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Effect of Bentonite and Barley Straw on the Restoration of the Biological Quality of Agriculture Soil Contaminated with the Herbicide Successor T 550 SE

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Abstract: Environmentally safe ways are sought to prevent the accumulation and to accelerate the degradation of herbicide active substances in agricultural soil. This study aimed to determine the effectiveness of finely-ground barley straw and bentonite in mitigating the effects of agricultural soil contamination with Successor T 550 SE. This herbicide was applied in the following doses: 0, 0.73, and 14.63 mg of the active substance per kg. The bentonite and spring barley straw were used at 10 g/kg. The action of these additives was compared to soil without the addition of straw and bentonite. The application of the experimental herbicide disturbed microbial systems, such as organotrophic bacteria, oligotrophic bacteria and their spores, actinobacteria, and fungi. A positive response to the herbicide dose of 14.63 mg a.s./kg was observed only for spores of oligotrophic bacteria. Further disturbances were observed in the agricultural soil biochemical properties, i.e., in the activity of dehydrogenases, urease, catalase, acid, and alkaline phosphatase, arylsulfatase, and β-glucosidase. A significant decrease in the activity of dehydrogenases, acid phosphatase, and arylsulfatase was observed following the application of 14.63 mg a.s./kg. The yield of maize decreased following the application of the analysed plant protection agent. Based on the soil quality index (BA), the addition of straw was more effective in restoring soil homeostasis than bentonite. Both bentonite and straw can be successfully used to improve agricultural soil biological activity. However, more effective mitigation of the negative effects of the herbicide in soil was observed in objects supplemented with barley straw. This improved the microbiological and biochemical properties of the soil. Barley straw was more effective than bentonite in restoring soil biological balance.

Keywords: soil microorganisms; soil enzymes; soil quality index; bentonite; barley straw

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1. Introduction

In the past, when herbicides were not widely used, mechanical weed removal was the weeding method of choice. However, such weed control has never been very effective. Moreover, as history shows, the great migration of the population from the countryside resulted in a reduction in the number of farm workers in rural areas and increased reliance on the use of herbicides [1]. Today, most cropping systems are based on synthetic herbicides. Since the 1950s and 1960s, a steady increase in plant resistance to herbicides has been observed [2].

Glyphosate was one of the most popular herbicides offering effective and efficient control of all emerging weeds. The intensive use of this agent resulted in limited application of plant protection agents containing other active substances and has led to the appearance of weeds resistant to glyphosate [3]. This has raised serious concerns about the ability to control weeds in the cultivation of agricultural crops [4]. Furthermore, the addition of surfactants to herbicides that improve the effectiveness of glyphosate against weeds further increases its environmental toxicity [5,6].

The negative effects of plant protection agents on the environment are associated with the dose applied, the active substance toxicity level [7,8], agent adsorption to soil colloids and persistence in the soil [9], as well as weather conditions [10].

It is a common practice to combine two active substances with different mechanisms of action into one product. The herbicide Successor T 550 SE, manufactured by Stähler International GmbH & Co. KG (Stade, Germany), contains two active substances: terbuthylazine and pethoxamid. Herbicide was launch market in 2009. According to Rodríguez-Cruz et al. [11], the half-life (LD_{50}) of pethoxamid (2-chloro-*N*-(2-ethoxyethyl)-*N*-(2-methyl-1-phenyl-1-propenyl) acetamide) under laboratory conditions ranges from five to eight days, whereas in field experiments it ranges from seven to 22 days. The mean half-life of terbuthylazine (6-chloro-*N*-(1,1-dimethylethyl)-*N*′-ethyl-1,3,5-triazine-2,4-diamine) in soil ranges from 11 to 35 days [12].

Baillie et al. [13] report that terbuthylazine is phytotoxic to aquatic plants, slightly toxic to aquatic invertebrates and moderately toxic to cold-water and warm-water fish. Velisek et al. [14] showed that long-term presence of terbuthylazine in water at the level of 0.003 mg/dm^3 induces disturbances of ontogenesis and histology of common carp in its early stages of life and inhibits the enzymatic activity of superoxide dismutase, leading to damaged nerve canals in the common carp tails at levels of 1.4 and 3.5 mg/dm^3. Haluzová et al. [15] conducted studies assessing the effect of pethoxamid on the development of common carp (*Cyprinus carpio* L.). This substance applied at 0.06, 0.22, and 0.60 mg/dm^3 was not found to cause any changes in cytochrome P450 or the activity of ethoxyresorufin-O-deethylase, whereas an increase in the activity of glutathione and S-glutathione transferase was observed. Arena et al. [16] report that pethoxamid demonstrates acute and chronic toxicity to adult honeybees and maggots, low and chronic toxicity to birds, long-term toxicity to wild mammals and acute toxicity to aquatic organisms.

Terbuthylazine is an s-triazine herbicide, which is most often used to control weeds in the cultivation of maize, and it has been used as an atrazine substitute for over ten years [17]. Terbuthylazine and another agent—pethoxamid (chloroacetamide)—are often used in Europe to control weeds in maize cultivation [18].

The use of plant protection agents in combination with organic additives is a very common agricultural practice which can modify herbicide action [19]. Organic and natural fertilizers can be such additives [20], although excessive amounts of such fertilisers, especially in floodplains, can have an adverse effect by increasing the toxicity of reduced intermediate compounds that accumulate as a result of anaerobic decomposition [21]. Cereal straw can also be used as an organic additive, as it is rich in cellulose and relatively affordable [22]. García-Delgado et al. [19] recommend using compost from plant waste as it minimises the impact of plant protection agents on soil microorganisms and reduces their possible leaching from the soil. The use of bentonite may also be an interesting solution due to its high water and sorption capacity [23]. Bentonite is used in various industries and is mainly composed of clay minerals [24], including montmorillonite and, in smaller amounts, quartz, limestone and sedimentary rocks. Increased quartz and plagioclase content may have an adverse effect on the water absorption capacity of bentonite [25,26]. The absorption of plant protection products by bentonites reactive surface can reduce their leaching to individual environmental compartments [27].

Enzymes participate in organic matter metabolism in the soil [28] and their activity fluctuates over time and depends on substrate availability [29]. Enzymatic activity is often used as an indicator of soil pollution level [30] since enzymes are highly sensitive to anthropogenic and natural factors affecting the soil [31].

Due to the high variability of herbicide toxicity and persistence in soil as well as environmental conditions under which these preparations are used, it is difficult to determine the impact of plant protection products on the environment [8] and it requires several parameters. This study aimed to determine the enzymatic activity in soil, the microorganism count and the maize response to the herbicide Successor T 550 SE. The effectiveness of barley straw and bentonite in mitigating the negative effects of the herbicide Successor

T 550 SE was also analysed. Therefore, the hypothesis has been put forward that the application of barley straw and bentonite is useful in alleviating the negative effects of Successor T 550 SE and that it restores the microbiological and biochemical equilibrium disturbed by the use of this herbicide.

2. Materials and Methods

2.1. Soil

The soil material (150 kg) used in the experiment was collected at the Teaching and Experimental Facility of the University of Warmia and Mazury in Tomaszkowo (NE of Poland, 53.71610 N, 20.41670 E) from the arable humic horizon, at a depth of 0–20 cm. It was proper brown soil formed from sandy loam. According to the World Reference Base of Soil Resources [32], it is classified as Eutric Cambisols. The experimental soil was collected from a field and then transported to the Teaching and Experimental Centre of the University of Warmia and Mazury, where a pot experiment was set up. Samples for the microbiological and enzymatic analyses were collected on day 30, when maize was at phase Biologische Bundesanstalt, Bundessortenamt and Chemica—BBCH 19 (9th leaf) and on day 60 when maize was at phase BBCH 53 (phase of visible efflorescence).

