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Assessment of the environmental condition in the area of the closed landfills of Krakowskie Zakłady Sodowe Solvay

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ABSTRACT

The soda waste sediment ponds of Krakowskie Zakłady Sodowe Solvay are located in the southern part of Krakow, within the city. The complex of sedimentation ponds consists of three parts, where approximately 5 million Mg of waste was deposited. Between the landfills flows the Wilga River. During field research, water samples were taken from river before it entered the sediments area and afterwards. Surface layer soil samples were also taken from sediment ponds that were not fully reclaimed, including the places where the ground cover was devastated. Physicochemical tests of collected water and soil samples (including tests of water extracts from soil) as well as biological tests were performed to assess phytotoxicity. The concentration of ions was investigated using the photometric method; results indicate that the chloride concentration in water of river after flowing through sediment ponds is 5.4 times higher. The concertation of sulfate is also higher, by about 18%. The analysis of the phytotoxic properties of the sediments included testing the phytotoxicity of the water extract from the sediments against *Lepidium sativum*. They were also not found to be phytotoxic to the test plants, and differences in the early growth of the test plants were statistically insignificant, although a slight inhibition was noted in the test sites compared to the control.

Keywords: retardation, soda waste, sediment ponds, ecotoxicity, recultivation.

INTRODUCTION

One of the main problems related to industrial activities are large amounts of waste generated, often harmful to the environment due to their properties, which depend on the raw materials used, the profile of the plant and the technology used. Currently, in line with the assumptions of the circular economy, the solutions that are aimed at reducing the amount of waste generated will be introduced in the industry. If technological possibilities allow, old waste landfills are exploited, e.g. tailings from metal ore mining. The technology for reprocessing and using old post-production waste collected in sedimentation ponds and heaps is still developing, enabling the operation of old landfills in an economically profitable manner. These are undoubtedly beneficial actions for the environment, but still not always possible. Old landfills may pose risk to the environment, especially to subsoil and water [Rozporządzenie Ministra Zdrowia, 2017, Rozporządzenie Ministra Środowiska, 2011]. These landfills also interfere with the natural environment. The impact time depends on the type of deposited waste, which is specified in landfills (e.g. leaching, immobilization, solidification) and which may lead to a reduction in the negative impact [Taha et al., 2024]. Old, unreclaimed landfills are often transformed into different ones in terms of nature, where valuable ecosystems can be developed. An example is the orchid landfill in the Ząbkowice district, where the waste from the extraction of gold and arsenic ore was collected. In 2003, in the area of the landfill issued by the Voivode of Lower Silesia, established ecologically in order to preserve the remains and rare species of plants and animals, in this specific place, the male orchid *Orchis mascula* [Rozporządzenie: Wojewody Dolnośląskiego, 2003]. Post-industrial waste landfills are often reclaimed for recreational use. An example of an area is Góra Kamieńsk, where recreational and sports facilities are located on the landfill of the "Bełchatów" Brown Coal Mine [Rawlik et al., 2019].

Soda production is one of the most important processes in the chemical industry. In Kraków, a soda factory was founded in 1906. The Solvay method was applied to produce soda ash (sodium carbonate, Na_2CO_3). During the manufacturing process, wastes were created, which were deposited in sedimentation settlers. These wastes had non-homogeneous solid or liquid form: distillation wastewater (with dissolved $CaCl_2$, $MgCl_2$, NaCl) and a huge amount of suspended particles of various chemical compound [Gołub et al., 2018, Steinhauser, 2008].

The areas of the closed soda waste sediment ponds of Krakowskie Zakłady Sodowe Solvay, located in the southern part of Krakow, within the city, have been partially reclaimed. Due to the type of waste collected there and the high ion leachability of chloride and sulfate ions, they posed a significant threat to the ground and water [Bloor et al., 2006, Kjeldsen et al., 2001]. Currently, 35 years after the end of waste storage, sedimentation settlers no longer pose such a great threat. The waste was washed by rainwater and the load of washed pollutants decreased significantly [Gliniak et al., 2012].

RESEARCH AREA

In 1906, Zakłady Sodowe Solvay started operating in Krakow. The location of the plants was determined by the availability of raw materials: limestone from the nearby Zakrzówek and Liban quarries, brine from the Wieliczka and Barycz mines, and water from the nearby Wilga River [Stachowski et al., 2000]. The commercial products of the plant included: soda ash (Na_2CO_3), caustic soda (NaOH), baking soda, salt ammoniac (NH_4Cl) and calcium carbonate ($CaCO_3$) [Szczepka, 2023].

