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BALANCE OF HEAVY METALS (Cd, Ni, Pb) IN STATIC FERTILISER EXPERIMENTS ON GRASSLANDS

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A b s t r a c t. On the basis of results obtained in 2 static experiments conducted on light and heavy soils with differentiated mineral fertilisation, an approximate balance of Cd, Ni and Pb was determined over 20 years of investigations. The experiments included 3 levels of fertilisers and the control plot. Ammonium nitrate, single or triple superhosphate and potassium salt were used for the treatments. Average amounts of heavy metals introduced with the fertilisers in both experiments over twenty years were as follows: 37-110 g Cd, 63-195 g Ni and 62-202 g Pb ha^{-1}.

The balance of heavy metals over twenty years assumed different values depending on the fertilisation level and soil conditions. However, irrespective of those factors, the balance assumes a negative value. The effect of nitrogen doses on the balance was slight and depended on the site trophicity. On the other hand, increasing doses of phosphorus and potassium treatment decreased the value of the negative balance difference of cadmium and, to a lesser degree, of nickel, but increased the lead balance.

K e y w o r d s: heavy metals, balance, meadow soils, long-term experiment, mineral fertilisers.

INTRODUCTION

Beside a positive effect on crop yields, the use of mineral fertilisers may also cause unfavourable changes of soil properties and yield quality. It is particularly true in the case of soil contamination with heavy metals. Cadmium content in phosphorus fertilisers arises most fears and controversies [5,10,11,13,25]. Those fertilisers may also be the sources of other metals, particularly nickel and lead. Results of investigations on the effect of mineral fertilisers on the contents of heavy metals, mainly cadmium, in the soils and plants are often divergent. Numerous experiments have shown that phosphorus treatment did not cause any significant increase in the cadmium content of soils and plants [17,21,25]. In some other papers [12], a visible increase of this metal content in the soil after application of phosphorus fertilisers was observed; however the same fertilisers did not raise cadmium content in plants. The effect of treatment on heavy metal accumulation and uptake by plants may be found particularly at regular applications of high fertiliser doses. Thus the results obtained in long-term field experiments may provide reliable data for the estimation of the soil and plant contamination with heavy metals.

The present work aimed at determining the effect of long-term application of diversified mineral fertiliser doses on the balance of such elements as cadmium, nickel and lead in two meadow communities. This paper continues the presentation of the results on a long-term effect of NPK treatment on heavy metal uptake by a meadow sward and metal content in the soil [3,4].

MATERIAL AND METHODS

The present work utilised results of two static experiments on the influence of long-term diversified mineral fertilisation on yields and chemical composition of a meadow sward and soil properties, conducted on the meadows in the vicinity of Kraków in 1969-1988 [9]. Climatic conditions were similar for both experiments, differences concerned physico-chemical soil properties and botanical composition of the sward.

Experiment I was conducted on a several-year-old meadow located on the leached brown soil (Eutric Cambisol), with granulometric composition of light loamy sand. Orchard grass (*Dactylis glomerata*) prevailed in the sward. The topsoil (0-10 cm) contained 0.66% of organic C and its pH was 5.2 (Table 1). Experiment II was set up on a permanent meadow, on silty alluvial gley soil (Eutric Fluvisol) with the composition of clayey silt. Organic C content was 3.38%, with pH_{KCl} = 6.1. Meadow grass (*Poa pratensis*), meadow foxtail (*Alopecurus pratensis*), couch grass (*Agropyron repens*) and dandelion (*Taraxacum officinale*) occurred in greater amounts. Heavy metal (Cd, Ni and Pb) contents in the light soil may be described as natural, and in the heavy soil as increased [14].

The experimental design included 7 objects which differed in the nitrogen, phosphorus and potassium doses. During the experiment, phosphorus doses remained stable, and were the same in both experiments, whereas nitrogen and potassium doses were changing (Table 2). Ammonium nitrate, single superphosphate (in 1969-1978) or triple (1979-1988) and potassium salt (40% K_2O in 1968-1972, 60% K_2O in 1973-1976, 58% K_2O in 1977-1988) were used for the treatment. Three cuts of the sward were harvested each year.