The experimental soil samples collected on the day that the experiment was set up were used to determine the counts of the following microorganisms: organotrophic bacteria (12.855×10^9 cfu/kg d.m.), oligotrophic bacteria (12.527×10^9 cfu/kg d.m.), oligotrophic spore-forming bacteria (3.232×10^8 cfu/kg d.m.), actinobacteria (12.270×10^9 cfu/kg d.m.) and fungi (1.603×10^7 cfu/kg d.m.). In the same samples, the enzyme activity in 1 kg d.m. of soil was determined for the following enzymes: dehydrogenases (16.154 µTFP), catalase (0.222 mol O_2), urease (0.567 mmol N-NH_4), alkaline phosphatase (1.781 mmol PNP), acid phosphatase (1.072 mmol PNP), arylsulphatase (0.245 mmol PNP), and β-glucosidase (0.322 mmol PNP).

2.2. Experimental Materials

The following substances were used in the pot experiment:

1. The herbicide Successor T 550 SE. The product contains two active substances: terbuthylazine and pethoxamid, which are characterised in online resources (Table S1).
2. Bentonite provided by CEBO Holland B.V. composed of sodium montmorillonite—93.85%, quartz—4%, feldspar—1%, calcite—1%, acrylic polymers 0.15%.
3. Straw containing: 54.7% C, 0.33% N, 0.06% P, 0.23% K.

The herbicide was applied at the dose of 0.00, 0.73 and 14.63 mg of the active substance per 1 kg d.m. of soil, while bentonite and barley straw were applied at the dose of 0 and 10 g/kg d.m. of soil. The doses of bentonite and finely ground barley straw were established based on preliminary tests and the available literature. According to the literature [33–38], introducing bentonite and finely ground straw to soil contaminated with chemical substances may contribute to restoring the soil homeostasis. Literature reports describe experiments in which bentonite was introduced to soil at 40 Mg/ha [35], from 25 g to 150 g/kg [36], 10 g/kg d.m. of soil [34] and at 2% of the soil amount [38]. In other experiments, barley straw was added at 9 g/kg d.m. of soil [37] and 5 g/kg d.m. of soil [33]. Based on the above-provided data, for the present experiment, the addition of bentonite and straw at 10 g/kg d.m. of soil was used. This identical dose of bentonite and finely ground straw was used to enable comparison of the effectiveness of both substances in neutralising homeostasis disturbances caused by the herbicide Successor T 550 SE.

2.3. Experimental Design

The soil used for the experiment was first mixed with macronutrients. The same fertilisation dose was applied in all combinations and included nitrogen as $CO(NH_2)_2$ (100 mg/kg of soil) (Chempur, Poland), phosphorus (KH_2PO_4 + KCl—44 mg/kg of soil) (Chempur, Poland) and magnesium ($MgSO_4 \cdot 7H_2O$—20 mg/kg of soil) (Stanlab, Poland).

Additional nitrogen fertilisation as $CO(NH_2)_2$ at 100 mg N/kg was applied at the phase 19 BBCH. The pre-sowing dose of mineral fertilizers was used on the basis of our earlier pot studies with corn. The second dose of nitrogen was applied when the first symptoms of nitrogen deficiency were noticed in the plants. The second dose of phosphorus and potassium was not used because no physiological diseases caused by phosphorus and potassium deficiency were observed on the plants. Soil was placed in 3.5 dm^3 PE pots (3 kg per pot). Seeds of maize LG 32.58 were sown (on day 1) at 7 seeds per pot and 5 seedlings were left in each pot after germination. The soil moisture content was maintained at a constant level (50% of the capillary water capacity) during the plant vegetation period. Each day (2, 3-times), the soil humidity was checked, and any deficit was replenished with deionised water. The maize was harvested at the inflorescence emergence phase (BBCH 53), which took place on day 60 of the experiment. The soil samples for the microbiological and biochemical analyses were collected from each pot on days 30 and 60.

The experiment involved nine combinations of soil (Table 1), herbicide, bentonite, and straw, and each combination was carried out in four replications.

Table 1. Combinations used in the experiment.

Object	Description
1	without Successor T 550 SE and without the neutralising substances (control)
2	with Successor T 550 SE at a dose of 0.73 mg of the active substance per kg and without the neutralising substances
3	with Successor T 550 SE at a dose of 14.63 mg of the active substance per kg and without the neutralising substances
4	without Successor T 550 SE but with bentonite
5	with Successor T 550 SE at a dose of 0.73 mg of the active substance per kg and with bentonite
6	with Successor T 550 SE at a dose of 14.63 mg of the active substance per kg and with bentonite
7	without Successor T 550 SE but with finely ground barley straw
8	with Successor T 550 SE at a dose of 0.73 mg of the active substance per kg and with finely ground barley straw
9	with Successor T 550 SE at a dose of 14.63 mg of the active substance per kg and with finely ground barley straw

2.4. Determination of Microbial Counts

The microorganism count was determined on days 30 and 60 of the experiment. The total count of oligotrophic bacteria and their spores, organotrophic bacteria, actinobacteria and fungi was determined. A detailed description of microbiological analyses is presented in the paper by Kucharski et al. [39], whereas the diversity of organotrophic bacteria, actinobacteria and fungi were calculated with the colony growth index CD [40] and ecophysiological diversity (EP) [41].

2.5. Determination of Enzymatic Activity

The enzymatic activity of the soil was determined at the same dates as the microorganism count. The enzyme activity was determined in the soil used for the experiment. These results are presented in the Sections 2 and 2.1. However, during the experiment, the enzyme activity was tested on days 30 and 60. The activity of the following enzymes was examined: dehydrogenases (EC 1.1), catalase (EC 1.11.1.6), acid phosphatase (EC 3.1.3.2), alkaline phosphatase (EC 3.1.3.1), urease (EC 3.5.1.5), arylsulfatase (EC 3.1.6.1) and β-glucosidase (EC 3.2.1.21). The detailed procedure for determining the soil enzyme activity is described by Kucharski et al. [39]. The soil quality index (BA), as proposed by Wyszkowska et al. [42] was also determined. The soil quality index (BA) was calculated from the activity of

seven enzymes: dehydrogenases (Deh), catalase (Cat), acid phosphatase (Pac), alkaline phosphatase (Pal), urease (Ure), β-glucosidase (Glu), and arylsulphatase (Aryl).

2.6. Maize Response to the Herbicide

The maize response to Successor T 550 SE and to the neutralising substances (bentonite and finely ground barley straw) was determined by observing the maize growth and development based on its yield. The growth phases of maize were determined according to the BBCH used around the world to describe crop growth phases. The BBCH scale and its history were first described by Meier et al. [43]. The scale uses a decimal code system with ten main phases (0 to 9), each of which is then divided into subphases. The moment when 10% of plants exhibits the features described in a given phase is regarded as the beginning of the phase. The full phase is a period when 50% of plants have entered the given development phase. On day 60 of the experiment, the maize (at phase BBCH 53) was cut down and its above-ground parts were dried in a Binder drier at 60 °C. Subsequently, the dry yield weight was determined and expressed in g/pot. Plants from each pot were dried and weighed separately.