Due to the large amounts of post-production waste generated at the same time (6000 m^3/day, approximately 9–10 m^3 for each tons of product), sediment ponds were built, in which post-production sludge was deposited. To build the embankments, among others, furnace ashes, slag, unburned limestone and clay (soil material) from nearby construction sites were used. The crown of the embankments reaches 20–30 m above the level of the Wilga River. The following were deposited in the sediment ponds: calcium carbonate ($CaCO_3$), calcium chloride ($CaCl_2$) and other compounds: SiO_2, P_2O_5, $CaSO_4$, $MgSO_4$, $BaSO_4$, NaCl as well as flints and limestone granules [Gliniak et al., 2014]. Until the plant was closed in 1986, 5 million tons of environmentally harmful post-production waste were deposited in tailing ponds, covering an area of approximately 100 ha. Due to the color of the deposited waste, which often had white dust floating above it, the sediment ponds came to be called "White Seas" (Figure 1). After the plants were closed, some of the sediments were reclaimed, to a greater or lesser extent [Rywczak, 2021, Okrutniak, 2011].

Sediment pond No. 1 was reclaimed comprehensively (mechanically and biologically) and currently an important point on the pilgrimage map of

Figure 1. The sedimentation ponds before reclamation (Photo by W. Gorgolewski) [https://krakowznieba.pl/lagiewniki-borek-falecki-i-zaklady-solvay/]

Poland is located there: the JPII Center, connected by a walking footbridge to the Sanctuary of Divine Mercy in Łagiewniki. Sediment pond No. 2 was reclaimed mechanically and partially biologically. The top of the landfill was covered with a layer of clean soil, hydroseeding was used (humus-producing species: legumes and grasses), and the slopes were planted with woody plants, improving the stability of the embankments (Figure 2). The third settler was not reclaimed, it became overgrown on its own as a result of natural succession, and currently there is a dog park there, eagerly used by Krakow residents and their pets. A railway line runs along the western border of the tailing ponds (Kraków-Zakopane), and a "shortcut path" runs through them, connecting the Kurdwanów Nowy and Wola Duchacka housing estates with the "Zakopianka" shopping center [Kupiec et al., 2012, Sroczyński, 2008].

Currently, the sediment ponds are naturally green. They create a hilly, irregular landscape, and the vegetation that has invaded this area, despite the environmental conditions, has formed a dense cover, also on the top. At sediment settler No. 2, there is a site of giant horsetail, patches of pink willow-herb, spindle bushes, and rare species of birds, such as the white-breasted warbler and landrail *(Crex crex)*. Unfortunately, through succession, invasive species also entered the area, such as the ubiquitous goldenrod and small-flowered impatiens (Figure 2). The Wilga River meanders between the sediments ponds. In the past, one of the ideas for developing the area was the construction of a golf course, the investment has not been completed (in the area of sediment pond No. 2, the remains of the construction can be seen: depressions prepared for ponds and foundations of the planned buildings). Over the years, the settlers have been drained, but the area is still unstable. Construction works exposed deposited sediments, which are partially solidified. At the boundary between the native soil and the sediments, a layer with the characteristics of lean concrete was formed, and the sediments were delaminated. Due to the nature of the sediments, the so-called limestone dough, the material partially liquefies after rain and impurities migrate deep into the profile [Gliniak et al., 2016].

MATERIALS AND METHODS

In order to assess the chemical properties and ecotoxicity of the deposited sediments, environmental water and soil samples were collected from sedimentation ponds and the Wilga River. Soil samples were taken using an Egner's stick from sediment ponds No. 2 and 3.

The samples from the Wilga River were taken in two places: in the place "before" the sediment ponds and "beyond" the sediment ponds (WBST – water from Wilga River before sedimentation ponds, WAST– water from Wilga River beyond sedimentation ponds). Immediately after collecting the sample, water temperature and pH were measured. The concentration of Cl^- and SO_4^{2-} ions was tested using the spectrophotometric method in the laboratory. For this purpose, a spectrophotometer LF 300 Photometer (Slandi) was used. The device has preloaded programs for analysis of individual ions. To conduct investigations with

Figure 2. The sedimentation ponds after reclamation

this photometer, reagents from Slandi need to be purchased: they are numbered. The test instructions include what amount of reagent to add. Similar analyses were carried out on prepared water extracts of the soil. During tests of one water sample (WAST– water from Wilga river beyond sedimentation ponds) concentration of SO_4^{2-} ions was so high that the sample was diluted 10 times before measurement (Table 1).