T a b l e 1. Some properties of soil before start of experiments

Soil properties	Light soil		Heavy soil	
	Layer (cm)			
	0-10	10-20	0-10	10-20
Particles < 0.02 mm (silt + clay), %	12	13	39	27
pH_{KCl}	5.2	5.4	6.1	6.2
Cation exchange capacity (CEC), cmol(+) kg^{-1}	6.9	5.0	38	34
Organic C, %	0.66	0.54	3.38	2.20
Total nitrogen, %	0.08	0.07	0.41	0.31
Cd, mg kg^{-1}	0.63	0.63	1.65	1.62
Ni, mg kg^{-1}	5.82	5.79	21.63	21.42
Pb, mg kg^{-1}	20.35	19.70	49.70	45.02

T a b l e 2. Scheme of experiments and doses of fertilisers

Treatment	Yearly doses in kg ha^{-1}									
	Experiment I and II						Experiment I		Experiment II	
	N			P	K		N	K	N	K
	1969-70	1971-72	1973-78	1969-88	1969-75	1976-78	1979-88		1979-88	
0	0	0	0	0	0	0	0	0	0	0
$N_1P_1K_1$	60	90	135	17.5	58	87	95	62	135	87
$N_2P_1K_1$	120	180	270	17.5	58	87	180	58	270	87
$N_3P_1K_1$	180	270	405	17.5	58	87	285	62	405	87
$N_2P_2K_2$	120	180	270	35.0	116	174	180	116	270	174
$N_3P_2K_2$	180	270	405	35.0	116	174	285	124	405	174
$N_3P_3K_3$	180	270	405	52.5	174	261	285	186	405	261

Total content of the studied metals was assessed in the applied fertilisers, soil samples collected prior to the experiment and in the meadow sward using the flame technique in a PU 9100X Philips atomic absorption spectrophotometer, with a background correction with a deuterium lamp [22].

Approximate balance of cadmium, nickel and lead over the 20-year diversified NPK fertilisation was determined on the basis of the specification of the amount of metals introduced with mineral fertilisers and the amount taken up by the meadow sward in the 1^{st}, 5^{th}, 10^{th}, 15^{th} and 20^{th} year of the experiment.

More detailed results concerning the experimental conditions may be found in the authors earlier publications [2-4, 7-9].

Table 3. Balance of Cd, Ni and Pb for the years 1969-78, 1979-88 and 1969-88 in experiment on light soil (I)

Treat-ment	1969-1978			1979-1988			1969-1988		
	Dose[1]	Uptake[2]	Balance	Dose	Uptake	Balance	Dose	Uptake	Balance
	g ha^{-1}								
	Cadmium (Cd)								
0	0	29.4	-29.4	0	30.5	-30.5	0	59.9	-59.9
$N_1P_1K_1$	16.7	58.4	-41.7	20.1	64.5	-44.4	36.8	122.9	-86.1
$N_2P_1K_1$	16.7	64.4	-47.7	20.1	42.2	-22.3	36.8	106.8	-70.0
$N_3P_1K_1$	16.7	71.7	-55.0	20.1	35.2	-15.1	36.8	106.9	-70.1
$N_2P_2K_2$	33.4	72.1	-38.7	40.2	64.8	-24.6	73.6	136.9	-63.3
$N_3P_2K_2$	33.4	82.4	-49.0	40.2	47.1	-6.9	73.6	129.5	-55.9
$N_3P_3K_3$	50.1	94.4	-44.3	60.3	52.8	+7.5	110.4	147.2	-36.8
	Nickel (Ni)								
0	0	69.4	-69.4	0	56.6	-56.6	0	126.0	-126.0
$N_1P_1K_1$	37.4	160.8	-123.4	25.7	145.5	-119.8	63.1	306.3	-243.2
$N_2P_1K_1$	37.4	169.3	-131.9	25.7	132.8	-107.1	63.1	302.1	-239.0
$N_3P_1K_1$	37.4	172.2	-134.8	25.7	102.9	-77.2	63.1	275.1	-212.0
$N_2P_2K_2$	74.8	202.7	-127.9	51.4	178.2	-126.8	126.2	380.9	-254.7
$N_3P_2K_2$	74.8	207.6	-132.8	51.4	149.9	-98.4	126.2	357.4	-231.2
$N_3P_3K_3$	112.2	239.8	-127.6	77.1	181.9	-104.7	189.3	421.6	-232.3
	Lead (Pb)								
0	0	227.6	-227.6	0	123.0	-123.0	0	350.6	-35.6
$N_1P_1K_1$	36.8	416.7	-379.9	25.0	294.3	-269.3	61.8	711.0	-649.2
$N_2P_1K_1$	36.8	459.9	-423.1	25.0	293.2	-268.2	61.8	753.1	-691.3
$N_3P_1K_1$	36.8	472.6	-435.8	25.0	231.1	-206.1	61.8	703.7	-641.9
$N_2P_2K_2$	73.6	510.0	-436.4	50.0	380.9	-330.9	123.6	890.9	-761.3
$N_3P_2K_2$	73.6	537.9	-464.3	50.0	291.8	-241.9	123.6	829.7	-706.1
$N_3P_3K_3$	110.4	586.6	-476.2	75.0	388.7	-313.7	185.4	975.3	-789.9