2.7. Statistical Analysis

The results were processed with the Statistica 13.1 software package [44], with a multivariate ANOVA and Duncan's tests at the significance level of 0.05. The percentage of the observed variability η^2 was calculated and homogeneous groups were identified with Tukey's test. The microorganism response to the herbicide Successor T 550 SE and to the experiment duration was compared using cluster analysis and by drawing up a dendrogram using Ward's method. A herbicide, bentonite, and straw impact index ($IF_{B/S}$) on the microorganism count and the soil enzyme activity was calculated from the following formula:

$$IF_{B/S} = \frac{A_{B/S} - A}{A} \quad (1)$$

where:

B—bentonite,

S—straw,

$A_{B/S}$—microorganism count/activity of enzymes in soil with the addition of the studied substances,

A—microorganism count/activity of enzymes in soil without the addition of the studied substances.

The maize resistance (RS) was calculated from the formula proposed by Orwin and Wardle [45]:

$$RS = 1 - \frac{2|D_0|}{C_0 + |D_0|} \quad (2)$$

where:

C_0—maize yield in uncontaminated pots during time t_0,

P_0—maize yield in pots under pressure of the herbicide Successor T 550 SE during time t_0.

$D_0 = C_0 - P_0$.

3. Results

Considering the variability of individual factors (Figure 1), it was shown that the oligotrophic spore-forming bacteria, organotrophic bacteria, and actinobacteria count was determined to the greatest extent by the type of substance used (straw, bentonite). However, the fungi count was most affected by the herbicide dose applied, whereas the count of oligotrophic bacteria was determined mainly by the interaction between the herbicide dose and the addition of barley straw or bentonite.

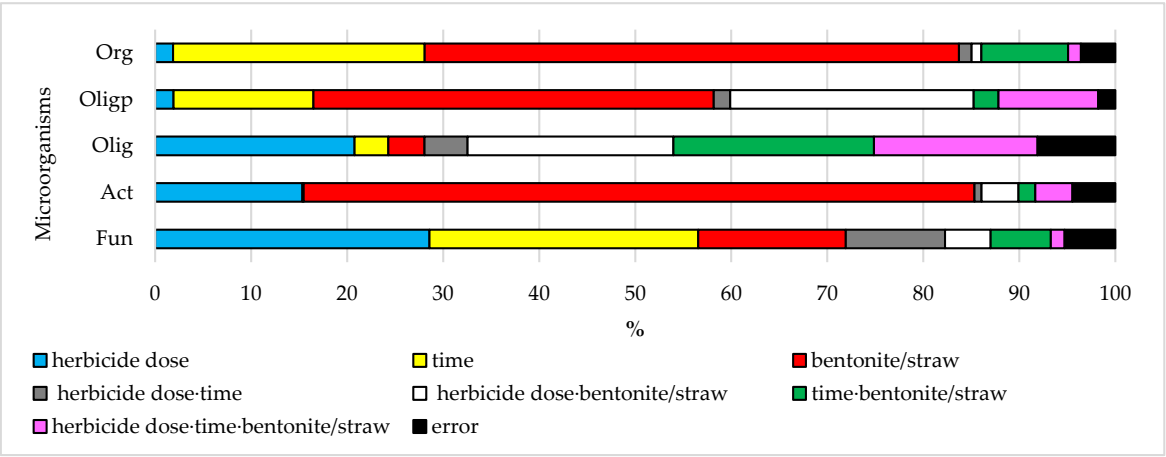

Figure 1. Percent of the observed variability microorganisms η^2 in soil contaminated with Successor T 550 SE. Org—organotrophic bacteria; Oligp—sporeforming oligotrophic bacteria; Olig—oligotrophic bacteria; Act—actinobacteria; Fun—fungi.

On day 30 of the experiment, although the studied herbicide did not have a significant impact on the count of organotrophic bacteria, actinobacteria or fungi, it decreased the total count of oligotrophic bacteria and increased the count of oligotrophic spore-forming bacteria (Table 2).

Table 2. Microbial counts in soil contaminated with Successor T 550 SE.

Dose mg a.s./kg d.m. Soil	Object/Number of Days					
	C_{30}	C_{60}	B_{30}	B_{60}	S_{30}	S_{60}
Organotrophic bacteria (10^9 cfu/kg d.m. soil)						
0	12.12 ^{d}	28.36 ^{cd}	34.12 ^{c}	112.97 ^{a}	64.99 ^{b}	127.13 ^{a}
0.73	11.11 ^{d}	16.15 ^{d}	20.27 ^{cd}	110.94 ^{a}	69.30 ^{b}	123.21 ^{a}
14.63	10.54 ^{d}	14.12 ^{d}	27.15 ^{cd}	63.96 ^{b}	64.05 ^{b}	114.55 ^{a}
Oligotrophic bacteria (10^9 cfu/kg d.m. soil)						
0	13.58 ^{cd}	13.75 ^{c}	13.782 ^{c}	12.95 ^{cde}	15.86 ^{b}	16.02 ^{b}
0.73	10.80 ^{fg}	17.74 ^{a}	11.78 ^{defg}	10.51 ^{g}	17.32 ^{ab}	10.89 ^{fg}
14.63	12.70 ^{cde}	12.45 ^{cdef}	13.47 ^{cd}	10.77 ^{fg}	11.67 ^{efg}	6.60 ^{h}
Sporeforming oligotrophic bacteria (10^8 cfu/kg d.m. soil)						
0	5.64 ^{e}	4.32 ^{fg}	4.61 ^{f}	2.06 ^{i}	8.77 ^{c}	5.95 ^{e}
0.73	14.88 ^{a}	7.15 ^{d}	4.58 ^{f}	2.78 ^{hi}	4.76 ^{f}	3.54 ^{gh}
14.63	10.04 ^{b}	9.11 ^{c}	2.71 ^{hi}	4.04 ^{fg}	7.54 ^{d}	2.46 ^{i}
Actinobacteria (10^9 cfu/kg d.m. soil)						
0	9.56 ^{g}	34.51 ^{ef}	90.31 ^{bc}	68.87 ^{d}	114.82 ^{a}	96.97 ^{b}
0.73	8.94 ^{g}	22.10 ^{fg}	41.95 ^{e}	38.81 ^{e}	78.75 ^{cd}	76.58 ^{cd}
14.63	7.07 ^{g}	9.25 ^{g}	12.37 ^{g}	46.51 ^{e}	78.51 ^{cd}	71.47 ^{d}
Fungi (10^7 cfu/kg d.m. soil)						
0	17.42 ^{de}	47.45 ^{a}	25.54 ^{c}	41.42 ^{b}	19.54 ^{d}	30.51 ^{c}
0.73	17.34 ^{de}	39.65 ^{b}	10.49 ^{fg}	18.55 ^{de}	15.39 ^{def}	25.19 ^{c}
14.63	16.29 ^{def}	27.23 ^{c}	6.53 ^{gh}	4.45 ^{h}	11.08 ^{efg}	12.31 ^{efg}

C—control; B—bentonite; S—straw; 30—30 days; 60—60 days; herbicide dose: 0; 0.73; 14.63 mg a.s./kg soil; a.s.—active substance. The same letters denote homogeneous groups within a given microorganisms group.

On day 60 the organotrophic bacteria count did not change depending on the herbicide dose and the negative impact of the herbicide on multiplication of the total oligotrophic bacteria diminished. The positive impact on multiplication of oligotrophic spore-forming bacteria persisted and a negative impact on the multiplication of actinobacteria and fungi was recorded. The addition of bentonite to the soil increased the organotrophic bacteria and actinobacteria counts, whereas the addition of straw increased the total bacteria count. Neither bentonite nor straw changed the fungi count at the first test date and the addition of these substances inhibited fungal proliferation at the second test date.

