

# Streamlining of the Reclamation Substrate in Practice

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## Summary

*The article deals with the innovation of substrate for land reclamation purposes made by the company OBSED a. s. It proposes a new and more economical composition of the reclamation substrate. It brings the results of the ecotoxicity tests applied on the pre-determined ratios of the individual components of the substrate. The ecotoxicity tests made used of white mustard (*Sinapis alba*) seeded in this reclamation substrate and its modifications. The article describes the manufacturing process of the original subsurface substrate for reclamation purposes and the carried out ecotoxicity tests on various quantitative proportions of the materials contained in the substrate. The aspect of innovation is grounded in altering the ratios of the input raw materials, including testing ecotoxicity of the new and more economical reclamation substrate, which still offers identical technical parameters as the previous reclamation substrate.*

*Keywords: reclamation substrate, sludge, fly ash, ecotoxicity test*

## Introduction

Anthropogenic actions have a number of drawbacks which need to be dealt with in a fast and effective manner. There are still many old ecological burdens to be remediated and reclaimed. However, the choice of the reclamation method depends on the locality in question. In Europe intense reclamation is applied especially in Germany, Great Britain as well as in the Czech Republic and Poland, where forestry reclamation prevails due to a shortage of overburden containing suitable soils or topsoil. On the American continents intense reclamation takes place mainly in the USA and Canada. [15]. The process of regeneration or reclamation is always modelled for a specific affected locality. There is a whole range of materials that can be applied within the technical reclamation. Among the conventional reclamation mixtures there is, for example, compost or soil. Less routine materials are the mixtures of various raw materials. [18, 19, 20] The trends in the application of reclamation substrates in the Czech Republic are analogous worldwide. Blended and treated reclamation mixtures have much better technical parameters than single-component substrates. The company of OBSED a. s. from Ostrava makes subsurface substrate for reclamation purposes from a mixture of fly ash and sludge, which is certified as a product. This type of reclamation substrate has numerous advantages as opposed to conventional substrates used in land reclamation. One of the technically favourable properties is less troublesome weathering of the soil, which is a desirable quality especially in hilly grounds.

Naturally, reclamation substrates must be environmentally sound and, thus, it is subject to very strict laboratory tests on a monthly basis. The laboratory tests of the substrate, containing fly ash and sludge in 3:1 proportion, have very good results. The sludge used for the substrate production is stored in a pre-determined and safe area, which is outside residential areas. The sludge may smell bad and could inconvenience the inhabitants. However, this problem could be eliminated thanks to the patented methods from Poland, which are grounded in the application of ethereal oils instead of flocculants used in sludge dewatering [16, 17].

The major question of this article is whether changing the proportion of the raw materials could have a decisive effect on the overall ecotoxicity of the substrate. The aim is to present changes in the proportions of fly ash and sludge without affecting the substrate quality; and to carry out tests of ecotoxicity by means of white mustard (*Sinapis alba*) which may prove that such a selected proportion is not ecotoxic. If the test results are favourable, the company OBSED a. s. could benefit from significant economic savings.

## Materials - Subsurface substrate for reclamation purposes

All the information concerning the reclamation subsurface substrate is regulated by the company standard [1]. The owner of the in-house document as well as of the product certification for the reclamation subsurface substrate is the company OBSED a. s. located in Moravská Ostrava, Czech Republic [2].

The product, i.e. the reclamation subsurface substrate, is made of three portions of fly ash and one portion of raw sludge treated with lime. In accordance with the Catalogue of Wastes in Regulation 381/2001 Coll. [3], fly ash is classified under No. 10 01 02. In this case, the fly ash is a certified product of Energetika Vítkovice. It is stored outside the heating plant premises in the form of a hydromixture which is subsequently dewatered [14].

Sewage sludge is classified under No. 19 08 05 (sludge from municipal wastewater treatment) [3] and comes from the wastewater plant in Frýdek-Místek.

