultra-pure water and filtration using Whatman 42 filters.

2.6. Statistical Analysis

The means of five replicates of the experiment were used in the statistical analysis. An analysis of variance (ANOVA) was performed with LSD tests at the significance level of $p < 0.05$ using Statistica 13.3 software.

3. Results

3.1. Above-Ground Parts Biomass of Dactylis Glomerata L.

Figure 2 shows the yield of above-ground parts of Dactylis glomerata L. A significantly ($p < 0.05$) lower yield was obtained in the control series compared to that with the soil amendments (BFS or CS). The use of BFS in the experiment increased the experimental plant yield by 53%, compared to the control series. A similar relationship was observed for the other soil amendment (CS), but its impact on the Dactylis glomerata L. yield was slightly lower (30%).

![Figure 2. Above-ground biomass yield of Dactylis glomerata L. under different treatment conditions (Control, BFS, and CS).](image)

3.2. Effect of BFS and CS on the Distribution of Heavy Metals in Dactylis Glomerata L.

The heavy metal (Cu, Cd, Pb, and Zn) content in the roots and above-ground parts of Dactylis glomerata L. was closely correlated with the level of HM contamination of soil and the addition of BFS or CS (Figure 3). The content of all the studied heavy metals was found to be significantly ($p < 0.05$) higher in the roots than in the above-ground parts. A positive impact on individual heavy metal contents in Dactylis glomerata L. was observed after the addition of BFS, which reduced the concentration of Cu, Cd, Pb, and Zn in these parts of the plant by 40%, 79%, 28%, and 57%, respectively. The highest accumulation of Cu, Cd, Pb, and Zn in the roots, compared to the control (no amendments), was found in the plants grown in BFS-amended soil. The effect was the most visible for Pb, whose content in the roots was 1.6 times higher than in the control.
3.3. Soil Properties

The soil pH was found to increase significantly (p < 0.05) after the addition of each of the soil amendments (Figure 4). This is demonstrated most strongly in the case of BSF, which caused a pH increase by 1.2. A comparison of the Cu, Cd, Pb, and Zn content in the experimental soils collected from post-industrial areas, with the acceptable levels laid down in the Regulation of the Polish Minister of the Environment [24], shows that the HM content in the soil was exceeded considerably (Table 2). It was particularly high for Pb and Zn, where the acceptable level was exceeded by over 22 and 4 times, respectively. The total Cu, Cd, Pb, and Zn content after the experiment is shown in Figure 5. The application of BSF significantly decreased total Cu, Cd, Pb, and Zn concentrations in the soil, as compared to the control (unamended soil), by 40%, 27%, 32%, and 49%, respectively.
| Parameter | Unit  | Value  | National Limit [24] |
|-----------|-------|--------|---------------------|
| pH        | -     | 8.34   | -                   |
| Cu        | mg/kg | 761.18 | 600                 |
| Cd        | mg/kg | 23.90  | 15                  |
| Pb        | mg/kg | 13,539 | 600                 |
| Zn        | mg/kg | 8683   | 2000                |