Figure 2 shows groups of microorganisms with similar characteristics. The dendrogram grouping microorganisms with respect to the duration of the experiment (Figure 2a) revealed two clusters. The first cluster grouped oligotrophic bacteria identified on days 30 and 60, while the second cluster grouped all the other studied microorganisms. The second dendrogram shows similar microorganism responses to the addition of bentonite and finely-ground barley straw to the soil (Figure 2b). There are three groups featuring similar responses: (1) oligotrophic bacteria in control samples, with an addition of bentonite and finely ground barley straw, (2) oligotrophic bacteria spores in the control sample, and (3) other microorganisms.

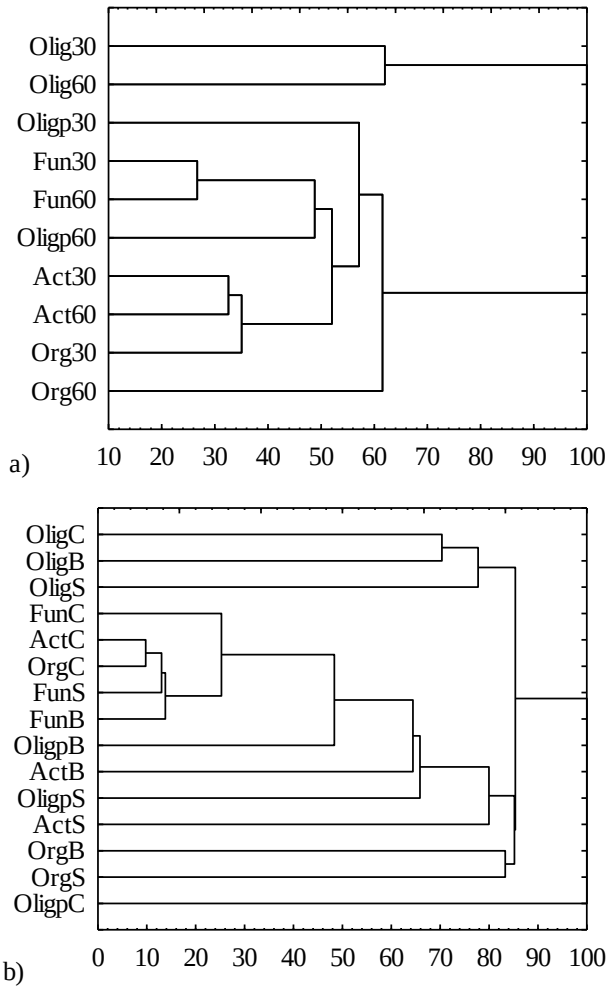

Figure 2. Similar responses of soil microorganisms to Successor T 550 SE. (**a**) to the factor of time; (**b**) to the factor of substance. Org—organotrophic bacteria; Oligp—sporeforming oligotrophic bacteria; Olig—oligotrophic bacteria; Act—actinobacteria; Fun—fungi; C—control; B—bentonite; S—straw; 30—30 days; 60—60 days.

Both the addition of barley straw and herbicide to the soil changed the soil microbial diversity (Figure 3). The addition of barley straw and bentonite decreased the EP index in fungi. Organotrophic bacteria and actinobacteria had similar EP indexes. The fungal colony growth index CD in the pots with no herbicide and in pots with herbicide at the dose of 0.73 mg a.s./kg was similar. The lowest fungal CD was observed in the pots with bentonite and herbicide added at the dose of 14.63 mg a.s./kg. The colony development index (CD) for organotrophic bacteria was the lowest in the pot with an addition of bentonite and treated with Successor T 550 SE at the dose of 0.73 mg a.s./kg. Similar results were found for actinobacteria: the lowest CD levels were observed in the soil with an addition of bentonite (control, herbicide dose of 14.63 mg a.s./kg). These changes are very well reflected by the index of the herbicide, bentonite and finely ground barley straw impact on the soil microbiome (Figure 4).

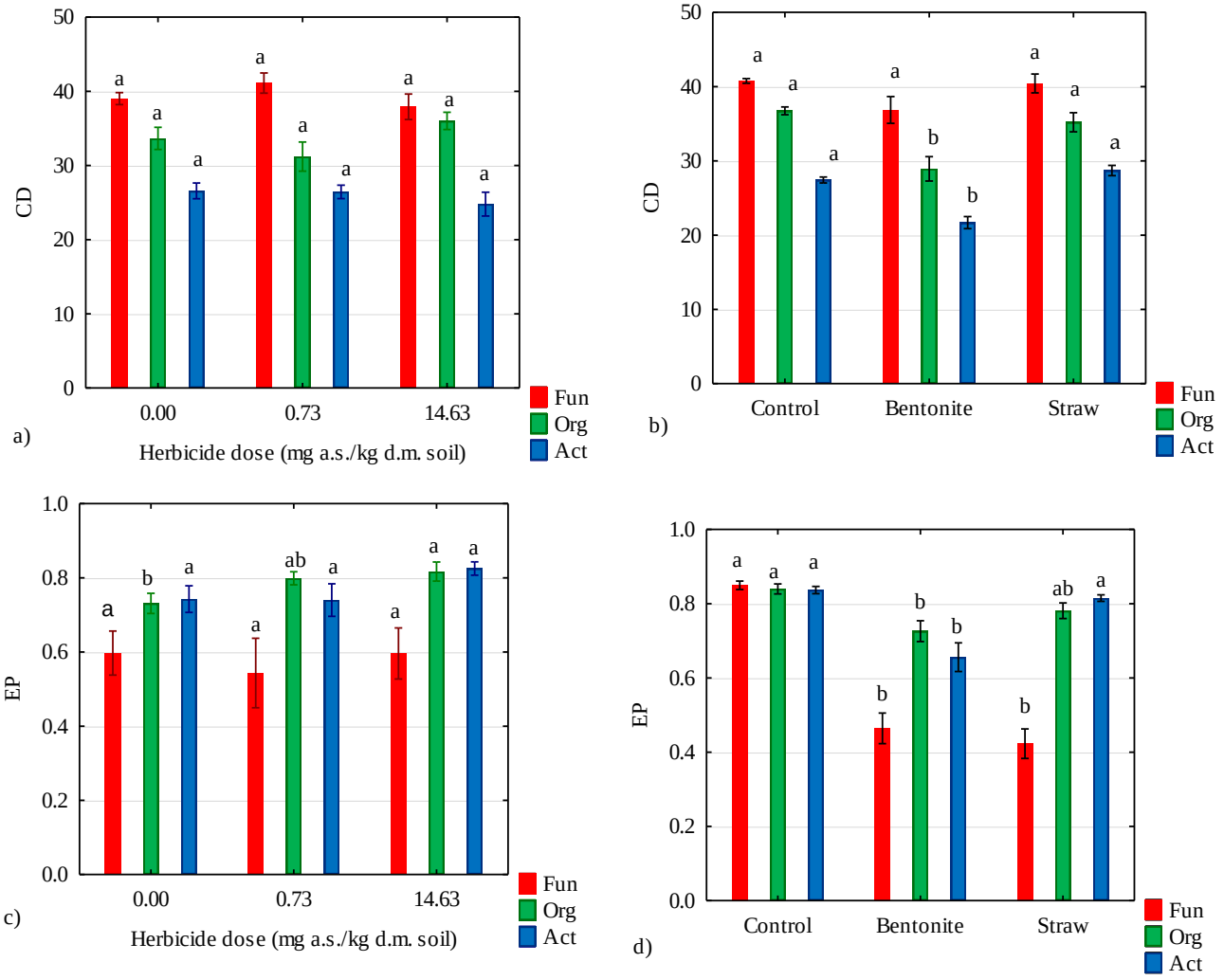

Figure 3. Ecophysiological biodiversity index (EP) and colony development index (CD) in soil contaminated with Successor T 550 SE. (**a**) CD in relation to a herbicide dose; (**b**) CD in relation to bentonite and straw; (**c**) EP in relation to a herbicide dose; (**d**) EP in relation to bentonite and straw. Fun—fungi; Org—organotrophic bacteria; Act—actinobacteria; herbicide dose: 0; 0.73; 14.63 mg a.s./kg soil; a.s.—active substance. The same letters denote homogeneous groups within a given microorganisms group.