The reclamation subsurface substrate (further referred to as the substrate) is used as a cover for the technical fillings in reclamation work. The maximum thickness of the applied substrate layer is 2.5 meters and is combined with the surface layer substrate, which is mixed from soil and sludge in 1:1 proportion. The combination of the two substrates is followed by biological reclamation [2].

In line with Regulation 474/2000 Coll., on determining the requirements for fertilisers, according to Appendix 1 [4], the substrate must comply with the limits stated in Table 1 as well as certain prescribed technical parameters included in Table 2.

The production technology of the substrate is grounded in mixing sludge and fly ash in a predetermined site, to where the raw materials are transported by a wheel loader. The materials are spread into two parallel strips in order to make one symmetrical strip, which is maximally 6 metres in width and 2.5 me-

tres in length. Next, the material is homogenised by a bridge-type shoveller. This process is repeated until complete homogenisation of the substrate is reached [5]. Figure 1 displays the diagram of the substrate production.

Having made a homogenous mixture, the substrate is left to condition for the minimum of 90 days. The conditioning ensures carbonation of the particle surface, a low-soluble carbonate coat forms and the pH value falls. The substrate is turned over on a regular basis in order to ensure the right course of the process. The substrate quality is checked each month by means of strict laboratory tests. After 90 days, at least, a thorough laboratory analysis of the substrate is carried out, and if the substrate manifests applicable quality features, it may be used in land reclamation [5].

### Methods

Samples designed for the test of ecotoxicity were drawn from the storing site of the material to produce the reclamation subsurface substrate. The sampling method fully corresponded with ČSN 015110 Standard - Material sampling, Basic Provisions [6] and the Guideline of the Ministry of the Environment of the Czech Republic – Sampling of waste [7].

The individual samples of raw sludge, fly ash and final substrate were drawn into black plastic bags. Immediately after sampling, the samples were transported into the laboratory. In the laboratory, the raw materials (sludge, fly ash) were mixed based on

Tab. 1 Limit values of hazardous elements in the substrate [4]

Tab. 1 Wartości graniczne zawartości pierwiastków niebezpiecznych w podłożu [4]

Hazardous element	Symbol	Value [mg/kg of DM]
cadmium	Cd	max. 2
lead	Pb	max. 100
mercury	Hg	max. 1
arsenic	As	max. 10
chromium	Cr	max. 100
copper	Cu	max. 100
molybdenum	Mo	max. 5
nickel	Ni	max. 30
zinc	Zn	max. 300

Tab. 2 Technical requirements for the reclamation subsurface substrate [2].

Tab. 2 Wymagania techniczne dla regeneracji podpowierzchniowej podłożu [2]

Technical parameters	Value	Unit
moisture	max. 50	%
combustibles in a dried sample	min. 5.0	%
pH value	6.5 - 9	
content of particles over 31.5 mm	max. 10	%
conductivity	max. 1.3	mS/cm

