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Research article

Testing of phytotoxicity of mining waste to determine the direction of future development

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Abstract: Energy industry and coal mining produce large amounts of mining waste. The material is accumulated on dumps and heaps which alter the landscape appearance. As the material is waste from mining, there are concerns from possible environmental contamination. On the other hand, some scientific publications mention favorable properties of this material and claim that it can be possibly used for example in land reclamation. This research is focused on the testing of phytotoxicity of samples collected from mining waste heaps. The pH values of tested samples ranged from 7.00 to 8.67. In the phytotoxicity tests, vital seeds of plants *Sinapis alba.* L., *Hordeum vulgare* L., and *Cannabis sativa* L. were used. The tests did not exhibit high phytotoxicity. Contrariwise, many results showed a high rate of growth stimulation (nearly 80%). Average values (median) of growth inhibition observed in individual samples for all three plant species in the share of 100% (with no addition of OECD soil) ranged from −13.96% to −33.73%. The obtained results can confirm the positive properties of mining waste in terms of phytotoxicity, resp. the experiments did not confirm the phytotoxic effects on the tested plants. Mining waste/material could have a positive effect on plants and thanks to its properties it could be used e.g., in land reclamation.

Keywords: coal mining; growth inhibition; *Sinapis alba* L.; *Hordeum vulgare* L.; *Cannabis sativa* L

Abbreviations: CR: Czech Republic; E: Environment; GI: growth inhibition; I: inhibition; PAH: polycyclic aromatic hydrocarbons; UTHSCSA: The University of Texas Health Science Center at San Antonio; WHO: water holding capacity

1. Introduction

Although the renewable sources of energy are recently gaining more and more attention, coal is still going to play an important role in meeting the globally increasing energy demand [1,2]. Fossil coal is a sedimentary rock developed due to the accumulation and transformation of plant matter. It consists of the mixtures of various organic compounds containing in particular: carbon (C), hydrogen (H), oxygen (O), sulfur (S) and nitrogen (N) as well as mineral substances (e.g., silicates and sulfides etc.), and rare elements such as arsenic (As), uranium (U), gallium (Ga), and germanium (Ge). Coal is one of the main energy sources in the Czech Republic (CR) and the demand for coal mining continually grows to meet industry requirements.

The increased extraction of coal leads to an increased amount of coal waste [1,3], which results in changes of the environment. On a European scale, mining and quarrying represent 28.1% [4]. In 2014, the production of mining waste in Silesia amounted to 27×10^6 Mg [3]. Due to coal mining, the mining industry has to face huge displacements of excessive material (mining waste) [2]. Mining technologies are advancing rapidly and the share of mining waste in the amount of acquired coal is decreasing [5]. The coal fuel cycle includes mining, transportation, storage, combustion and conversion. Coal mining brings about a great amount of impurities such as dust particles, organic and inorganic compounds like slates, slate clays or waste sandstones [6]. The mining material is characterized primarily by rocks and rock material (clay stones, mudstones, sandstones etc.). There are also large amounts of silica (SiO_2) in it, alumina (Al_2O_3), iron oxides (Fe_xO_y), potassium oxide (K_2O), carbon (C) and calcium oxide (CaO), sodium oxide (Na_2O) or titanium oxide ($Ti0_2$). Trace elements such as arsenic (As), lead (Pb), cadmium (Cd), mercury (Hg) [7,8], barium (Ba), chromium (Cr), tin (Sn), zinc (Zn), cobalt (Co), copper (Cu), molybdenum (Mo) and nickel (Ni) can be detected in the mining material as well [9]. Some elements (e.g., zinc, copper, molybdenum or manganese) are important and needed for organisms in low concentrations while their high concentrations may be toxic [3]. Contents of these elements are not stable and change in time with the oncoming processes [7]. Coal is also a source of polycyclic aromatic hydrocarbons (PAH). Chemical processes occurring in organic compounds stored in the dumps and on the heaps can be also affected by temperature and its changes. Phenols and their derivatives, which may get into waters also represent a potential risk [10]. Mining brings to the surface huge amounts of rock fragments called burrows, mine waste dumps, spoil banks or dumps of carbonaceous rocks, which accumulate there [1] and often become dominant in the landscape [5].