[1]Amounts of heavy metals introduced with the fertiliser; [2]Uptake with yields.

RESULTS

The primary factors, decisive for the heavy metal balance, are the amount of these elements introduced into the soil with fertilisers and their removal with plant yield. Amounts of the studied metals brought into the soil were increasing along with the level of fertilisation (Tables 3 and 4). The amount of applied cadmium was the same in both experiments and over 20 years it remained at the level of 36.8g in the objects with low phosphorus fertilisation level (P_1), 73.6 g with medium level (P_2) and 110.4 g · ha^{-1} in the objects receiving high doses of phosphorus

Table 4. Balance of Cd, Ni and Pb for the years 1969-78, 1979-88 and 1969-88 in experiment on heavy soil (II)

Treatment	1969-1978			1979-1988			1969-1988		
	Dose[1]	Uptake[2]	Balance	Dose	Uptake	Balance	Dose	Uptake	Balance
	g ha^{-1}								
	Cadmium (Cd)								
0	0	25.5	-26.5	0	21.5	-21.5	0	48.0	-48.0
$N_1P_1K_1$	16.7	45.5	-28.0	20.1	44.6	-24.5	36.8	90.1	-53.3
$N_2P_1K_1$	16.7	55.6	-38.9	20.1	48.3	-28.3	36.8	103.9	-67.1
$N_3P_1K_1$	16.7	58.4	-41.7	20.1	40.5	-20.4	36.8	98.9	-62.1
$N_2P_2K_2$	33.4	67.7	-34.3	40.2	45.9	-5.7	73.6	113.6	-40.0
$N_3P_2K_2$	33.4	65.5	-32.1	40.2	45.9	-5.7	73.6	111.4	-37.8
$N_3P_3K_3$	50.1	68.2	-18.1	60.3	53.1	+7.2	110.4	121.3	-10.9
	Nickel (Ni)								
0	0	108.6	-108.6	0	95.2	-95.2	0	203.8	-203.8
$N_1P_1K_1$	37.4	182.8	-145.4	27.6	200.3	-172.7	65.0	383.1	-318.1
$N_2P_1K_1$	37.4	2551.1	-214.0	27.6	235.7	-208.1	65.0	487.1	-422.1
$N_3P_1K_1$	37.4	298.8	-261.4	27.6	230.7	-203.1	65.0	529.5	-464.5
$N_2P_2K_2$	74.8	234.5	-159.7	55.2	231.9	-176.7	130.0	466.4	-336.4
$N_3P_2K_2$	74.8	252.8	-178.0	55.2	229.8	-174.6	130.0	482.6	-352.6
$N_3P_3K_3$	112.2	258.4	-146.2	82.8	256.4	-173.6	195.0	514.8	-319.8
	Lead (Pb)								
0	0	317.8	-317.8	0	163.5	-163.5	0	481.3	-481.3
$N_1P_1K_1$	36.8	444.8	-408.0	30.5	312.5	-282.0	67.3	757.5	-690.0
$N_2P_1K_1$	36.8	480.8	-444.0	30.5	340.0	-309.5	67.3	820.8	-753.5
$N_3P_1K_1$	36.8	545.8	-509.0	30.5	313.7	-283.2	67.3	859.5	-792.2
$N_2P_2K_2$	73.6	538.4	-464.8	61.0	374.6	-313.6	134.6	913.0	-778.4
$N_3P_2K_2$	73.6	584.3	-510.7	61.0	420.3	-359.3	134.6	1004.6	-870.0
$N_3P_3K_3$	110.4	646.2	-535.8	91.5	491.6	-400.1	201.9	1137.8	-935.9

[1]Amounts of heavy metals introduced with the fertiliser; [2]Uptake with yields.

