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POLLUTION OF FERTILIZERS WITH HEAVY METALS

ZANIECZYSZCZENIE NAWOZÓW MINERALNYCH METALAMI CIĘŻKIMI

Abstract: The aim of presented investigations was a comparison of heavy metal content in mineral fertilizers used in Poland and determining the sources of their origin in the fertilizers. The methods assumed as referential in fertilizer analysis were used to assess the contents of Cd, Cr, Cu, Hg, Ni, Pb and Zn in 44 samples of mineral fertilizers, including 29 fertilizers containing phosphorus (2 superphosphates and 27 multicomponent fertilizers), 14 nitrogen fertilizers and 1 potassium salt.

Considerable amounts of heavy metals were determined only in phosphorous and multicomponent fertilizers. A small content of lead was assessed in nitrogen fertilizers and in potassium salt, whereas usually only trace amounts of the other metals were detected. In the group of phosphorus and multicomponent fertilizers zinc, chromium and nickel occur in the biggest quantities, whereas mercury and lead in the smallest ones. The potential main source of cadmium, chromium and zinc in fertilizers containing phosphorus is phosphorite raw material while the source of nickel in multicomponent fertilizers is admixtures of ground dolomite. The permissible content of cadmium (50 mg $\cdot$ kg^{-1}), lead (140 mg $\cdot$ kg^{-1}), or mercury (2 mg $\cdot$ kg^{-1}) were exceeded in none of the analyzed fertilizers or mineral fertilizers not used for liming.

Keywords: mineral fertilizers, heavy metals, sources of pollution

Introduction

Mineral fertilizers used as a source of nutrients for plants may sometimes have a negative impact on the environment, mainly on soil and waters. Soil pollution with heavy metals is particularly dangerous [1]. Small contents of these metals in nitrogen and potassium fertilizers do not pose any hazard of soil or plant contamination, however phosphorus and multicomponent fertilizers, and some industrial wastes used for soil deacidification are usually significant factor in heavy metal balance in the environment [2, 3].

Some research projects implemented at the beginning of the last decade in the rural area of the EU [4] demonstrated that at great diversification the applied phosphorus

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fertilizers contained among others on average: 13 mg Cd, 60 mg Cr, 26 mg Cu, 13 mg Pb and 236 mg Zn per 1 kg of fertilizer. Multicomponent fertilizers were characterized by a considerably lower content of these elements, whereas in nitrogen fertilizers respectively: 0.9; 3.4; 2.0; 1.9 and 5.0 mg $\cdot$ kg^{-1} were assessed.

Another source of heavy metals is sulphuric acid used at manufacturing fertilizers containing phosphorus in a water soluble form. However, the basic sources of metals polluting fertilizers containing phosphorus are phosphorites used as a source of phosphorus. African sedimentary phosphorites from Senegal, Togo, Tunisia, some Moroccan deposits and those from North Carolina (USA) contain over 250 mg Cd $\cdot$ kg^{-1} P [2, 5]. During chemical processing of these minerals, cadmium, like uranium, passes mostly into soluble phase and then, in result of technological process, to the fertilizers. It favours mobility of this element and its entering the food chain [6]. The other metals usually form hardly soluble salts and mostly pass to phosphogypsum.

Presented paper aims to compare heavy metal contents in mineral fertilizers used in Poland and determine the sources of these metals origin in fertilizers.

Materials and methods

Heavy metals content was determined in 44 samples of mineral fertilizers, including 2 superphosphates, 27 samples of two- and multicomponent fertilizers, 14 samples of nitrogen fertilizers and one potassium salt. The fertilizers originated from the following enterprises: "Anwil" S.A. Wloclawek, AUREPIO" Ltd., "FOSFAN" S.A. Szczecin, "LUBON" S.A., "POLICE" S.A. Police and Zaklady Azotowe in Tarnow-Moscice.

According to the recommended methods of fertilizer analysis [7], samples of the analyzed fertilizers were dissolved in nitric acid (1:1) with a supplement of redistilled hot water, and the content of Cd, Pb, Ni, Cu, Cr and Zn in the solution were assessed using ICP-AES Ultrace 238 spectrometer made by Jobin Yvon (France). Mercury contents were determined in a fresh weighed portion of fertilizer by means of AMA 254 mercury analyzer. The analyses were conducted in 2 replications and if the *relative standard deviation* (RSD) was higher than 5 %, two additional assessments were conducted.