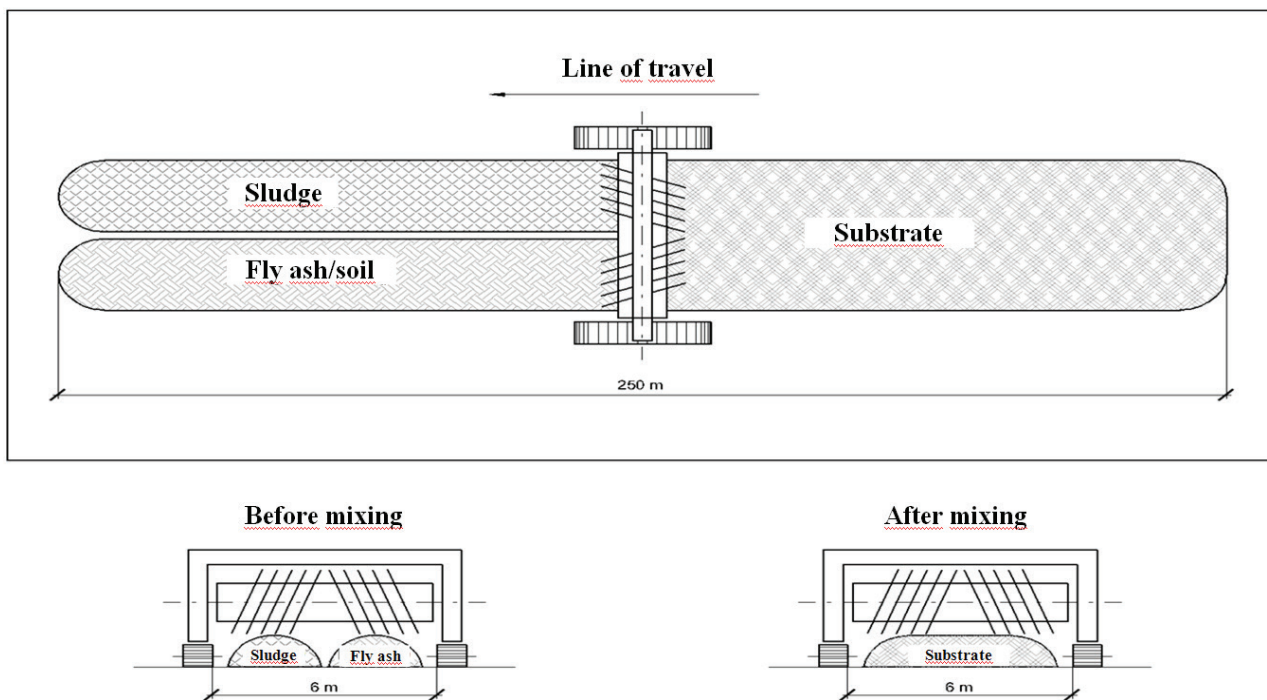


Fig. 1 Diagram of the reclamation substrate production [5].

Rys. 1. Schemat produkcji podłoża regenerowanego [5]

Tab. 3 Sample designation + volume ratios

Tab. 3 Oznaczenie próbek - wskaźniki objętościowe

Sample designation	Volume ratio
	sludge : fly ash
I	1:1
II	1:2
III	2:1
IV	3:1
V	1:3

pre-set quantitative (volume) ratios, labelled as I, II, III, and IV, stated in Table 3. Five kilograms of each sample was prepared. The sample labelled as V is the certified final product of the company OBSED a. s. The first round of ecotoxicity tests was carried out immediately, without the 90-day conditionEng. This method without conditioning was selected as the pilot one, in order to rough out the most suitable material proportions in the substrate. Halves of each newly mixed substrates (2.5 kg) were exposed to the natural conditions for 90 days, i.e. conditioning with regular turn-overs.

The samples I, II, III, IV and the final product labelled as V were prepared for the first round of the ecotoxicity tests using white mustard (*Sinapis alba*). First, aqueous solution was prepared according to the guideline of the Waste Department of the Ministry of the Environment of the Czech Republic (MECR) in

order to evaluate leaching of the waste [8], and ČSN EN 12457-4 Standard – “Characterisation of waste - Leaching – Compliance test for leaching granular waste materials and sludge – Part 4: One stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 10 mm (with/without particle size reduction)” [9].

In the next stage of the test, we prepared stock solutions according to the guideline of the Waste Department to determine ecotoxicity, Chapter 3.3.1 [10]. The course of the test was divided into the individual stages subject to the MECR Guideline [10], displayed in Table 4. The test was analogous to the flow-chart which makes part of the MECR Guideline in Chapter 3.4 “Methodology of ecotoxicity tests” [10].

The ecotoxicity tests characterise the course of action of the testing organism in a pre-set concentration of aqueous solution during a certain time period. The

Tab. 4 Stages of the ecotoxicity test and a short description [2,10]

Tab. 4 Etapy badania ekotoksyczności i krótki opis [2,10]

Ecotoxicity test stages	Test character
OPENING TEST	The tested solution is prepared from neat aqueous leachate of the waste.
PRELIMINARY TEST	Selection of a suitable range of aqueous solution concentrations.
BASIC TEST	Determination of a suitable concentration series based on the results of the preliminary test.
VERIFICATION TEST	If the toxic effect is $\leq 50\%$ in the opening test and the value of LC 50 (EC, IC) cannot be determined based on the preliminary test, a verification test is carried out, with at least triple amount of organisms as opposed to the basic tests.

core of the test is to observe the organism's reaction to the tested substance. The organism may react to the tested substance in the form of death, inhibition, stimulation, physiological processes or growth. The test is accompanied by a check, i.e. a blank sample without the tested substance [2].