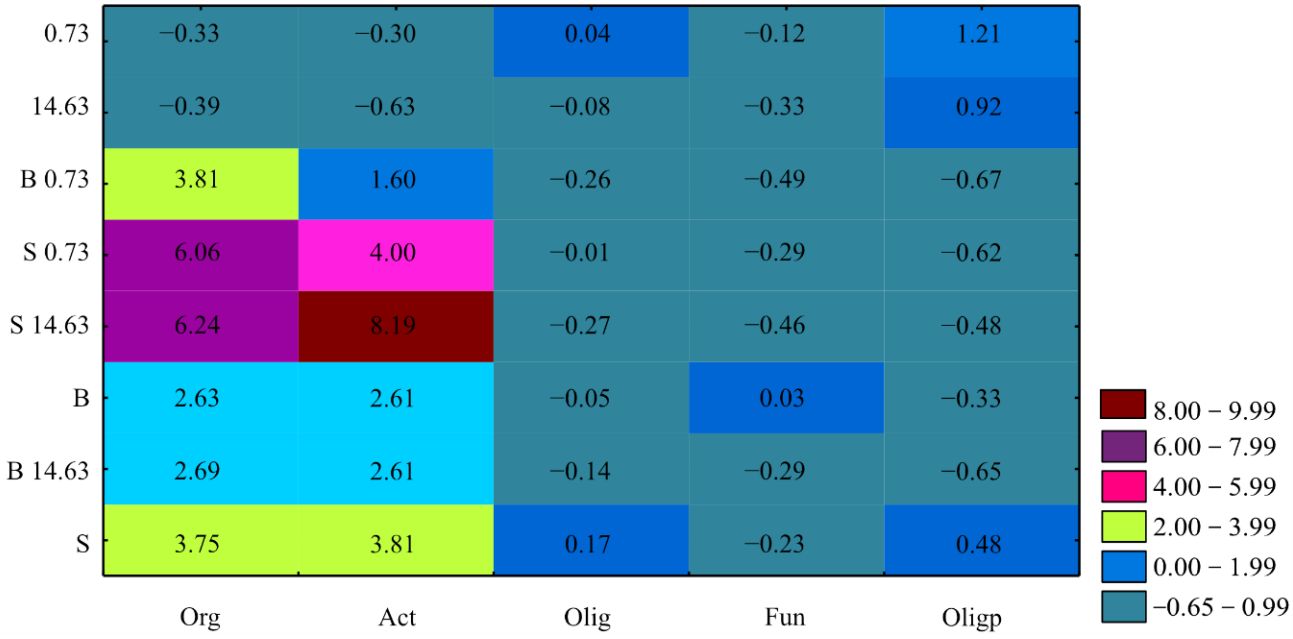

Figure 4. Impact index (IF) of the Successor T 550 SE, bentonite and straw on the microbial counts in soil. Org—organotrophic bacteria; Act—actinobacteria; Olig—oligotrophic bacteria; Fun—fungi; Oligp—sporeforming oligotrophic bacteria; B—bentonite; S—straw; herbicide dose 0; 0.73; 14.63 mg a.s./kg soil; a.s.—active substance.

Of all the studied factors, the activity of dehydrogenases, catalase, urease and acid phosphatase was impacted to the greatest extent by bentonite and barley straw (Figure 5). The activity of alkaline phosphatase and β-glucosidase was affected to the greatest extent by the interaction of two factors: time and a neutralising substance.

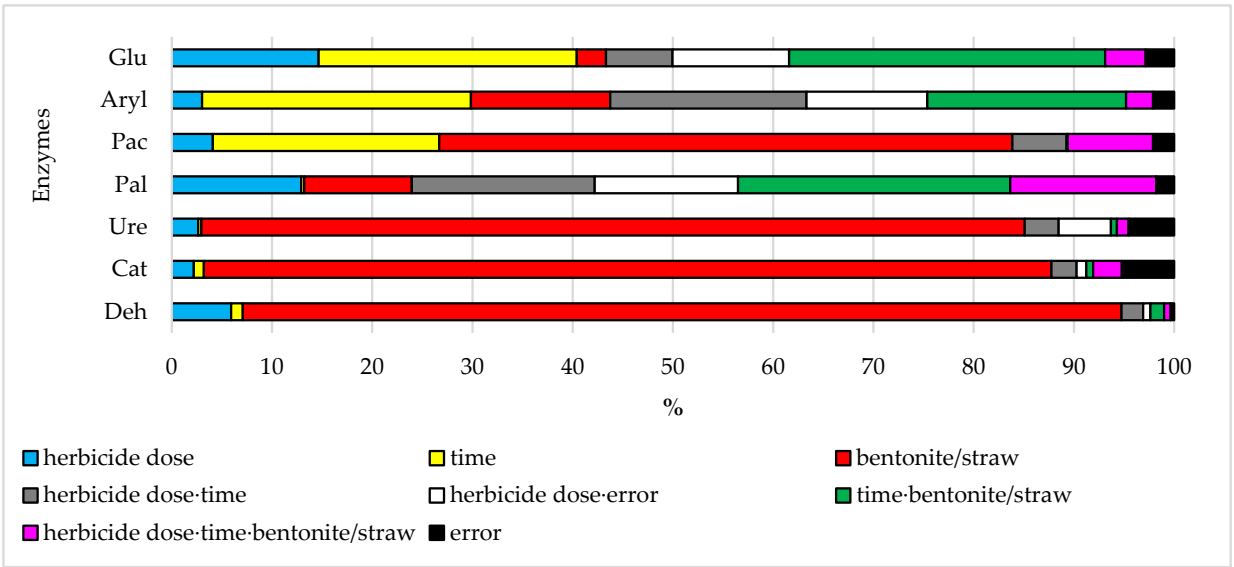

Figure 5. Percent of the observed variability enzymes η^2 in soil contaminated with Successor T 550 SE. Glu—β-glucosidase; Aryl—arylsulfatase; Pac—acid phosphatase; Pal—alkaline phosphatase; Ure—urease; Cat—catalase; Deh—dehydrogenases.

The highest resistance to the impact of the analysed herbicide was demonstrated by urease and alkaline phosphatase, while the lowest resistance was recorded for dehydrogenases and acid phosphatase (Table 3). In the case of the first two enzymes, the herbicide introduced to the soil in a series without an addition of bentonite and barley straw did not

cause any significant changes in the activity, while it significantly decreased the activity of acid phosphatase (on day 30 and day 60 of the experiment) and dehydrogenases. Inhibition of the latter enzyme was caused mainly by the herbicide applied at the higher of the doses (14.63 mg of the active substance per kg of soil). Bentonite added to the soil non-contaminated with the herbicide significantly decreased the activity of dehydrogenases and stimulated the activity of urease and phosphatases. It did not change the activity of arylsulfatase, while it increased the activity of β-glucosidase on day 30 and decreased it on day 60.

Table 3. Enzymatic activity in soil contaminated with Successor T 550 SE.