The analysis of the phytotoxic properties of the sediments included testing the phytotoxicity of the water extract from the sediments against *Lepidium sativum*. For this purpose, a water extract from sediment was prepared (standard No. PN-97/Z-15009). On the basis of the water extract, a numerous of dilutions were prepared, respectively: 100%, 50%, 25%, 12.5% and 6.25%). Each portion of the solution had a volume of 5 ml (each in 3 repetitions). This samples were introduced into the petri dishes, and 3 dishes were also prepared as control objects, with distilled water. For species *Lepidium sativum*, 10 seeds were introduced into each dish. The dishes were covered and placed in the incubator, where they were incubated in the dark for 72 hours at 23 °C. After removal from the incubator, germination and early growth of the test plants were assessed.

To assess the phytotoxicity of sediments, the Phytotoxkit test for the solid phase was also performed. Test objects were prepared on Phytotoxkit test plates using the collected sediment samples and reference soil (control objects). The phytotoxicity of the sediments towards *Lepidium sativum, Sorghum saccharatum*, and *Sinapis alb*a was assessed. The test plants were incubated in an incubator at 23 °C in the dark.

RESULTS AND DISCUSSION

Spectrophotometric measurements indicate that the chloride concentration in water of river after flowing through sediment ponds is 5.4 times higher, sulfate concentration also increases – by 18% (Table 1). This proves that ions are still being leached out of the settlers. Also, pH is higher for the collected water samples after flowing through sediment ponds WAST pH = 7.05 (for comparison, the pH of a sample taken from the point before the Wilga River flowed into the settling area is WBST pH = 5.86). These results have shown that the river water after flowing through sediment ponds is more alkaline. Thus, it can be concluded that ions continue to be leached from the sediments and migrate into the river water [Wójcik et al., 2011, Sybilski et al., 2009]. Another question arises as to whether such an increase in ion concentrations adversely affects plant growth [Dojlido, 1987, Langmuir, 1997]. The concentrations of the tested ions in water extracts from soil samples are much lower (Table 1).

The results of phytotoxicity tests against *Lepidium sativum*, carried out on water extracts from the soil samples taken from soda sedimentation ponds, mostly did not show a negative impact of the tested material on the early growth of the test plants. In all tested solutions, the average root lengths in the water extracts from the tested soils were shorter than in the control sample (Figure 3). After performing the ANOVA analysis and Tukey's post hoc tests, it was found that only in the sample taken from point No. 2 (the soil from not reclaimed sedimentation pond, higher level, but from an excavation for a pond, sediments on surface layer), at a concentration of 25%, the root length differed significantly from the root length in the control sample. For none of the tested points, there were no statistically significant differences in root length between individual concentrations. No such differences were found between water extracts with a concentration of 100% from all soil samples (Figure 4).

Table 1. The results of determination of ion concentrations

Parameter	WBST	WAST	1	2	3	4
c_{Cl-} [mg/l]	39.4	214ˣ	br	18.4	19.8	br
c_{SO42-} [mg/l]	108	127	br	24	53	br

Note: br – below range, ˣ – before measurement, the sample was diluted ten times. WBST – water from Wilga River before sedimentation ponds, WAST– water from Wilga River beyond sedimentation ponds. 1 – soil from unreclaimed sedimentation pond, higher level; 2 – soil from unreclaimed sedimentation pond, higher level, but from an excavation for a pond, sediments on surface layer; 3 – soil from unreclaimed sedimentation pond, lower level, just below the surface, mixed with the ground; 4 – soil from unreclaimed sedimentation pond, behind Podmokła street.