The feasibility study of mining gives insufficient or none attention to the issue of the environment including questions concerning waste generation during deposit preparation, coal extraction and processing [11]. Regarding the huge amounts of mining material, it is extremely important to find an efficient way of managing this type of waste. The currently most common method of neutralization is waste storage in dumps which have become inseparable elements in the landscape of Moravian-Silesian Region [9,12]. Nevertheless, some experimental studies suggest that coal gangue, as well as ashes from combustion municipal sewage sludge [13], can be used as soil amendments, thus improving its quality. Such an application of waste material is restricted by legislation though. Possibilities of using mining waste and conditions for managing this type of waste are regulated by Directive no. 2006/21/EC on the management of waste from extractive industries [1].

The purpose of this research was to determine the characteristics of material stored on dumps and heaps in the Moravian-Silesian Region. The research introduces results of the tests of phytotoxicity (Phytotoxkit^{TM}) conducted on mining wastes and based on studying the growth of germinating plant species of *Cannabis sativa* L., *Sinapis alba* L., and *Hordeum vulgare* L.

2. Material and methods

2.1. Study area and sampling method

Municipalities Karviná and Orlová are situated in the Czech-Polish borderland (Ostrava-Karviná coal district). The area forms a south-western part of the Upper-Silesian black coal basin which represents important coal reserves in Europe [14,15]. The Upper-Silesian Basin came to existence through tectonic activities in the period of the Cadomian, Variscan and Alpine structural stages. These changes in the Devonian [16] and Carboniferous deposits gradually led to the coverage of the southern part of the Ostrava-Karviná Coalfield with mesobasic and tertiary rocks [15]. Upper-Carbonian deposits are stratigraphically divided (Ostrava – paraliccoal molase; Karviná – continentalcoal molase) [16]. Coal seams contain primarily bituminous coal with a rapidly increasing inertit content towards the lower boundary of the Karviná limnic formation. Thanks to its high quality (sulfur less than 0.7% and phosphorus from 0.02–0.11%), the coal is also used to produce coke [15].

Tested samples originate from the border area of coal extraction (Ostrava-Karviná) with the following mining works: Mining plant 1 Lazy, Mining plant 1 ČSA, Mining plant 2 – north (the former ČSM north) and Mining plant 1 Darkov – auxiliary plant. 4 sampling points were selected: 1 (49°50'18" N; 18°26'46" E), 2 (49°50'28" N; 18°30'17" E), 3 (49°49'47" N; 18°33'39" E) and 4 (49°49'22" N; 18°33'38" E) (Figure 1).