(P_3). The amounts of nickel and lead were slightly bigger in the experiment on the heavy soil and in the objects where phosphorus and potassium fertilisation levels were the highest (P_3K_3), i.e.: 195 g Ni and 202 g Pb ha^{-1} as compared to 189 g and 185 g, respectively in the experiment on the light soil. Considerably bigger differences between the experiments in the amount of heavy metals removed with yields were observed. The amount of removed cadmium, nickel and lead was increasing with the increasing level of the applied mineral fertilisation. Thus, the lowest uptake of those metals was detected in the control object (0) while the biggest in the object receiving high doses of fertilisers ($N_3P_3K_3$).

Table 5. Average content of heavy metals in mineral fertilisers applied in experiments

Fertilizer	Number of samples	Cd	Ni	Pb
		mg kg^{-1}		
Single superphosphate	8	7.2	13.8	9.2
Triple superphosphate	5	22.7	24.3	12.9
Potassium salt (40% K_2O)	4	traces	5.0	8.8
Potassium salt (58% K_2O)	10	traces	3.6	10.7
Potassium salt (60% K_2O)	4	traces	3.6	10.7
Ammonium nitrate	10	traces	traces	traces

Effect of the kind of treatment on the heavy metal uptake was changing over the years of fertiliser application and depended on the meadow habitat. In the experiment on the light soil, the amount of cadmium taken up over 20 years generally decreased if higher nitrogen doses were applied. A similar tendency was observed for nickel and, to a lesser degree, for lead. However, in the first years of diversified treatment, i.e. in 1969-1978 (and particularly 1969-1973) heavy metal amounts removed with the sward yield definitely increased along with nitrogen doses. A rising level of phosphorus and potassium fertilisation increased the amounts of the removed metals (Cd, Ni and Pb), and the effect was increasing with time.

In the experiment conducted on heavy soil, both the kind and level of mineral treatment generally affected heavy metal uptake in a similar way as on the light soil. Over the 20 years, the amount of nickel taken up was changing to the highest degree (at the maximum by 38% in the $N_3P_1K_1$ object) along with nitrogen doses. However, under the influence of increasing phosphorus and potassium doses the amounts of absorbed heavy metals, mainly cadmium and lead, increased slightly (c.a. 10%).

Balance of the studied heavy metals in the plant-fertiliser design assumes a negative value both in the experiment on the light and heavy soils. Only in the object with the highest level of fertilisation ($N_3P_3K_3$), balance was positive for cadmium in 1979-1988. In the experiment on the light soil, 37-86 g Cd, 125-255 g Ni and 351-790 g Pb ha^{-1} more was removed with the meadow sward yield over 20-year period when compared to the amount of metals introduced with fertilisers. Respective values for the heavy soil are as follows: 11-67 g Cd, 204-464 g Ni and 481-936 g Pb ha^{-1}; thus the values for cadmium are lower and for nickel and lead higher. The highest negative balance difference for cadmium was noted in the objects with low levels of phosphorus and potassium treatment. As the treatment doses increased, this balance difference decreased. An opposite dependency was found for lead: the biggest difference occurred in the $N_3P_3K_3$ experimental objects.

Nickel balance on the light soil was quite uniform, except for the control object and over 20 years ranged between 220 and 255 g ha^{-1} (in the control 126 g ha^{-1}), while in the heavy soil, the highest value of the negative balance was revealed in the $N_3P_1K_1$ (-464 g) and $N_2P_1K_1$ (-422 g Ni ha^{-1}) objects with relatively uniform values in other objects treated with phosphorus and potassium.