Dose mg a.s./kg d.m. Soil	Object/Number of Days					
	C_{30}	C_{60}	B_{30}	B_{60}	S_{30}	S_{60}
Dehydrogenase (μmol TPF/kg d.m. soil/h)						
0	14.15 ^{f}	30.18 ^{b}	8.30 ^{i}	18.53 ^{e}	28.64 ^{c}	33.59 ^{a}
0.73	17.24 ^{ef}	25.44 ^{d}	6.14 ^{j}	13.41 ^{g}	32.87 ^{a}	29.09 ^{b}
14.63	12.24 ^{h}	6.45 ^{j}	4.18 ^{k}	6.94 ^{j}	27.35 ^{c}	28.27 ^{c}
Catalase (mol O_2/kg d.m. soil/h)						
0	0.21 ^{gh}	0.31 ^{e}	0.25 ^{f}	0.33 ^{de}	0.45 ^{a}	0.40 ^{b}
0.73	0.20 ^{h}	0.36 ^{cd}	0.26 ^{f}	0.21 ^{gh}	0.45 ^{a}	0.39 ^{bc}
14.63	0.19 ^{h}	0.21 ^{gh}	0.25 ^{f}	0.23 ^{fg}	0.40 ^{b}	0.41 ^{ab}
Urease (mmol N-NH_4/kg d.m. soil/h)						
0	0.61 ^{e}	0.78 ^{e}	0.90 ^{de}	1.39 ^{dc}	2.97 ^{b}	3.20 ^{ab}
0.73	0.69 ^{e}	0.83 ^{de}	0.70 ^{e}	0.95 de	3.05 ^{b}	3.63 ^{a}
14.63	0.60 ^{e}	0.35 ^{e}	0.88 ^{de}	0.82 ^{de}	3.08 ^{b}	1.82 ^{c}
Acid phosphatase (mmol PNP/kg d.m. soil/h)						
0	1.65 ^{g}	3.12 ^{f}	3.46 ^{ef}	3.90 ^{e}	6.50 ^{b}	7.28 ^{a}
0.73	0.88 ^{h}	1.65 ^{g}	3.49 ^{ef}	4.33 ^{d}	4.42 d	7.66 ^{a}
14.63	0.87 ^{h}	1.63 ^{g}	3.14 ^{f}	3.57 ^{e}	5.35 ^{c}	6.91 ^{b}
Alkaline phosphatase (mmol PNP/kg d.m. soil/h)						
0	2.98 ^{fgh}	2.68 ^{ghi}	6.16 ^{dc}	5.40 ^{d}	10.76 ^{a}	6.70 ^{bcd}
0.73	2.22 ^{hi}	1.87 ^{i}	6.63 ^{dc}	4.76 ^{e}	7.10 ^{b}	8.53 ^{b}
14.63	2.25 ^{hi}	1.75 ^{i}	6.39 ^{dc}	3.79 ^{fe}	10.12 ^{a}	6.48 ^{cd}
Arylsulfatase (mmol PNP/kg d.m. soil/h)						
0	0.14 ^{c}	0.26 ^{a}	0.02 ^{e}	0.04 ^{e}	0.03 ^{e}	0.04 ^{e}
0.73	0.11 ^{cd}	0.21 ^{b}	0.02 ^{e}	0.03 ^{e}	0.04 ^{e}	0.02 ^{e}
14.63	0.10 ^{d}	0.20 ^{b}	0.02 ^{e}	0.03 ^{e}	0.04 ^{e}	0.04 ^{e}
β-glucosidase (mmol PNP/kg d.m. soil/h)						
0	0.28 ^{i}	0.47 ^{b}	0.34 ^{fgh}	0.33 ^{h}	0.38 ^{d}	0.42 ^{c}
0.73	0.28 ^{i}	0.49 ^{a}	0.34 ^{fgh}	0.35 ^{fe}	0.29 ^{i}	0.46 ^{b}
14.63	0.28 ^{i}	0.36 ^{de}	0.33 ^{h}	0.33 ^{h}	0.24 ^{j}	0.35 ^{ef}

C—control; B—bentonite; S—straw; 30—30 days; 60—60 days; a.s.—active substance. The same letters denote homogeneous groups within a given enzyme group.

Barley straw added to the soil had a greater impact on the soil enzyme activity than bentonite (Figure 6). The activity of dehydrogenases, catalase, urease, acid phosphatase, alkaline phosphatase and β-glucosidase in soil increased following the addition of the straw. Fertilisation with finely-ground straw had a positive effect on the biochemical properties of soil, which is shown by the soil quality index BA (Figure 7).

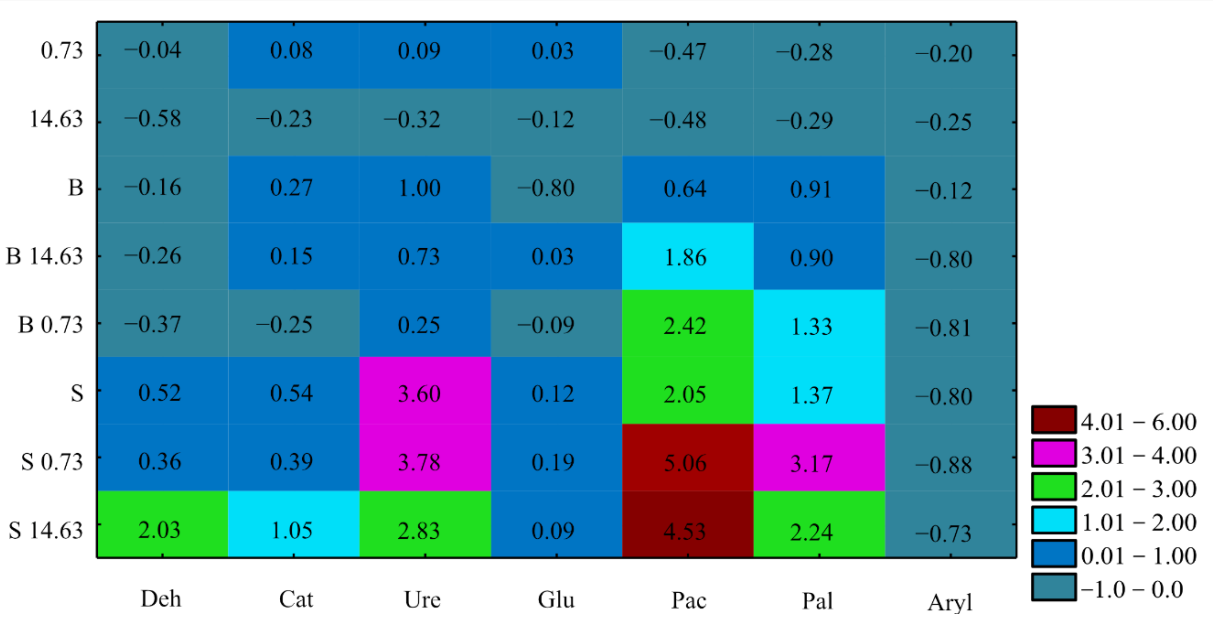

Figure 6. Impact index (IF) of the Successor T 550 SE, bentonite and straw on the enzymatic activity of soil. Glu—β-glucosidase; Aryl—arylsulfatase; Pac—acid phosphatase; Pal—alkaline phosphatase; Ure—urease; Cat—catalase; Deh—dehydrogenases.; B—bentonite; S—straw; herbicide dose: 0; 0.73; 14.63 mg a.s./kg soil; a.s.—active substance.