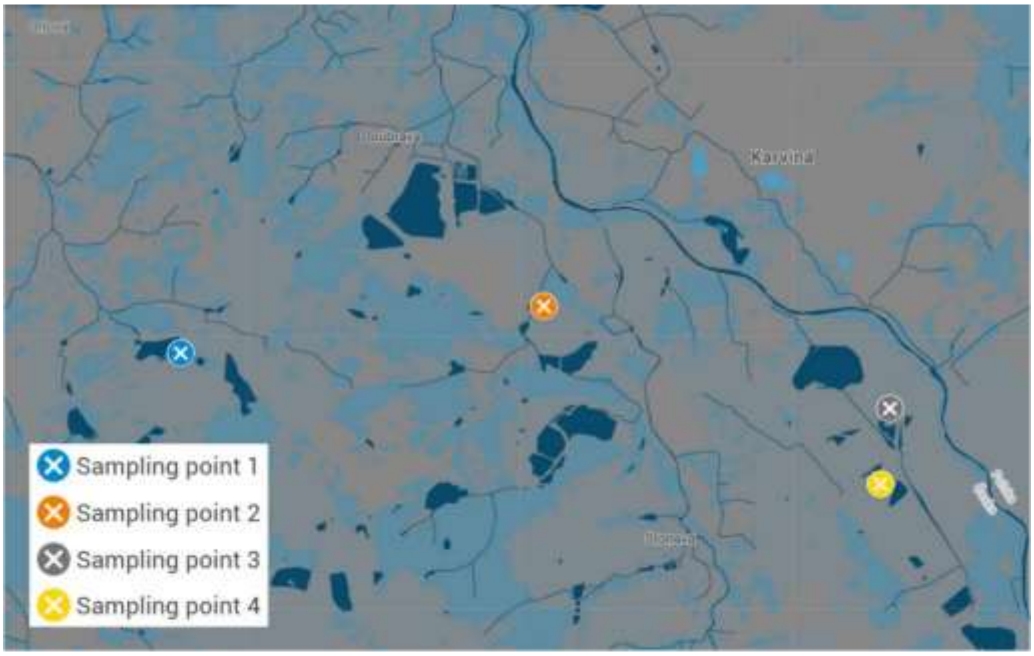

Figure 1. Schematic map of sampling points.

Representative mixed samples were taken from a depth of 0–20 cm on the predetermined sites (where mining material/waste was stored on dumps and heaps in the vicinity of mining plants). The samples were placed in an air-tight polyethylene container (volume 3 L) and transported in cooler at a constant temperature (+ 4 ℃) to the laboratory of the Department of Applied and Landscape Ecology, Mendel University in Brno (Czech Republic) for the phytotoxicity tests.

2.2. Sample preparation and phytotoxicity tests

Prior to the phytotoxicity test, the samples were dried at a laboratory temperature to constant mass, all admixtures or stones were removed, and the samples were then sifted through a 2 mm sieve with using the shaker ModelRetsch AS 200. A Phytotoxkit^{TM} test [17] was chosen for the assessment of the effect of samples on higher plants. The tests were made on the vital and nice-looking seeds of *Cannabis sativa* L. (hemp), *Sinapis alba* L. (white mustard), and *Hordeum vulgare* L. (barley grass). The tests are based on the principle of seed cultivation with the use of contaminated material and on the monitoring of inhibition or stimulation of the root growth in germinating plants. Root lengths are compared with the control sample whose seeds are cultivated on the reference substrate (the composition of certified OECD soil: 74% sand, 20% kaolinite, 5% peat and 1% $CaCO_3$). OECD soil serves as a substrate in toxicity tests and its use as a medium is recommended for various ecotoxicological tests [18]. Tests performed by authors [19–22] indicate that thanks to its good germinating capacity, sensitivity to disturbances and rapid symptoms of response to stress induced by the toxic material, *Sinapis alba* L. is greatly recommended for such tests. Another plant species used in the tests for its phytoremediation potential was *Cannabis sativa* L. *Hordeum vulgare* L. was used to compare the test results. A number of tests were carried out with varied shares of the tested material (from sampling points 1–4). Samples were prepared with the use of certified OECD soil (reference substrate) with the shares of mining waste 25%, 50%, 75%, 90% and 100% (only the contaminated sample was without the OECD soil addition). The control sample consisted of 100% OECD soil without the addition of the tested material. One sample represented a volume of 90 mL of tested material in the given content. The tests were at all times performed in triplicates (I–III). The chemical properties of each sample (1–4), such as pH, were measured using the HACH TEST KIT.

The sample amount of 90 mL was placed and evenly spread over the lower part of the phytotoxkit/sampler, covered with filter paper and moistened with distilled water according to instructions. The amount of distilled water added to the samples was determined based on the calculation of water holding capacity (WHC) according to instructions [17]. Seeds of the chosen plant were inserted in one plane and at the same distance from one another 1 cm from the upper edge of the filter paper. The seeds were placed so that the growth of their roots in the direction of gravity was not limited. Closed samplers were properly marked with the number of sampling point, percentage of OECD soil in the sample, and the number of replicates (I–III). The prepared samplers were placed into the holder in a vertical position and transferred into the incubator. Conditions favorable for incubation were created by using the Ecocell incubator. The samples were incubated in dark at a temperature of 25 ± 1 ℃ for 72h. The course of the tests was photographically documented (Figure 2).