DISCUSSION

In the environment which is not subjected to major anthropogenic pollution, heavy metal balance is determined mainly by the content of impurities in the applied fertilisers and their removal with yield [13]. Mineral fertilisers used in the experiments contained different amounts of the studied metals. Phosphorus fertilisers were the main source of those elements. Single superphosphate contained on average 7.4 mg Cd, 13.8 mg Ni and 9.2 mg Pb kg^{-1}, while triple superphosphate, respectively, 22.7 mg, 24.3 mg and 12.9 mg kg^{-1}. Potassium salt contained on an average 3.6 mg Ni and 10.2 mg Pb kg^{-1} and trace amounts of cadmium. Similarly ammonium nitrate revealed only trace amounts of the examined metals.

Cadmium contents in superphosphates used in the experiment falls within the lower section of the range stated in literature [10,11,13]. It might be due to the fact that apatites from the Kola Peninsula were used for production. Unlike phosphorites, especially from Senegal or Congo, they are deficient in this element [11, 16, 26]. Thus cadmium amounts introduced into the soil over 20 years of the experiment are relatively low, i.e. from 36.8 g in the treatments with low phosphorus level to 110.4 g ha^{-1} in the objects receiving high doses of phosphorus, Also the amount of cadmium introduced with fertilisers did not definitely depend on the kind of superphosphate, which is caused by cadmium translocation during the production process, from raw phosphorus to phosphoric acid, and then to fertiliser [3]. Nickel, and particularly lead, remain mainly in the gypsum waste, thus smaller amounts of these metals are introduced in a triple superphosphate as compared to a single one [11,26]. However, depending on the kind of potassium salt, differences in the heavy metal amounts (Ni and Pb) are small and have no significant influence on their quantity introduced into the soil with fertiliser.

The amount of heavy metals removed with yield results from the yield amount and metal contents in the plant mass. The content of Cd, Ni, and Pb in the meadow sward of the discussed experiments over 20 years were presented in another work [4]. Increasing nitrogen doses caused an increased uptake of cadmium, and to a lesser degree - nickel, only in the initial period, particularly in the light soil. In the following years of fertiliser application, a decrease in cadmium content was detected in objects receiving high nitrogen doses as compared to small, and sometimes medium doses. However, phosphorus and potassium fertilisation did not significantly influence these metals uptake, or even decreased their contents in the sward. Lead contents in the sward did not depend to any significant degree on the fertilisation level.

The mechanism of influence of the applied fertilisers on heavy metal contents in plants is a complicated process. It may be a result of increasing soil acidification, changes in the sward botanical composition and amount of yield, or an increase in the soil metal content [4].

The amount of heavy metals taken up by plants is related to the amount of yield. Yield of plants dry mass in both experiments was increasing with increasing fertilisation levels, initially in nitrogen and then also phosphorus and potassium treatments [2,3,7,9]. Over the whole investigated period, sward yield from the objects receiving high doses of fertilisers was on the average 3 times higher in the first and 2.5 times higher in the second experiment when compared to the control. Thus, the amount of heavy metals absorbed by plants increased with the level of applied fertilisation. If we assume the amount of metals taken up by the sward from the control soil for over 20 years as 100, then in the object where the highest fertiliser doses were used ($N_3P_3K_3$), the amount of cadmium removed from soil is 245, nickel 335, and lead 278. The respective values for the experiment II are: 252, 253 and 236. It results not only from an increase in the dry mass yield but also from the activity of other, above-mentioned factors.

The approximate balance of the discussed heavy metals over 20-year period of investigations assumes a negative value. The effect of nitrogen doses applied against the background of various phosphorus and potassium fertilisation levels was slight and depended on the site trophicity. A decrease in the value of the balance difference along with increasing nitrogen doses (particularly in the objects with low doses of phosphorus and potassium on light soils) may be explained by a lower yield of dry mass caused by insufficient doses of phosphorus and potassium, and excessive soil acidification [9]. However, the applied phosphorus and potassium treatments decreased the value of the negative balance difference, particularly for cadmium and, to a lesser degree, for nickel and lead. It results from the amounts of these metals introduced with superphosphate and potassium salt. Cadmium amount present in the superphosphate used in the experiment I, constituted c.a. 33% of its removal with yield from objects with low phosphorus doses, 55% with medium and 75% with high doses. In the experiment II, these values are higher i.e., respectively,: 38, 65 and 90%. Proportions of other metal used in the fertilisers and removed with yield were definitely lower, i.e. 22, 34 and 45% in experiment I and 14, 28 and 38% in experiment II (nickel) and respectively 9, 15 and 19%, and 8, 14 and 18% (lead).