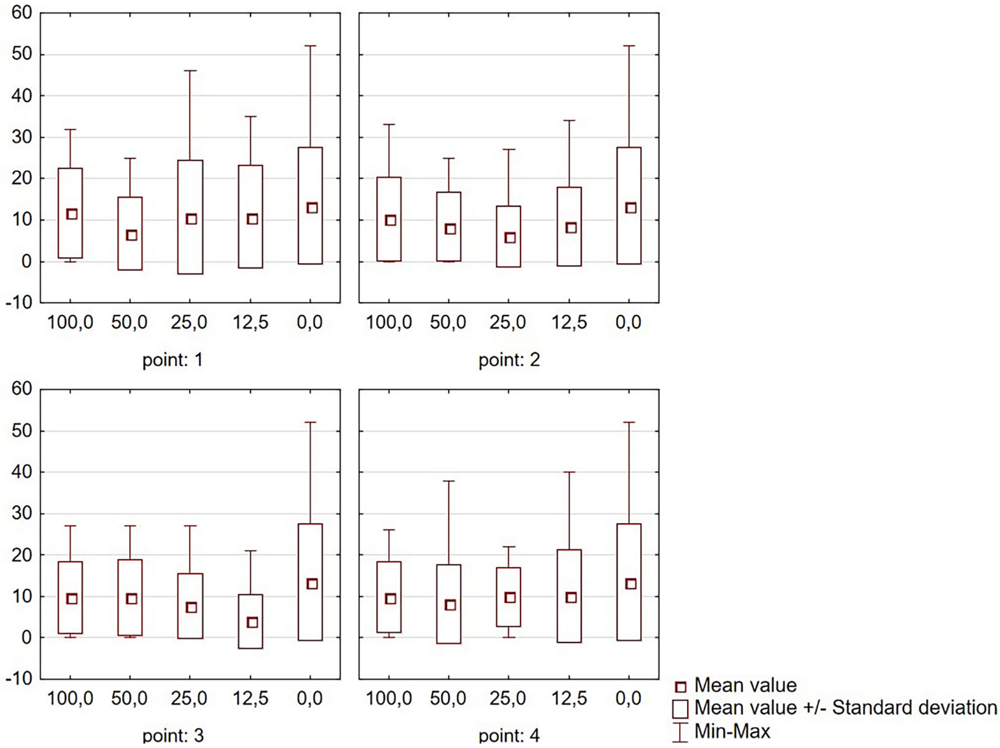

Figure 3. Range of root lengths in water extracts from the soils taken on Solvay settling ponds (0% concentration corresponds to control sample)

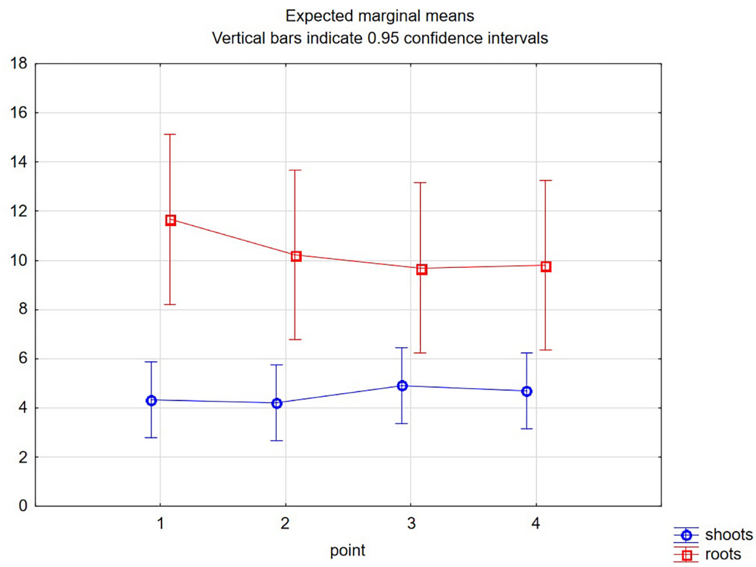

Figure 4. Lengths of shoots and roots in water extracts (100% concentration) from particular sampling points

In the case of above-ground shoots of test plants, the average value of their length is also higher in the control sample than in the other samples (Figure 5). However, the ANOVA analysis and Tukey's post hoc tests did not show any statistically significant differences (Figure 4).