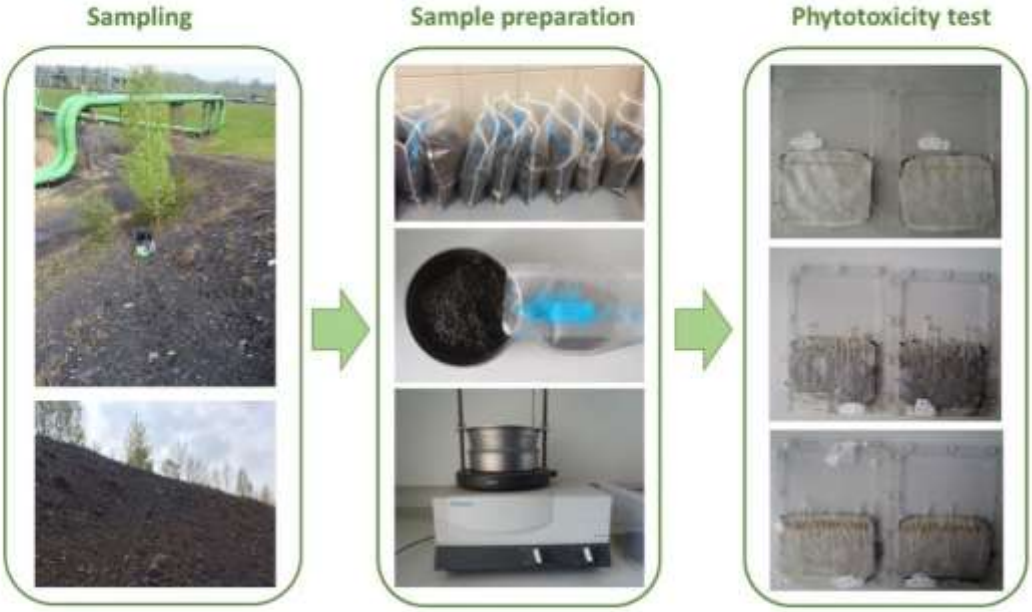

Figure 2. Tests of phytotoxicity.

The samples were removed after the incubation time had elapsed. Root lengths were measured in all samples by using Image Tool 3.0 for Windows (UTHSCSA - The University of Texas Health Science Center at San Antonio, San Antonio, U.S.A.) with accuracy to millimeters, and the values were recorded in the laboratory papers (Phytotoxkit result sheet). The principle of the test consisted of the comparison of average plant root lengths on the tested samples with the average root lengths of plants growing in the control sample (OECD soil). The recorded data were used to calculate growth inhibition (GI) of plant roots on the studied samples. The calculation was principally based on the following equation (1):

$$GI(\%) = \frac{(A-B)}{A} \times 100 \tag{1}$$

A: mean root length in the reference substrate (mm)
B: mean root length on the tested substrate (mm) [17].

3. Results and discussion

GI informs about the effect of the studied sample on higher plants. If GI > 0, it is an inhibition, i.e., the sample in question inhibits the growth of plants more than the control soil. The higher GI, the more toxic for plants the sample is. If GI < 0, stimulation and the given sample promotes the growth of plants more than the control soil. Results in negative numbers express the growth stimulation rate [20]. If the inhibition (I) is lower than 10% (I < 10%), the given sample is non-toxic or slightly toxic; in the case of 10% < I < 50%, the sample is toxic and if I > 50%, the effect of the sample is strongly toxic [22]. The pH values of soil can be divided into five degrees, strong acidic pH < 5.0, acid pH 5.0–6.5, neutral pH 6.5–7.5, alkaline pH 7.5–8.5, strong alkaline pH > 8.5 [23]. The results of the pH values of samples 1–4 are shown in Table 1.

Table 1. The pH values of the samples 1–4.

Sampling point	1	2	3	4
pH	8.31 ± 0.1	7.00 ± 0.1	7.70 ± 0.1	8.67 ± 0.1

The pH values of tested samples ranged from 7.00 to 8.67. The pH value of sample 2 was neutral, the pH value of samples 1 and 3 was alkaline. The highest value of pH was in sample 4, i.e., pH was strong alkaline.