Results and discussion

Two of the analysed multicomponent fertilizers containing phosphorus (Table 1) were enriched in microelements.

NPKMg fertilizer (No. 16) contained admixtures of boron and zinc, whereas NPKMgS (No. 23) had supplements of boron, manganese and zinc. These supplements were not included in the statistical characteristics of this fertilizer group presented in Table 1.

The executive Regulation of the Minister of Agriculture and Rural Development referring to some provisions of the law on fertilizers and fertilization [8], stated among others permissible contents of arsenic, cadmium, lead and mercury in mineral fertilizers not used for liming. These are: 50 mg As and Cd, 140 mg Pb and 2 mg Hg $\cdot$ kg^{-1} d.m. of fertilizer. The quantities of cadmium, lead and mercury assessed in all analyzed fertilizers were much smaller than the above stated values. In the group of phosphorus

Table 1

Heavy metals content in investigated phosphorus and multicomponent fertilizers

Sample numbers	Chemical composition of fertilizer	Cd	Cr	Cu	Hg	Ni	Pb	Zn
		$[mg \cdot kg^{-1}]$						
1	P = 19	10.74	102.8	9.5	0.132	23.0	2.11	115
2	P = 20	10.41	115.8	20.6	0.064	23.8	4.15	221
3	NPS = 18 – 46 – 5	19.09	274.7	17.6	0.025	36.4	0.82	306
4	PKMgS = 16 – 25 – 2 – 15	10.94	78.5	4.7	0.063	53.5	1.77	95
5	PKMgS = 10 – 20 – 4 – 15	6.06	75.3	6.8	0.105	107.1	1.47	51
6	PKS = 14 – 14 – 23	7.74	69.6	8.4	0.188	15.0	2.09	81
7	PKMgS = 10 – 20 – 2 – 15	7.55	67.6	16.7	0.263	78.8	1.9	93
8	PKS = 11 – 22 – 18	7.57	64.3	14.1	0.103	19.3	1.07	100
9	PKMgS = 12 – 20 – 6 – 10	6.17	119.4	13.9	0.252	246.0	2.54	84
10	PKS = 14 – 24 – 11.5	9.07	83.5	14.9	0.258	71.5	6.49	280
11	NPKS = 4 – 11 – 11 – 27	6.86	65.4	21.6	0.258	20.9	2.25	144
12	NPKMg = 4 – 12 – 20 – 2	12.29	66.4	18.8	0.028	77.5	1.78	107
13	NPKMgS = 5 – 10 – 25 – 2 – 13	4.81	69.5	19.3	0.052	68.5	2.61	83
14	NPKMgS = 4 – 12 – 12 – 2 – 26	5.43	63.2	9.8	0.101	65.7	2.85	107
15	NPKMgS = 5 – 10 – 15 – 3 – 22	5.38	79.3	11.3	0.095	135.5	3.49	128
16	NPKMg = 5 – 10 – 21 – 3	7.64	87.53	13.43	0.071	68.27	4.95	2235*
17	NPKMg = 4 – 17 – 24 – 6	9.05	72.5	12.0	0.064	62.4	1.63	48
18	NPKMg = 3 – 13 – 25 – 6	3.29	52.4	9.4	0.053	55.7	1.93	69
19	NPKMgS = 3 – 13 – 25 – 6 – 10	4.35	61.8	10.5	0.058	59.8	2.17	78
20	NPKMg = 3 – 12 – 18 – 4	6.24	121.7	7.2	0.031	187.1	2.44	109
21	NPKMg = 11 – 8 – 20 – 2	3.33	71.4	5.7	0.024	84.1	4.12	68
22	NPKMg = 5 – 15 – 30 – 2	4.8	112.3	4.5	0.029	193.8	0.50	82
23	NPKMgS = 10 – 8 – 15 – 5 – 35	2.94	65.67	540*	0.019	104.7	4.31	4240*
24	NPKS = 6 – 20 – 30 – 7	6.29	109.9	4.6	0.037	59.3	0.61	126
25	NPKS = 8 – 24 – 24 – 9	8.5	123.2	6.4	0.034	36.4	1.14	162
26	NPKMgS = 5 – 10 – 20 – 7 – 9	3.37	168.7	10.7	0.017	396.0	1.59	77
27	NPKMgS = 12 – 12 – 12 – 2 – 27	4.02	114.5	4.1	0.028	160.9	3.51	71
28	NPKS = 8 – 11 – 24 – 17	3.17	38.7	1.7	0.031	7.6	0.56	64
29	NPKMgS = 4 – 12 – 32 – 2 – 9	4.96	112.2	2.1	0.036	85.1	1.42	101
Minimum content		3.17	38.7	1.7	0.017	7.6	0.50	48
Maximum content		19.09	274.7	21.6	0.263	396.0	6.49	306
Mean content		7.09	94.6	10.6	0.090	90.0	2.19	113
Relative standard deviation [%]		49.4	48.8	54.5	90.3	94.8	60.1	56.0