When analyzing the results of the Phytotoxkit test, it can be noticed that for *Sorghum saccharatum* the average root length is clearly higher in the control sample than in the case of the other points (Figure 6). The ANOVA analysis (Figure 7) and Tukey's post hoc test confirm that these are statistically significant differences for the soil collected in points 1–3 (i.e. from sediment pond No. 2). It can therefore be concluded that these soils are toxic to *Sorghum saccharatum*. Significant differences in root length were also observed between the soil from point No. 2 (the smallest average

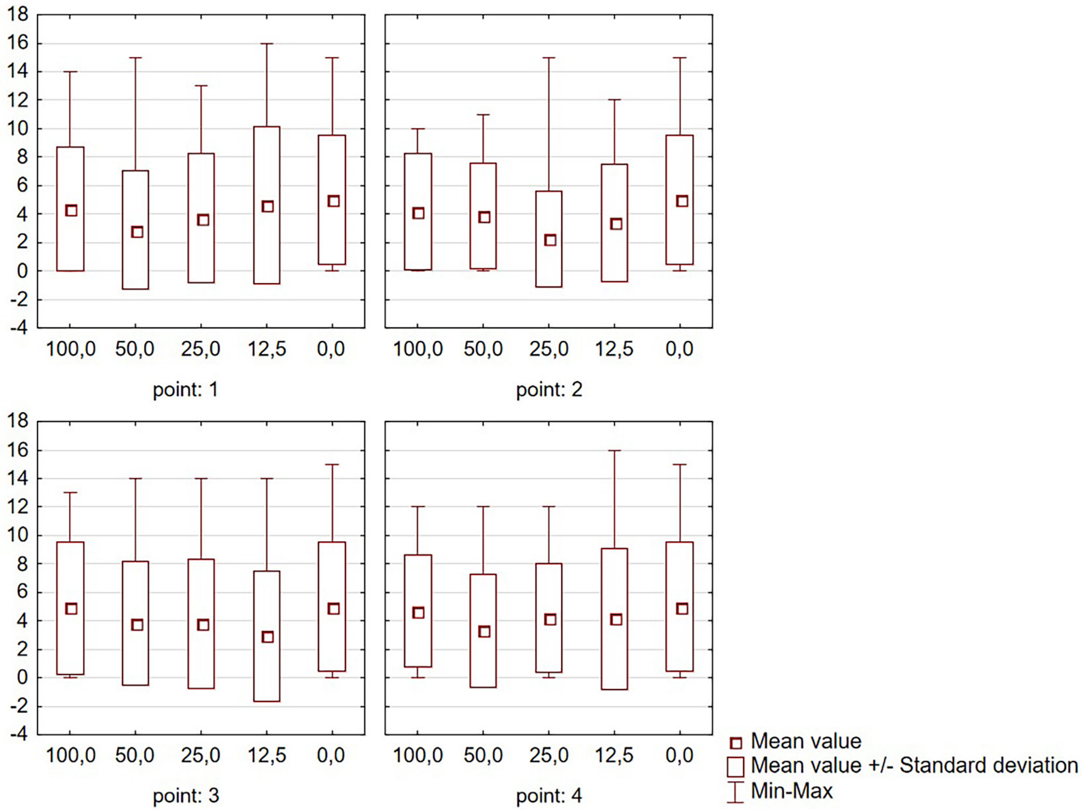

Figure 5. Range of above-ground shoot lengths in water extracts from the soils taken on Solvay settling ponds (0% concentration corresponds to control sample)