The highest inhibition of root growth (71.55%) was demonstrated in *Cannabis sativa* L. during the testing of sample 4 (Sampling point 4) with the share of mining material 75%. Depending on the mining material share, the growth inhibition (Sample 4) ranged from −10.78% to 71.55%. All samples (Samples 1–4) in the share of 90% caused growth inhibition. Contrariwise, samples without the addition of OECD soil caused growth stimulation. The lowest growth inhibition, i.e., growth stimulation was recorded in Sample 1 (Sampling point 1) (−34.26%), also in the share of 75%. The growth inhibition of *Cannabis sativa* L. in Sample 1 ranged from −34.26% to 28.30%. Sample 2 (Sampling point 2) exhibited growth inhibition from −19.88% to 24.76%. In Sample 3 (Sampling point 3) of *Cannabis sativa* L., the growth inhibition ranged from −13.24% to 29.95% (Figure 3).

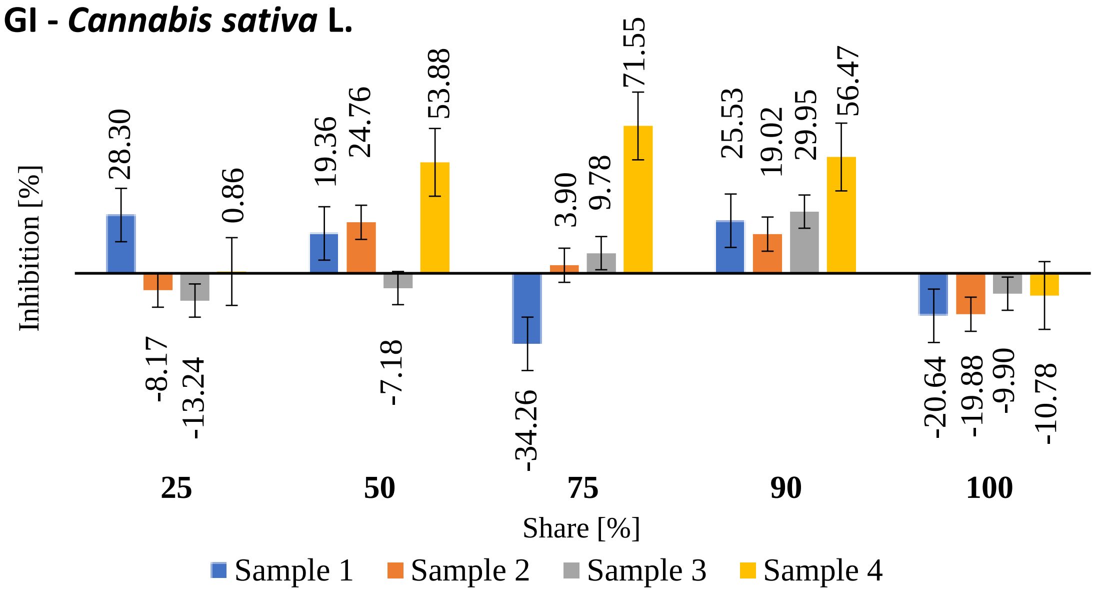

Figure 3. Growth inhibition of *Cannabis sativa* L.

The growth of roots in *Hordeum vulgare* L. was stimulated by Sample 1 in all shares except share of 90%. The inhibition was demonstrated to range from −35.86% to 5.16%. Sample 2 (except for the share of 90%, in which the inhibition was 12.53%) stimulated the growth of roots by 3.59–22.71% as compared with the reference sample. Inhibition results in Sample 3 ranged from −1.42% to 20.69%. Sample 4 without the addition of OECD soil stimulated the growth (inhibition value was −21.16%) but caused inhibition ranging from 1.89% to 23.18% in all other shares. Results of growth inhibition in *Hordeum vulgare* L. are shown in Figure 4.