#### Ecotoxicity test using white mustard – *Sinapis alba*

The ecotoxicity test using the organism of white mustard (*Sinapis alba*) is based on growing the plant for 72 hours in a cabinet-type incubator at the temperature of 20°C. Thirty mustard seeds are geometrically arranged on a filter paper into Petri dishes. Concurrently, the seeds are fed by the tested-substance aqueous solutions of certain concentration and enriched with stock solutions. In the set Petri dish, 10 ml of aqueous solution are added. After 72 hours we measured the individual lengths of the roots using a slide gauge and they were contrasted with the check. If a seed did not germinate or did not form a root, a zero value was recorded for the seed. The measured values of mustard root length are expressed as a mean root length of the check ( $L_{\checkmark}$ ), the mean root length of the sample ( $L_{\emptyset}$ ) and inhibition/stimulation of the root growth. If necessary, the value of 72 IC 50 is determined using the Probit Method [2, 10, 11].

The test was carried out with certified seed material of over 90% germinating capacity, category C1 CERTIFIED SEEDS, lot number 0-0289-71526/02, according to the Central Institute for Supervising and Testing in Agriculture – department of seeds and plantEng. The supplier of the certified seeds is the company MORSEVA spol. s. r. o. Olomouc [2].

##### a) Ecotoxicity test without 90-day conditioning

The opening test was executed with the samples I, II, III, IV and V, the results of which are in Chart 1.

Only in samples II and V, growth inhibition below 50 % was proved, which means that only these two samples are suitable. As sample V is the final product,

the only prospective substrate is sample II as a promising substitute for the original substrate. It contains only two portions of fly ash, instead of the original three portions, which would be an economically promising option. A Verification Test was carried out in both the samples- see Chart 2.

The Verification Test confirmed the results of the previous one. The result variances were below 30%, and, thus, the test is considered valid. The values of the tested standard also demonstrated favourable values. Therefore, sample II was exposed to natural conditions for 90 days and, subsequently, the ecotoxicity test was repeated.

##### b) Ecotoxicity test after 90-day conditioning of the sample

The Opening Test was executed on the sample II which underwent 90-day conditionEng. Charts 2 and 3 plot the results of the Opening and Verification Tests.

The Verification Test confirmed the validity of the Opening Test. The test carried out with the standard also showed permissible values.

#### Summary of the major research results

The first ecotoxicity tests were carried out with all the samples using white mustard (*Sinapis alba*) without the 90-day conditionEng. Next, with regard to the test results, the ecotoxicity test was repeated with sample II after 90 days dedicated to sufficient conditionEng.

The first set of tests was executed with samples I, II, III, IV and V without the 90-day conditioning, except for the “final product”, i.e. sample V. Only samples II and V proved growth inhibition below 30%, or below 50%, and thus it was not possible to determine the value 72 IC 50, stipulated by Regulation 376/2001 Col [12]. As the samples II and V do not exceed the limit set by the Regulation mentioned above, the samples do not manifest H14 – ecotoxic-