* Fertilizers supplemented with Cu and Zn – values not taken into account in statistical calculations.

containing fertilizers (Table 1) assessed cadmium contents were lower or much lower than 38 %, mercury lower than 13 % and lead lower than 5 % than the permissible values. On average, analysed phosphorus containing fertilizers had the highest concentrations of zinc, then chromium and nickel and the lowest content of mercury, lead and cadmium.

In the tested fertilizer group the most diversified were the contents of nickel and mercury, for which relative standard deviation was higher than 90 % (Table 1). Relative standard deviations describing diversification of the contents of the other assessed metals in the fertilizers were apparently lower, ranging from 48.8 % to 60.1 %. The most probable source of cadmium, chromium and zinc in fertilizers containing phosphorus are phosphorites used for their manufacturing. This is evidenced not only by the highest and statistically strongly significant values of correlation coefficients describing the relationship between these metals contents and phosphorus content in a fertilizer (Table 2), but also the fact that the biggest amounts of these metals were assessed in monobasic and dibasic ammonium phosphate (NPS = 18 – 46 – 5, No. 3, Table 1).

Table 2

Correlation coefficients (r) between macroelements and heavy metals content in investigated phosphorus and multicomponent fertilizers

Element	No. of samples	Cd	Cr	Cu (n – 1)	Hg	Ni	Pb	Zn (n – 2)
N	19	0.564**	0.655**	0.186	–0.089	0.075	0.047	0.657**
P	29	0.765***	0.771***	0.076	–0.270	–0.223	–0.251	0.645***
K	26	–0.135	–0.024	–0.394*	–0.387	–0.112	–0.386*	–0.038
Mg	18	–0.573*	–0.151	–0.146	–0.090	0.240	–0.268	–0.563*

r significant at: * $\alpha \le 0.05$, ** $\alpha \le 0.01$, *** $\alpha \le 0.001$.

Generally, increased amount of nickel is determined in the fertilizers which also have magnesium among their components. Very high diversification of nickel content in the tested fertilizers: 7.6–396.0 mg · kg^{-1} d.m. was caused by ground dolomite supplement in the fertilizers. The dependence was even more pronounced in the studies on a similar fertilizer group conducted several years ago [9]. Dolomite supplements to fertilizers used at that time might have been also more polluted with cadmium and zinc than currently applied magnesium raw materials, which dilute cadmium and zinc in the investigated fertilizers (negative coefficients of correlation between Cd and Zn contents and Mg content in the fertilizer – Table 2), while formerly they used to be a significant source of these metals concentrations in fertilizers.

A significant, although statistically weaker, in comparison with phosphorus, relationship should be noticed between the contents of nitrogen, cadmium, chromium and zinc in the analyzed fertilizers containing phosphorus (Table 2). The more nitrogen the fertilizer contained, the more polluted it used to be with these metals. Mercury, like copper and lead contents in a fertilizer does not show any apparent dependence on any of the analyzed macroelements making up the studied fertilizers.

The rates of phosphorus and multicomponent fertilizers are usually determined on the basis of their phosphorus contents. Therefore, the results of conversion of the heavy metal amount assessed in the analyzed fertilizers with reference to phosphorus content in these fertilizers were compiled in Table 3.