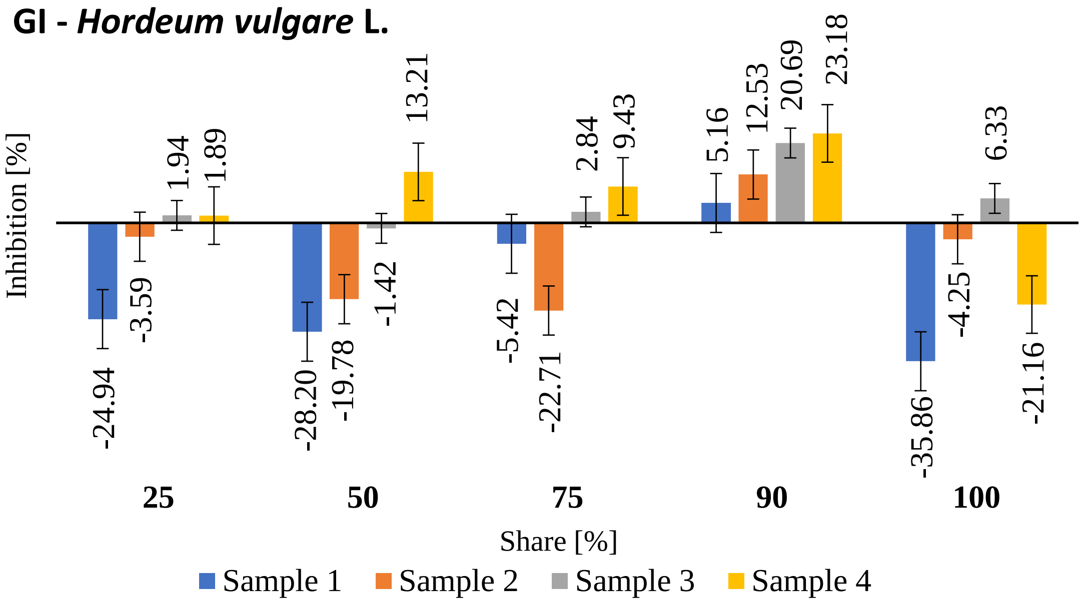

Figure 4. Growth inhibition of *Hordeum vulgare* L.

Figure 5 presents the results of root growth inhibition in *Sinapis alba* L. where the only case of growth inhibition was a 100% share of Sample 1 (14.61%). All other shares of Samples 1–4 demonstrably stimulated the growth of *Sinapis alba* L. Sample 1 stimulated the growth by 0.66%−30.48% (with the exception of the 100% share see above). In Sample 2, the growth stimulation was recorded from 26.63% to 43.59%. The highest value of growth stimulation in *Sinapis alba* L. was in Sample 3 with the share of 25% (total range from 51.76% to 79.15%). Sample 4 exhibited relatively high growth stimulation too. Sample 4 inhibited the growth from −38.26% to −69.25%.