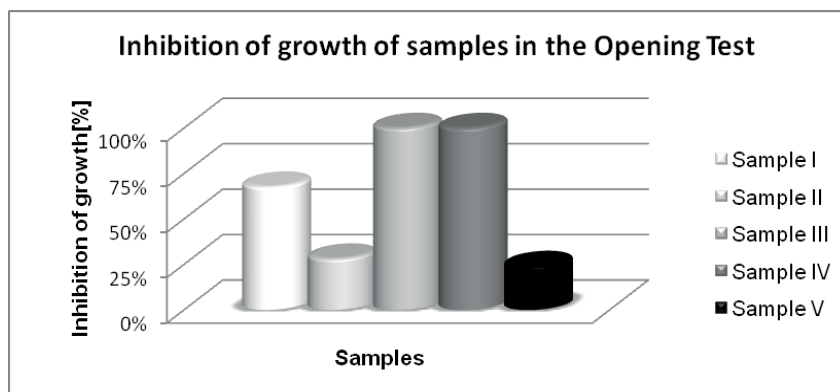


Chart 1 Inhibition of growth of samples I-V in the Opening Test

Wyk. 1 Hamowanie wzrostu próbek I - V w teście startowym

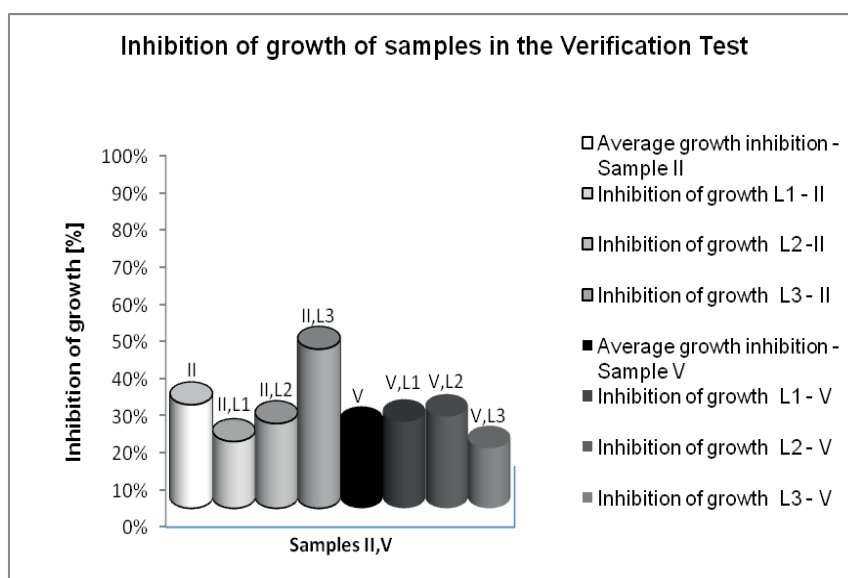


Chart 2 Inhibition of growth of sample II and V in the Verification Test

Wyk. 2 Hamowanie wzrostu próbki II i V w teście weryfikacji

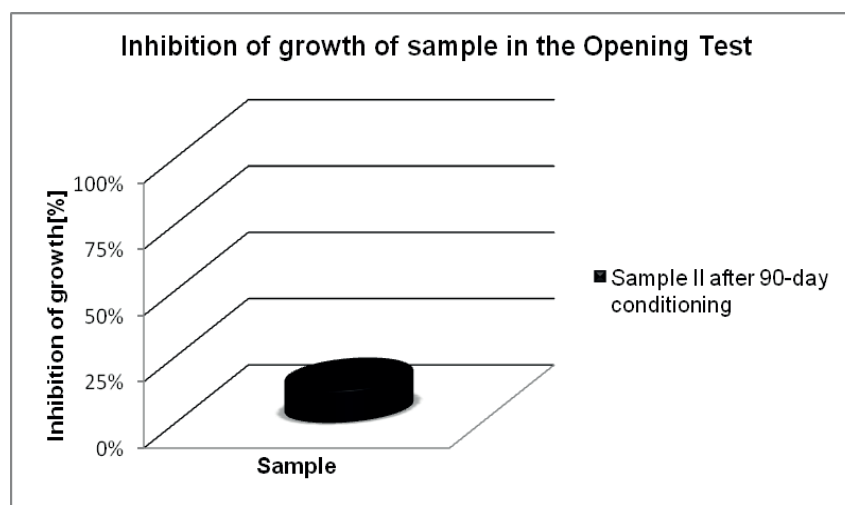


Chart 3 Inhibition of growth of sample II after 90-day conditioning in the Opening Test

Wyk. 3 Hamowanie wzrostu próbki II po 90-dniowego klimatyzacji w próbie startowej

ity. Regulation 294/2005 Coll. determines the limits according to Appendix 10, Table 10.2, "Requirements for ecotoxicology test results" [2, 13]. Considering the positive result in the case of sample II, it was exposed to 90-day conditioning in the natural conditions.