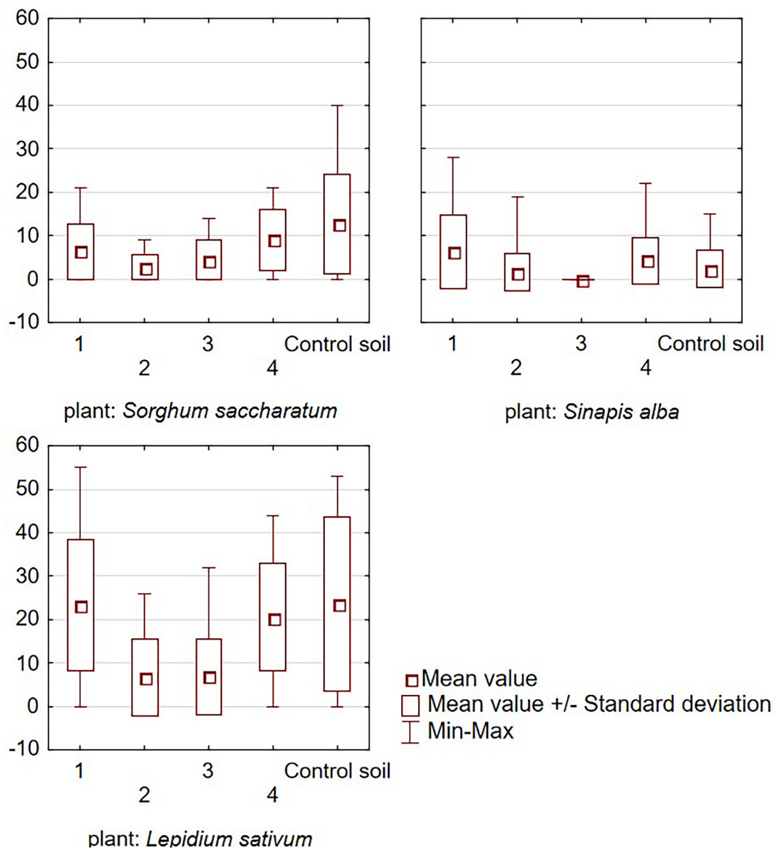

Figure 6. Range of root lengths of plants growing on soil taken from Solvay settling ponds

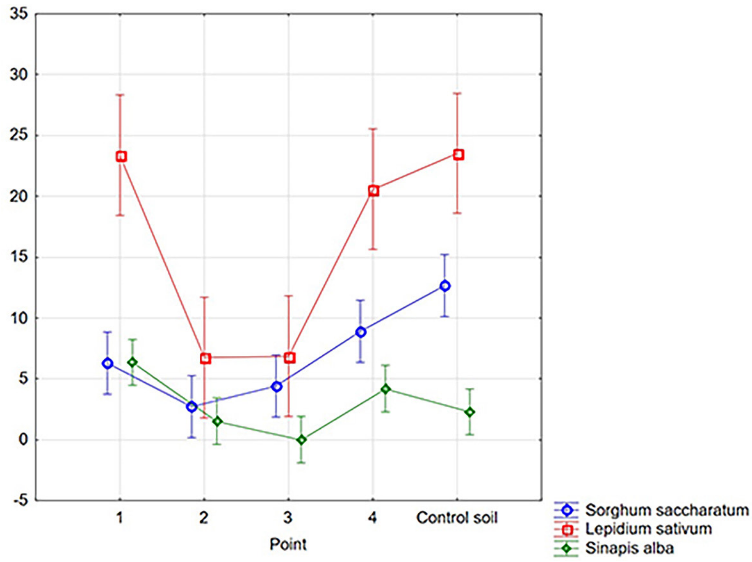

Figure 7. Root lengths of plants growing on soil taken from Solvay settling ponds (ANOVA analysis)

root length value) and the soil from point No. 4. In the case of cress, the average root length is also higher in the control sample than in the other samples. When analyzing Figure 4, it can be assumed that these differences will be statistically significant for sampling points No. 2 and 3 (sediments on top/from surface layer). This is confirmed by ANOVA analysis and Tukey's post hoc test. For cress, two significantly different groups of root lengths can be distinguished: points No. 1, 3 and control (for them, there are no statistically significant differences in root lengths between groups) and points No. 2 and 3 with clearly shorter roots (the roots from these two points also do not show significant differences in length between groups). *Sinapis alba* seeds responded differently to soil properties. In the case of this plant, the longest average root value was recorded for the soil from point No. 1. Their length is significantly different from the *Sinapis alb*a roots growing on the land from points No. 2 and 3 (where white sediments were visible in the soil to the naked eye) and the control. There are also significant differences between the root lengths for points No. 3 and 4.

A phytotoxicity test against *Lepidium sativum* was carried out on the water samples taken from the Wilga River. The research was carried out on the water samples taken before (WBST) and after (WAST) the river flowed through the sediment ponds. For sample WBST, clearly longer roots and shoots were observed in the control sample than in the tested water at different concentrations (Figure 8). ANOVA analysis and Tukey's post hoc test confirm that these differences are statistically significant. However, based on statistical analysis, no significant differences were found in the length of shoots and roots of plants germinating in different concentrations of the tested water.

In the case of the water sample taken from Wilga after flowing through the settling ponds (WAST), the longest roots were observed in the control sample. Their length is significantly different from those of samples with a concentration of 25% and 100% of the tested water. In the case of shoots, the longest ones were obtained in the tested sample with a concentration of 50%; however, the ANOVA analysis and Tukey's post hoc test showed that the lengths of shoots in all tested samples and the control sample did not exhibit statistically significant differences (Figure 9).

The lengths of shoots and roots were also compared in samples with 100% concentrations taken at both points (Figure 10). No significant differences were found in their lengths. On the basis of the tests performed, it can be concluded that despite the increase in ion concentrations (Table 1) in the water collected after the river flowed through the sediments ponds, it did not result in an increase in the phytotoxicity of these waters [Kapanen et al., 2001].