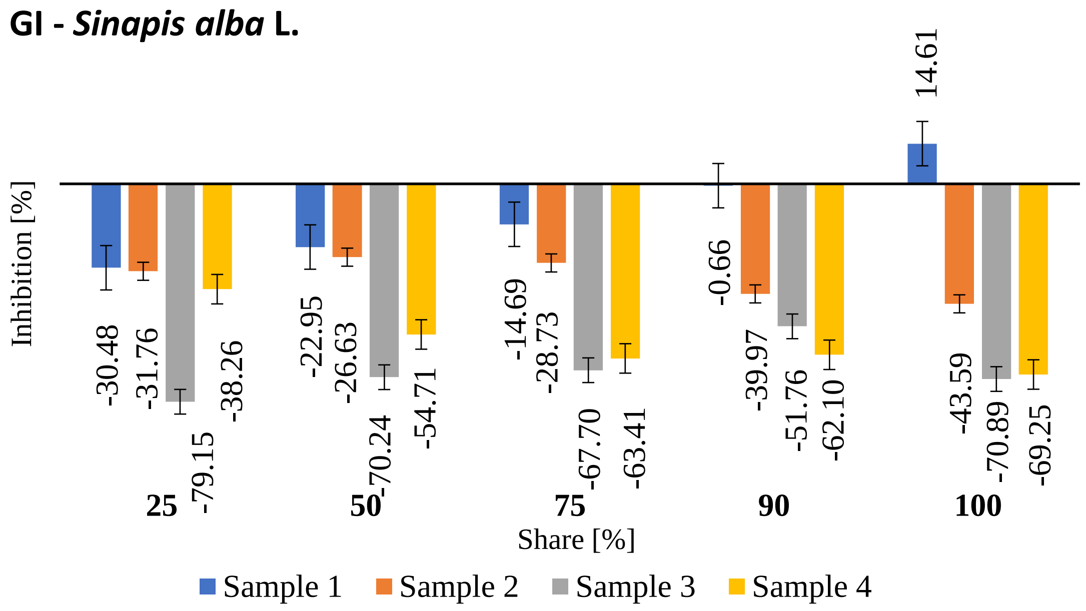

Figure 5. Growth inhibition of *Sinapis alba* L.

In real conditions, the tested samples usually occur in 100%, i.e., without the addition of OECD soil or other additives. This is why Figure 6 shows results of phytotoxicity tests for Samples 1–4 (Sampling points 1–4) in the share of 100% (without the addition of OECD soil). Growth inhibition was recorded in Sample 1 by testing *Sinapis alba* L. (14.61%) and in Sample 3 by testing *Hordeum vulgare* L. (6.33%). In all other cases for *Sinapis alba* L., *Hordeum vulgare* L. and *Cannabis sativa* L. the growth of germinating plants was promoted from 4.25% to 70.89%. For the overall evaluation of the phytotoxicity of samples with the share of mining waste 100%, a median was created from all values of growth inhibition. Resulting values of plant growth inhibition after this adjustment were −13.96% for Sample 1, −22.57% for Sample 2, −24.82% for Sample 3 and −33.73% for Sample 4.

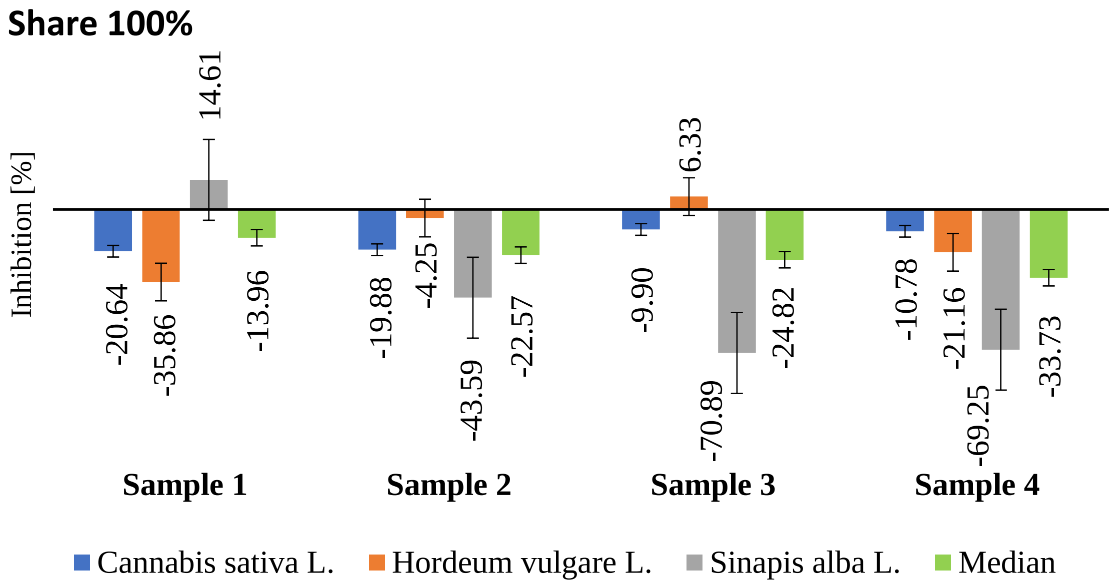

Figure 6. Growth inhibition–comparison of the share of 100%.