**Figure 5.** Contents of Cu, Cd, Pb, and Zn in the soil after the application of BFS and CS.

**4. Discussion**

Recent years have brought a significant development in the use of various materials that can act as soil amendments in aided phytostabilisation. Reports in the literature discussing aided phytostabilisation usually cover the use of soil amendments which aid the heavy metal immobilisation process, such as biochar, compost, limestone, and halloysite [25–27]. A wide range of waste materials, including slags of different types, can be used as novel and potentially usable soil amendments and they can often provide an alternative to more traditional materials [28–30]. The use of plants in remediated treatments stimulates soil-forming processes and restoration of biological life on degraded surfaces, thereby enabling gradual improvement of the soil quality and protecting it against erosion by water and wind [31]. Moreover, remedied land has a positive impact on the landscape and increases the greenery area, which is of particular importance in a post-industrial space [32]. A significant role in aided phytostabilisation is played by the creation of uninterrupted plant cover. Its growth can be supported by the use of soil amendments [33]. The authors demonstrated a significant impact of two types of slag used as soil amendments on the growth and yield of *Dactylis glomerata* L. The highest yield of this plant, compared to the control series, was found following the application of BFS. Similar findings were obtained in a study of the basic slag impact on the yield of *Phaseolus vulgaris* L. by Negim et al. [34]. Wang and Cai [35] examined the addition of BFS to the soil used for the cultivation of *Zea mays* L. and demonstrated that the level of available Fe increased following its addition.
It is noteworthy that maintaining the correct Fe level in a plant is extremely important for key processes such as photosynthesis, cellular respiration, metabolism of nucleotides, and synthesis of chlorophyll, which results in obtaining an appropriate amount of plant biomass [36]. Moreover, BFS contains large amounts of Ca, Mg, and Fe, which are important elements in the plant growth process, and its use seems justified in nutrient-deficient soils [37]. Further research demonstrating the positive impact of a 3% BFS addition to the soil on Pinellia ternata L. yield was conducted by Ng et al. [38]. Measures aimed at HM immobilisation in the soil sorptive complex to prevent them from migrating to other parts of the food chain deserve special attention [39]. Therefore, the use of various soil amendments to improve the physical and chemical soil properties and to reduce the heavy metal absorption by plants seems to be the most beneficial method for their remediation [40]. The rational use of BFS or CS as a regulator of HM availability to plants reduces the element uptake by plants, which was demonstrated by the authors. It should be noted that HM concentration in plants depends mainly on the HM amounts and species in which they occur, on specific features of plant species, the ability of the root system to take up an element and on the soil pH, granulometric composition, and organic matter content [41,42]. The most positive impact in this study was observed following the addition of BFS, which reduced the Cu, Cd, Pb, and Zn in the above-ground parts of Dactylis glomerata L. Lead is one of the main environmental pollutants, highly toxic to humans, which is why it is important to reduce its presence in soil and plants [43]. Since it migrates to the above-ground parts of plants mainly through roots [44], high Pb concentrations bring about strongly toxic symptoms of poisoning in plants, manifested in inhibition of metabolic processes (photosynthesis, respiration, transpiration, and water uptake) [45]. Apart from metal concentration in soil, the total metal amount accumulated in the roots is the most important parameter used to assess the phytostabilisation potential in plants [46]. It is owing to the BFS used by the authors in the current study that intensive accumulation of the element in Dactylis glomerata L. roots was observed, thereby preventing its migration to further parts of the food chain. Lead is neutralised in root cells by accumulation in vacuoles, cell walls, intercellular spaces, dictyosomal pores, and endoplasmic reticulum [47]. The soil solution pH is a factor with a great impact on physicochemical and biological processes in soils [48]. Moreover, it is one of the main parameters used in the assessment of soil contamination bioavailability [49]. The availability of mobile heavy metal species increases in acidic soils, which is a consequence of increased element solubility and their decreased adsorption on soil colloids [50]. Therefore, the use of remediation techniques which increase the soil pH seems to be reasonable. The soil amendments used by the authors (BFS and CS) led to a significant increase in the soil pH. This effect was also confirmed by Liang et al. [51], who studied different soil amendments and their use in the remediation of Pb contaminated soils. The authors obtained a similar increase in the soil pH after the application of blast furnace slag. Cadmium mobility in soils increases at pH 6.0–5.5, whereas the mobility of Zn and Cu increases at pH 5.5–5.0 and Pb at pH < 4.5 [52]. An analysis of the results showed that the content of Cu, Cd, Pb, and Zn in the soil was significantly reduced by the addition of BFS. Negative charges on the BFS surface can adsorb positive ions by electrostatic attraction, and the presence of pores on its surface provides favourable conditions for heavy metal adsorption [51]. The authors observed that the use of BSF produced much better remediation results for the soil on which Zea mays L. was grown than the use of biocarbon or fly ash.

Reutilisation of industrial waste such as slags depends greatly on their properties, which can vary considerably depending on the source (e.g., iron slags, steel slags). Some slags can contain elevated concentrations of elements (e.g., Cr, V, F, Cl) that can have a potential risk of soil and groundwater contamination due to their leachability. Therefore, the slags should be characterised in detail before their application in soil remediation. To evaluate and avoid the environmental risks from slags, suitable environmental regulations and standards should be taken into consideration. Slags as soil amendments should be used at specific doses due to their cementitious properties, which can affect the physical
structure of soil [53]. Future projects on aided phytostabilisation with the slag amendments should include environmental risk analysis to prevent hazardous pollutants from entering the environment. The long-term performance of slags for metal immobilisation in soil–plant systems should be also evaluated.

5. Conclusions

The condition of the environment is deteriorating, and the area of degraded lands requiring remediation is increasing, which is why it is necessary to seek new methods to restore their usability. Aided phytostabilisation is particularly noteworthy, as it is safer and less interfering with the environment than other remediation techniques. The experiment demonstrated that BFS and CS contribute to improving heavy metal immobilisation effectiveness. BFS proved to have the most positive effect on the *Dactylis glomerata* L. biomass yield and increased the contents of Cd, Cu, Pb, and Cr in roots and soil pH, and significantly decreased the total Cu, Cd, Pb, and Zn concentrations in soil. Slags are cheap industrial by-products which are available in large amounts. Moreover, slag use can help in recycling and thereby decrease the environmental impact of this product. The current study was performed under small-scale greenhouse conditions under a controlled environment. Further studies, especially in field conditions, are highly recommended to confirm the efficiency of *Dactylis glomerata* L., BFS, and CS in aided phytostabilisation of soils contaminated with heavy metals.

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