The second part of the test followed after the 90-day conditioning of sample II. First, the Opening Test was evaluated as it showed identical values with sample V, i.e. the original substrate and product. Based on the test results, a Verification Test was executed, which confirmed the previous positive result. Chart 5 plots the results of both the Opening and the Verification Tests.

For better visualisation, Chart 6 compares the values from the Opening Test before and after the 90-day conditionEng.

Based on Chart 6 there is a clear difference between the result before and after the 90-day condi-

tionEng. Sample II complied with the required values set for substitution of the original substrate already before conditionEng. Still, the process of conditioning had a significantly positive effect and proved its importance in the production of the reclamation subsurface substrate.

### Conclusion

The major aim of the test, discussed herein, was to innovate the original subsurface substrate for reclamation purposes and to obtain a more economical substrate which would still have identical technical parameters. The innovation is grounded in altering the original ratios of the raw materials. Applying an ecotoxicity test we experimentally excluded conditions which would not be suitable for the purposes in question. Only sample II, which contained 2:1 proportion of fly ash and sludge, proved to be applicable. As the

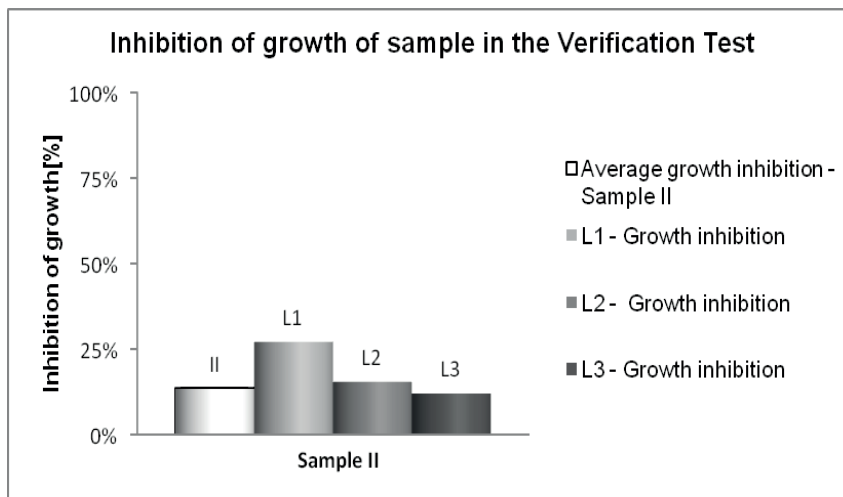


Chart 4 Inhibition of growth of sample II after 90-day conditioning in the Verification Test

Wyk. 4 Hamowanie wzrostu po kondycjonowaniu próbki II po 90-dniowym teście weryfikującym

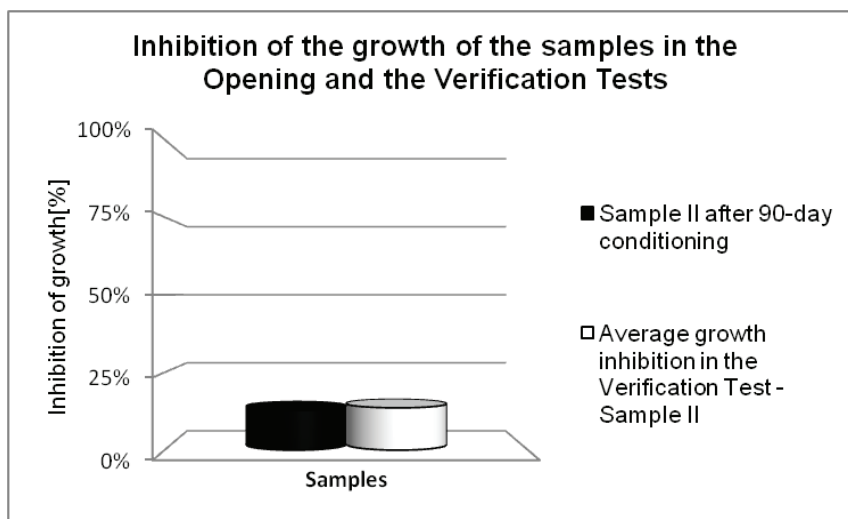


Chart 5 Inhibition of the growth of the samples in the Opening and the Verification Tests