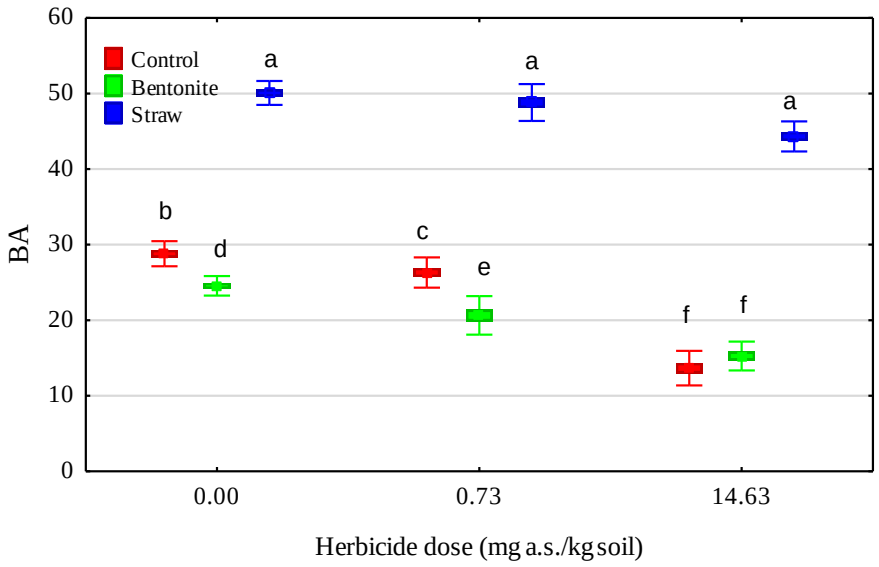

Figure 7. Effect of Successor T 550 SE on soil quality index (BA). The same letters denote homogeneous groups.

Maize proved to be highly sensitive to the studied herbicide (Figure 8). The index of resistance to the herbicide applied at the lower dose was 0.75, whereas it was only 0.05 to the herbicide at a higher dose. Fertilisation with straw significantly increased the maize resistance to the analysed herbicide, whereas bentonite application had no effect.

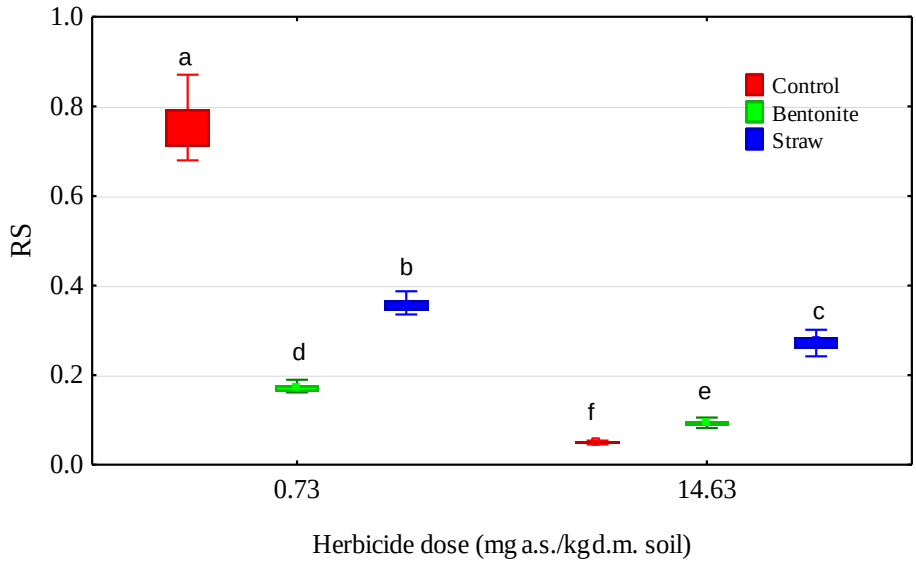

Figure 8. Resistance index (RS) of maize to soil contaminated with Successor T 550 SE. The same letters denote homogeneous groups.

4. Discussion

As a sedimentary clay, bentonite contains a high level of nutrients [46]. It is a good buffer for contaminants since it prevents soil acidification and thereby increases the activity of bacteria [47,48]. The current study confirmed the significant impact of both a mineral substance (bentonite) and an organic substance (finely ground barley straw) on organotrophic bacteria, oligotrophic spore-forming bacteria and actinobacteria. Straw is rich in cellulose, which is an ideal source of carbon and hydrogen for microorganisms inhabiting the soil [22]. The use of bentonite also resulted in an increase in the count of microorganisms compared to samples without the addition of this substance, however, the impact of this substance on microorganisms was weaker than that of straw. Ueshima et al. [49] indicated that bentonite can protect microbial cells from toxins as it surrounds their surface. However, an increase in the bentonite density can lead to the death of some microorganism species, e.g., sulphate-reducing species [50]. Bentonite can be an energy substrate for some microorganisms, mainly chemoautotrophs [51]. Heijnen et al. [52] showed that the introduction of 5% of bentonite clay prior to inoculation into loamy sand improved the bacterial survival rate as a large number of protective microhabitats were formed in the soil. The additives used in the present experiment did not have a beneficial effect on all microorganisms. The highest fungi count was observed on day 60 of the experiment in control pots, i.e., those without bentonite or barley straw. Fukunaga et al. [50] observed lower activity of microorganisms in natural bentonite than in soils or water sediments. This is because it is difficult for microorganisms to get from the wet surface of bentonite inwards. This can be caused by the high density of bentonite, i.e., its low porosity. According to Ritz and Young [53], fungi can fill the soil pores owing to their filamentous structure and the soil pores regulate the growth and development of mycelium. The poor growth of fungi can be attributed to the fact that soil pores were filled with bentonite, which disrupted their development. An increase in the microorganism count following the application of mineral and organic fertilizers was also observed by Thang et al. [54]. The positive effect of barley straw on microorganisms in soil with the addition of herbicide Aurora 40 WG was reported by Baćmaga et al. [55].

Zain et al. [56] showed that herbicides inhibit the growth of soil microorganisms, although their impact depends on the herbicide type. The authors report that paraquat inhibited the multiplication of bacteria (and actinobacteria to the highest degree), while glyphosate had the strongest inhibitory effect on fungi, and metsulfuron-methyl had the weakest impact on microorganisms. According to Zhang et al. [57], the application of

endosulfan at the doses of 0.1 and 1.0 mg/kg of soil changed the structure of bacteria in the soil, but only the highest dose of this substance affected the change of fungi structure. Ayansina and Oso [58] studied the use of two herbicides (atrazine and atrazine combined with metolachlor) and observed a decrease in the number of microorganisms following the application of the optimal dose and of one and a half times higher dose. Moreover, they found that these herbicides eliminated some microbial species. In our research, the unexpected growth of microbial counts for spore forming bacteria after 30 days in variants without soil amendments was most likely the result of the use of herbicide as a source of nutrients and energy. Oligotrophic bacteria grow vigorously in an environment poor in nutrients and with low energy flow, and thanks to a large inflow of carbon sources, they can obtain a higher abundance and biomass [59,60]. The reason for the decreasing in the number of this group of microorganisms on day 60 could be the negative effect of the metabolites formed during the decomposition of the active substances included in the preparation, which showed greater toxicity than the starting compounds themselves. This may have contributed to the imbalance between energy depletion and the availability of non-metabolised substrates. In addition, too many nutrients could cause osmotic shock resulting from the sudden access of these nutrients to bacterial cells [61].

The use of plant protection products may contribute to a change in the soil microorganism diversity despite the unchanged metabolism [62].