Post-production waste from the soda industry, due to its physical and chemical properties, can negatively affect the environment, causing pollution of all its components. The complex of above-ground sludge ponds of Krakowskie Zakłady Sodowe Solvay, posed a significant environmental problem in the southern part of Krakow. The studies of the quality of the natural

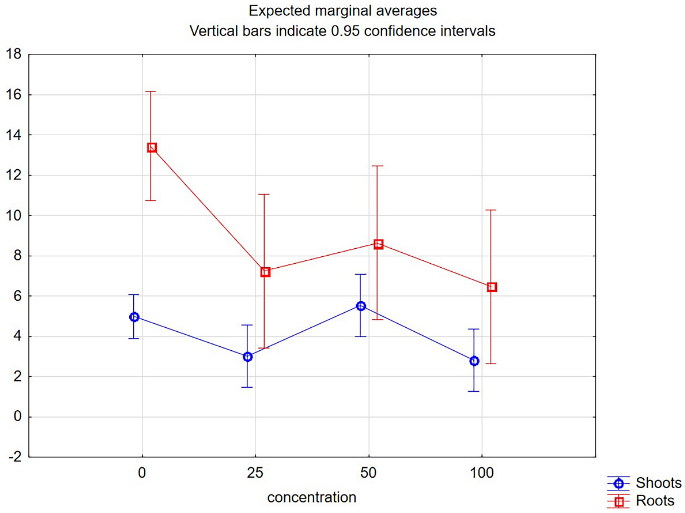

Figure 8. Lengths of *Lepidium sativum* roots and shoots in a water sample taken from the Wilga River before the river entered the settling ponds' area

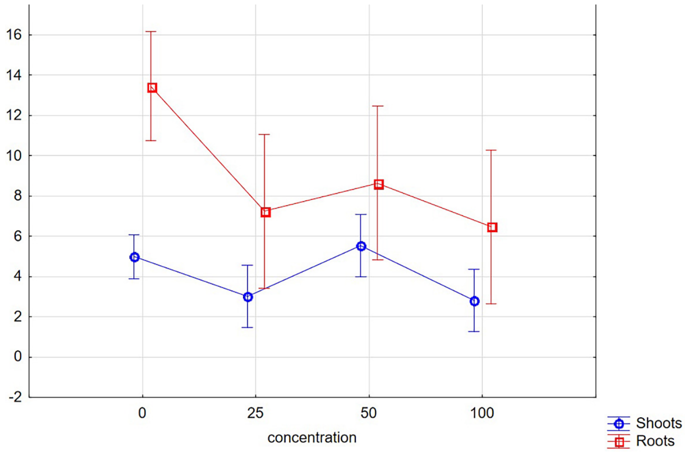

Figure 9. Root and shoot lengths of *Lepidium sativum* in a water sample taken from the Wilga River after the river passed through the area of settling ponds

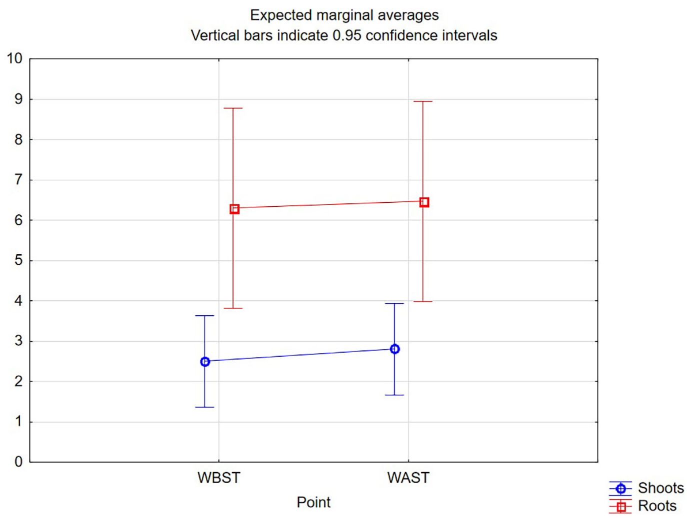

Figure 10. Lengths of *Lepidium sativum* roots and shoots in a water sample taken from the Wilga River before and beyond the area of settling ponds without dilution