With a negative heavy metal balance, the soil level of those metals should decrease. However, due to the omission of some elements of this balance, particularly the part that is leaching and penetrating the subsoil together with precipitation, the discussed heavy metal balance in the environment is not complete. The determined amount of 1 M HCl soluble cadmium and nickel in the soil sampled after 20 years of diversified fertilisation, showed that as the nitrogen dose increased, the amount of these metals, particularly cadmium definitely decreased in the topsoil, particularly in the light soil [3]. Phosphorus fertilisation slightly increased the content of this cadmium form only in the heavy soil. Changes in the soluble cadmium content in the light soil under the influence of increasing nitrogen doses were higher than expected from this metal balance. Cadmium leaching deep into the soil profile was probably responsible for the above phenomenon. Soil acidification due to the nitrogen applied as ammonium nitrate and small soil sorption capacity favoured the process. Possible cadmium leaching was also suggested by the results of other experiments conducted on sandy soils [6,23]. Nickel, and particularly lead, are strongly fixed by the soil and their leaching is limited [1,18,20].

Very few results of studies on the heavy metal balance, especially on grasslands, are available in literature. In the lysimetric experiments carried out in the Puławy region, cadmium and lead balance was positive, while nickel balance depended on the kind of soil [23, 24]. However, in a four-year field crop-rotation, approximate balance of cadmium and nickel was negative, and the value of balance difference depended on NPK fertilisation level and soil liming [15]. Similarly, the simplified balance of cadmium calculated on the basis of the selected years of static fertilisation experiment on the mountain meadow, depended on the yield amount [17]. In objects treated with nitrogen, the amount of cadmium introduced into the soil with fertilisers was lower than the amount removed from the soil with yield. In an experiment on grasslands (pasture) in New Zealand, where different phosphorus fertilisers with diversified cadmium content (10-41 mg kg^{-1}) were applied for 10 years, plants took up only 1.5-4.5% of the cadmium applied with fertilisers. The results of our 20-year experiments on meadows do not confirm the results of studies in New Zealand. It may be due to the conditions under which the experiments were carried out, particularly different soil properties (among others considerable acidification and low content of organic matter, particularly in the light soil).

CONCLUSIONS

The results obtained from two, 20-year static experiments on the effect of diversified NPK treatment allow to draw the following conclusions:

1. The amount of heavy metals (Cd, Ni, and Pb) introduced into the soil was determined mainly by their contents in phosphorus fertilisers. Subject to the fertilisation level, the respective amounts over 20 years were as follows: 37-110 g Cd, 63-195 g Ni and 62-202 g Pb ha^{-1}.

2. Amounts of the studied metals removed with plant yield increased with the increasing level of the applied nitrogen, phosphorus and potassium treatment. In the experiment on the light soil in the object with the highest level of fertilisers ($N_3P_3K_3$), plants absorbed 246% Cd, 335%Ni and 278% Pb in comparison with the control (100%). In the experiment on the heavy soil, the uptake was, respectively: 252% Cd, 253% Ni and 236% Pb.

3. Approximate heavy metal balance is negative both in the experiment on the light and heavy soils. Increasing nitrogen doses generally caused a rise in the balance difference for Cd, Ni and Pb, especially on the heavy soil. Phosphorus and potassium fertilisation decreased the value of the balance difference for cadmium and nickel, but increased it for lead.

4. Shares of heavy metals introduced into the soil with fertilisers were increasing in the uptake of these metals by plants as phosphorus and potassium fertilising doses were rising. In the objects with low doses of fertilisers (P_1K_1), subject to site conditions, the shares were as follows: 33-38% Cd, 14-22% Ni, and 8-9% Pb. With high fertiliser doses (P_3K_3) the shares were respectively: 75-90, 38-45 and 18-19%.

5. Changes in the amount of heavy metals removed with yield and changes in their balance result from their various contents in the fertilisers. Moreover, side-effects of fertilisation, particularly on the amount of yield and changes in the soil properties, especially acidification related to the nitrogen dose introduced as ammonium nitrate, are numerous.

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