There are currently numerous products for fertilization and nutrition of plants on the market including materials which can replace humus and are innovative and environmentally friendly. They are produced from mining waste. In their study, Kugiel and Piekło [5] describe the Polish company Haldex s.a. dealing with the extraction of material for energy use. At the same time, the authors inform that the company also deals with the zero-waste processing of mining waste including the innovative manufacture of commercial products. One of them is a product replacing properties of humus which promotes the development of plants. Phytotoxicity was not demonstrated in the product which among other things contains waste from coal extraction. By contrast, it was shown that it favorably affects soil characteristics.

Another possibility of mining waste management is the repeated use of these materials for remediation of landfills other than hazardous [3]. However, the materials used for the remediation of landfills should not have toxic properties. Czop et al. [3] performed a number of tests on the mining waste from Silesia with using monocotyledonous (*Triticum*) and dicotyledonous (*Lepidium* and *Sinapis*) plants, demonstrating the positive influence of mining waste on the growth of roots in the monocotyledonous plants. In the dicotyledonous plants, no significant inhibition of mining waste was demonstrated as compared with other materials. In testing the mining waste phytotoxicity, it was observed the correct development of plant seeds (equal growth of above-ground and under-ground plant parts). The performed tests were concluded by stating that the tested waste not only did not hinder the development of the plants but promoted it and accelerated their growth. Mining waste does not exhibit toxic characteristics and can be used in the process of reclamation [3].

In their research, Huang et al. [2] assessed the regeneration of plants growing on heaps from coal mines from a long-term point of view as a method for enhanced regeneration of ecosystem damaged by mining activities. They did not demonstrate any negative influence of mining waste on the succession of plants, although such a method of regeneration is rather demanding in terms of time.

An example of good practice can be the environment-friendly application of coal ash into the soil with the expectation of its improved quality, increased productivity of crops and minimization of environmental risks connected with inappropriate disposal of this material [24–26]. The influence of adding ash from thermal power plants (use of brown coal) and potassium humate on the phytotoxicity of materials from extractive industries (rock dumps substrate of coalmines) was studied by Shpak et al. [27]. The authors demonstrated a positive influence of ash dumps on the growth of stem length, total leaf area and both density and length of roots in *Sorghum bicolor* L. plants. Korkov et al. [28] dealt with the development and survival of natural and artificial plant community on heaps of coal gangue from closed coal mines in connection with the use of phytoremediation. The vegetation was composed of plant species from the following families: Poaceae, Fabaceae, Brassicaceae, Euphorbiaceae and Asteraceae. In their study, Tomaszewska-Krojańska and Pranagal [1] mention that thanks to its positive characteristics, coal gangue can be used as a sorbent and added to the soil in order to enhance its properties.

Many authors demonstrated in their publications the positive influence of mining waste/material on the plants and admitted a possibility of its use for application e.g., into the soil. Results of this research showed that mining waste/material does not exhibit phytotoxic effects, thus confirming the conclusions of other authors. Therefore, mining waste should not be a problem in the future and could be managed as a raw material with numerous applications (reclamation of degraded sites, soil additive etc.).

4. Conclusion

The currently continuing high need of energies and demand for coal stimulate corporations to mining activities. This is associated with the generation of huge amounts of mining waste which is not primarily used for energy purposes but stored in the vicinity of coal mines. Dumps of such materials alter the landscape appearance and are subject of many questions concerning their harmfulness for the environment. In this study, tests of the phytotoxicity of mining waste were performed at diverse contents of mining material with the addition of OECD soil. Although the growth of plant roots was inhibited in some cases, the results of inhibition were in low values. An exception was Sample 4 with the shares of 50%, 75% and 90%, in which higher inhibition was recorded. Taking into account the phytotoxicity of mining wastes without the addition of OECD soil (100% share of mining waste) and the research results of other authors, it can be stated that the researched material was not phytotoxic. Results of laboratory tests performed by us demonstrated that conditions for the growth of plants can be improved by the application of waste material from coal mining. The material could be used for the reclamation of degraded sites as well as of localities which need to improve soil characteristics and conditions for the growth of vegetation.

Conflict of interest

All authors declare no conflicts of interest in this paper.

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