environment, conducted after the plant operation ended, showed a significant impact of the deposited post-production sludge on the quality of groundwater and surface water in the vicinity of the settling ponds. No permanent aquifer was found inside the settling ponds; the ponds are fed by rainwater, which infiltrates deep into the profile, leaching sulphates and chlorides from the sludge, among other things, which has changed the properties of the sludge over the past more than thirty years. Saline seepage waters, produce seeps at the foot of the slopes and at the banks of the Wilga River, causing strong mineralization of the waters beneath the settling ponds. The tests carried out in 2006 showed an average mineralization of the waters under the settling ponds of about 22 000 mg/dm^3, the average conductivity of the waters was 1.7 mS/cm, chloride content at: 12 744 mg/dm^3, sulfate: 991 mg/dm^3, calcium (Ca^{2+}) at 4536.5 mg/dm^3 and magnesium (Mg^{2+}):195 mg/dm^3. In addition, the sediments were found to contain carbonates, ranging from 72–96%, as well as Ca, K, Fe and Na. Contaminated waters exhibit sulfate aggressiveness toward concrete. Analysis conducted in 2004 of sulfate and chloride contents in water extracts from the settling ponds and in water taken from the Wilga River at points before the settling ponds and after flowing through the area of settling ponds showed their above-normal contents. They were also not found to be phytotoxic to the test plants, and differences in the early growth of the test plants were statistically insignificant, although a slight inhibition was noted in the test sites compared to the control. More than 30 years after the settling ponds were closed, changes have occurred in the area, related to the changes in the properties of the settling ponds: dewatering and solidification of the sediments, a reduction in the content of sulfate and chloride in the upper layers of the profile, due to their leaching, and a change in the pH of the sediments (in the upper layers around pH = 7.5 in the deeper layers at pH = 13) [Gaszyński et al., 2006, Sroczyński et al., 2009]. Currently, the settling ponds, which are not reclaimed or partially reclaimed, are a green area, overgrown with reclamation vegetation with an increasing dominance of natural succussion, including alien and invasive species. The reclamation layer and the layer of soil that has formed on the crowns of the settling ponds have allowed a fairly dense vegetation cover to develop. A threat to the environment, in addition to the existing leachate, may also be a break in the settler embankments or a breach in the continuity of the reclamation layer, as a result of, for example, construction work, which may lead to waste entering the environment and intensive migration of contaminants into the ground-water environment [Zając et al., 2007, Zając et al., 2016].

CONCLUSIONS

Analysis of the soil samples taken from the area of the settling ponds of the former Solvay soda plant and water samples from the Wilga River, flowing through the area of the settling ponds, allows drawing the following conclusions:

1. Analysis concertation of the chloride and sulfate in the water samples from the Wilga River showed no exceedance of the permissible content of these substances in the water. Also, the pH of the samples was within the norm. It was found that the content of the tested ions was higher in the water samples taken downstream of the settling tanks, which indicates the continuous leaching of ions from the deposited sediments and the transport of pollutants to the groundwater and the river Wilga.
2. It was found that the sediment ponds do not significantly affect the toxicity of the water in the Wilga River, but it should be remembered that water samples were taken in winter, when the amount of rainwater infiltrating deep into the settling ponds was relatively small.
3. The analysis of the leachability of chlorides and sulfates from soil and sediment samples taken from the upper layer of the settling pond (from a depth of 30 cm) did not show any danger to the environment. This is due to the fact that the sediments are washed by rainwater and the historical nature of the facility (35 years since the plants were closed). From the top layers, chlorides and sulfates were washed deep into the profile, also the pH of the top layers of sediments changed (from pH 13 to about 7.5).
4. The Phytotoxkit test showed a different reaction of the test plants against the tested soils. Toxicity occurred at points where the sediments were closer to the surface.
5. The analysis of environmental quality in the area of the sediment ponds of the former Solvay plant needs to be continued. Particularly after heavy rainfall, contaminants may continue to be leached into the environment.

6. The planned reclamation and construction works within the framework of the project for reclamation and revitalization of settling pond No. 2 may contribute to triggering the migration of pollutants from the deeper layers of sediments due to the disruption of the continuity of the embankment or the reclamation layer.
7. Settling ponds No. 2 (partially reclaimed) and No. 3 (not reclaimed), are covered with a layer of vegetation, mainly spontaneous, which confirms the important role of natural succession in the formation of ecosystems in post-industrial areas and the enormous ability of species inhabiting such areas to adapt under such extreme environmental conditions.

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