Wyk. 5 Zahamowanie wzrostu próbek w testach otwarcia i testach weryfikacyjnych

only one among the prepared samples in line with Regulation 376/2001 Coll. [12], it did not exceed the limit stated in the Regulation and, thus, the samples do not manifest H14 – ecotoxicity. Next, according to Regulation 294/2005 Coll. [13] the substrate may be applied within the ground surface work as it does not exceed the growth inhibition/stimulation of 30% [2, 12]. After the 90-day conditioning the ecotoxicity test was repeated. In the Opening Test, sample II had much lower values than before the 90-day conditioning. The values were even lower than in the

original reclamation substrate. The test pointed at the suitability of the innovated ratios in the production of reclamation subsurface substrate as well as the importance of its conditioning. The results were confirmed during the Verification Test.

The test results imply that only sample II with 2:1 proportion of fly ash and sludge and after the 90-day conditioning is a suitable alternative to the original substrate in the 3:1 proportion of fly ash and sludge.

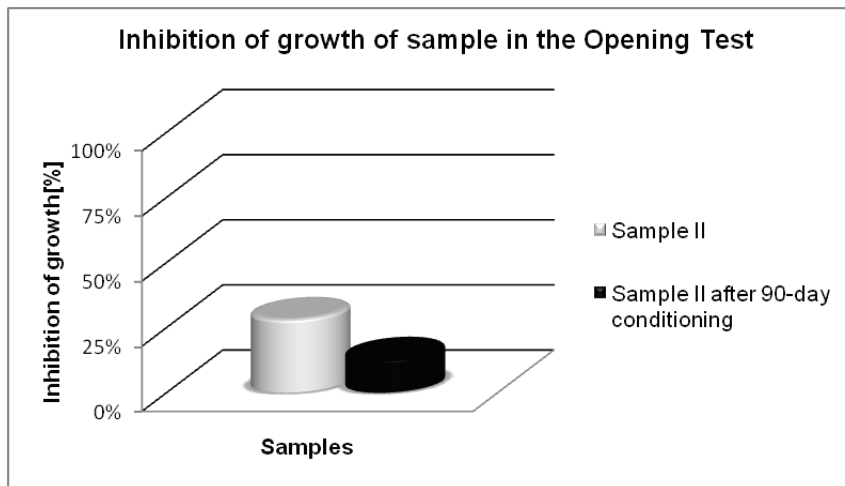


Chart 6 Inhibition of growth of sample II in the Opening Test

Wyk. 6 Zahamowanie wzrostu próbki II w teście otwarcia

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### Streszczenie

W artykule opisano innowacyjne podłoże do celów melioracyjnych wykonanych przez firmę OBSED s.r. ekotoksyczności dla zaproponowanych różnych receptur podłoża. Testy ekotoksyczności wykonano używając nasion gorczycy białej (*Sinapis alba*) zasianych na podłożu do rekultywacji oraz jego modyfikacjach. W artykule przedstawiono proces produkcji oryginalnego podłoża w celach rekultywacji podpowierzchniowej i przeprowadzonych badań ekotoksyczności, w różnych proporcjach ilościowych materiałów zawartych w podłożu. Aspekt innowacji zawiera się w zmiennych proporcjach surowców wejściowych, oraz w badaniu ekotoksyczności nowego i bardziej ekonomicznego podłoża do rekultywacji, który charakteryzuje się takimi samymi parametrami technicznymi jak stosowane podłoża do rekultywacji.

Słowa kluczowe: podłoże do rekultywacji, osady, popioły lotne, badanie ekotoksyczności