Soil biodiversity is determined to a large extent by the diversity of cultivated plants and by the intensification of crop cultivation [63]. The research carried out by Tomkiel et al. [9] with the use of ethyl carfentrazone found the highest EP and CD indices for organotrophic bacteria. Mahapatra et al. [64] reported on changes in bacterial biodiversity and the disruption of bacterial, fungal and actinobacteria populations following the application of imidacloprid. According to Ayansina and Oso [58], preparations such as atrazine and a mixture of atrazine and metolachlor can contribute to the elimination of some microorganisms from the soil.

Enzymes produced by microorganisms participate in biogeochemical cycles of elements in the environment [65] and they transform organic compounds [66]. The use of plant protection products at the recommended rates generally has little or no effect on soil functions, however the application of increased amounts of herbicides makes it necessary to follow the product instructions and to prevent their accumulation in soil [8]. When considering the effects of plant protection products on enzymatic activity, different relationships can be observed. Mahapatra et al. [64] found that the use of imidacloprid reduced the activity of enzymes such as β-glucosidase, acid phosphatase and urease, while dehydrogenases and alkaline phosphatase were not adversely affected. The negative impact of plant protection products on soil enzymatic activity was confirmed by Niewiadomska et al. [67], who used the following herbicides: Stellar 210 SL + Olbras, Maister Power 42.5 OD, Laudis 44 OD, Collage 064 OD, Hector Max 66.5 WG + Trend 0.1% and Arigo 51 WG + Trend 0.1%, which demonstrated an inhibitory effect on the activity of dehydrogenases and alkaline phosphatase.

Bentonite has a high sorption capacity and is considered to be an inexpensive matrix for enzyme immobilization. Its non-toxicity and chemical inactivity enable easy binding of enzymes [68]. According to Akhtar et al. [69], the use of straw increases the contents of organic carbon, nitrogen, phosphorus and water in the soil. Moreover, the addition of straw increased the enzymatic activity in the soil. Thang et al. [54] also showed that the combined use of manure, crop residues and mineral fertilisers increased the activity of β-glucosidase, alkaline phosphatase and arylamidase. The present study also showed that the use of organic matter as finely-ground barley straw generally increased the soil enzymatic activity. In samples with the addition of bentonite, the biochemical properties were also improved, however to a lesser extent than in samples with straw. Such a situation was observed for all enzymes except arylsulfatase, whose higher activity was observed in soil without the herbicide.

An increase in the activity of invertase, acid phosphatase, urease and catalase in soil following the application of straw at the dose of 5000 kg/ha was reported by Akhtar et al. [69] and a positive effect of organic substances added in the form of finely ground barley straw in combination with basalt powder to the soil with the herbicide Aurora 40 WG was observed by Baćmaga et al. [33]. Studies conducted by Mi et al. [70] confirmed the positive effect of bentonite on soil enzymatic activity. They observed that the activity of such enzymes as catalase, invertase, urease and alkaline phosphatase increased following the application of bentonite. Baćmaga et al. [34] report that both bentonite and basalt powder had diverse effects on the enzymatic activity of soil contaminated with fungicides (Amistar 250 SC and Falcon 460 EC). Bentonite reinforced the negative effect of fungicides on dehydrogenases, although the application of both substances mitigated the negative effect of fungicides on catalase and urease.

Wyszkowska et al. [37] proposed a soil quality index (BA), which aptly reflects soil biological condition as it is based on the activity of several major enzymes. It includes: dehydrogenases, urease, catalase, alkaline phosphatase, acid phosphatase, arylsulfatase, and β-glucosidase. In the current study, the highest BA index was observed in pots with the addition of finely ground barley straw. This index decreased with the increasing level of soil contamination with the herbicide Successor T 550 SE. The same conclusions were drawn by Baćmaga et al. [71], who observed that this index decreased following the application of a mixture of diflufenican, mesosulfuron-methyl, and iodosulfuron-methyl-sodium at the highest doses.

Maize is one of the most popular crops grown for fodder and food [72]. It contains many valuable organic and mineral compounds, which are essential in the diet [73]. Since weeds in fields compete with maize, herbicides should be used to maximise maize grain yield and profits [74]. In the present study, it was observed that both maize yield and resistance to the herbicide Successor T 550 SE decreased significantly following its application in a dose of 14.63 mg of the active substance per kg d.m. of soil in control objects. VanGessel et al. [75] also reported the negative impact of herbicides on crop growth. They conducted an experiment using dicamba, diflufenzopyr, nicosulfuron, rimsulfuron, and thifensulfuron preparations used in maize cultivation, in which they observed the greatest inhibition of maize growth following the application of rimsulfuron with thifensulfuron. Moreover, they concluded that the choice of the herbicide used had the greatest impact on maize damage. When selecting a herbicide, many different factors should be taken into account, such as date of application, crop safety, loss of yield due to the competition of already existing weeds and the effectiveness of a given herbicide when applied in combination with another preparation. According to VanGessel et al. [76], the maize yield decreased following the application of foramsulfuron, foramsulfuron, and iodosulfuron, primisulfuron and dicamba, as well as dicamba and atrazine. They also observed that the application of a mixture of herbicides with similar active substances caused a varied response. An addition of bentonite and finely ground barley straw to the soil treated with a dose of 0.73 mg of the active substance per kg d.m. of soil did not improve the maize resistance to the studied preparation. However, the value of the RS index calculated for maize in soil with a dose of 14.63 mg of the active substance per kg d.m. of soil supplemented with barley straw increased, which indicates that this additive can mitigate the negative effects of high doses of Successor T 550 SE. Applying external organic additives to arable soils is one of the oldest and most commonly used agricultural practices. Such additives are greatly beneficial to crops and to the soil itself [77]. They can suppress fungal and bacterial pathogens in the soil and improve the growth and development of crops. The application of easily degradable organic additives to soil (alfalfa straw, wheat straw, glucose) has a positive effect on soil respiration and its catabolic properties. Moreover, frequent supplementation of this kind has a positive effect on soil fungistasis [78]. Joshi et al. [79] demonstrated that organic additives (cereal straw and liquid manure) contribute to the degradation of a herbicide (sulfosulfuron) in soil.

Although bentonite is often used in soil remediation, mainly as a natural sorbent [80], it did not increase the maize resistance to Successor T 550 SE in the present study. This is probably caused by changes in the physical and chemical properties of the soil caused by this clay [36,50]. The studies presented in this manuscript are based on controlled conditions. On the one hand, this is an advantage because the role of individual research factors can be precisely defined, but on the other hand, the role of environmental factors is not taken into account. Therefore, the next stage will be field research.

5. Conclusions

A mixture of terbuthylazine and pethoxamid at the doses of 0.73 mg and 14.63 mg of the active substance per kg of soil may cause disruptions in soil microbial complexes. It may also change soil biochemical properties and worsen soil productivity. The additions of bentonite and barley straw used in the study improve the microbiological and biochemical properties of the soil and thus can accelerate the microbiological transformation of the active substances present in the herbicide Successor T 550 SE. Soil supplementation with barley straw and bentonite had a positive effect on increasing the number of organotrophic bacteria, actinobacteria, and on the activity of dehydrogenases, catalase, urease, acid and alkaline phosphatase. A much better effect was observed following the addition of finely ground barley straw in terms of the activity of these enzymes and microorganisms compared to bentonite.

Supplementary Materials: The following are available online at https://www.mdpi.com/2077-0472/11/1/27/s1, Table S1 Characterization of the Successor T 550 SE herbicide used in the experiment.

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Data Availability Statement: Data are available by contacting the authors.

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