Table 3

Heavy metals content in investigated phosphorus and multicomponent fertilizers

No. of sample	Chemical composition of fertilizer	Cd	Cr	Cu	Hg	Ni	Pb	Zn
		$[mg \cdot kg^{-1}\ P_2O_5]$						
1	P = 19	56.5	541.2	50.2	0.695	121	11.1	605
2	P = 20	52.1	579.0	102.9	0.320	119	20.8	1105
3	NPS = 18 – 46 – 5	41.5	597.2	38.3	0.054	79	1.8	665
4	PKMgS = 16 – 25 – 2 – 15	68.4	490.8	29.6	0.394	334	11.1	594
5	PKMgS = 10 – 20 – 4 – 15	50.5	627.3	56.8	0.875	892	12.3	425
6	PKS = 14 – 14 – 23	55.3	497.1	59.9	1.343	107	14.9	579
7	PKMgS = 10 – 20 – 2 – 15	75.5	676.0	167.0	2.630	788	19.0	930
8	PKS = 11 – 22 – 18	68.8	584.5	128.2	0.936	175	9.7	909
9	PKMgS = 12 – 20 – 6 – 10	51.4	995.0	116.1	2.100	2050	21.2	700
10	PKS = 14 – 24 – 11.5	64.8	596.4	106.4	1.843	511	46.4	2000
11	NPKS = 4 – 11 – 11 – 27	62.4	594.5	196.4	2.345	190	20.5	1309
12	NPKMg = 4 – 12 – 20 – 2	102.4	553.6	156.7	0.233	646	14.8	892
13	NPKMgS = 5 – 10 – 25 – 2 – 13	48.1	695.0	193.0	0.520	685	26.1	830
14	NPKMgS = 4 – 12 – 12 – 2 – 26	45.3	526.8	81.3	0.842	547	23.8	892
15	NPKMgS = 5 – 10 – 15 – 3 – 22	53.8	793.3	113.0	0.950	1355	34.9	1280
16	NPKMg = 5 – 10 – 21 – 22	76.4	875.3	134.3	0.710	683	49.5	22350*
17	NPKMg = 4 – 17 – 24 – 6	53.2	426.5	70.6	0.376	367	9.6	282
18	NPKMg = 3 – 13 – 25 – 6	27.4	436.7	78.3	0.442	464	16.1	575
19	NPKMgS = 3 – 13 – 25 – 6 – 10	33.5	475.4	80.8	0.446	460	16.7	600
20	NPKMg = 3 – 12 – 18 – 4	52.0	1014	59.8	0.258	1559	20.3	908
21	NPKMg = 11 – 8 – 20 – 2	41.6	892.5	71.4	0.300	1051	51.5	850
22	NPKMg = 5 – 15 – 30 – 2	32.0	748.7	30.0	0.193	1292	3.3	547
23	NPKMgS = 10 – 8 – 15 – 5 – 35	36.8	820.9	6750*	0.238	1309	53.9	53000*
24	NPKS = 6 – 20 – 30 – 7	31.5	549.5	23.2	0.185	297	3.1	630
25	NPKS = 8 – 24 – 24 – 9	35.4	513.3	26.8	0.142	152	4.8	675
26	NPKMgS = 5 – 10 – 20 – 7 – 9	33.7	1687	107.4	0.170	3960	15.9	770
27	NPKMgS = 12 – 12 – 12 – 2 – 27	33.5	954.3	33.9	0.233	1341	29.3	592
28	NPKS = 8 – 11 – 24 – 17	28.8	352.1	15.4	0.282	69	5.1	582
29	NPKMgS = 4 – 12 – 32 – 2 – 9	41.3	935.0	17.5	0.300	709	11.8	842
Mean content		50.1	690.7	83.7	0.702	769	20.0	798

* Fertilizers supplemented with Cu and Zn – values not taken into account in statistical calculations.

The higher the content of "pure component" in a fertilizer, the smaller fertilizer mass is supplied to the soil. For this reason in 2002, Scientific Committee of Toxicology, Ecotoxicity and the Environment operating by the European Commission [2, 4] suggested a three-stage programme of decreasing the maximum cadmium content in fertilizers: by 2006 – 60 mg Cd, by 2010 – 40 mg Cd and by 2015 – 20 mg Cd $\cdot$ kg^{-1} P_2O_5. Reduction of cadmium content in fertilizers to the level of below 20 mg Cd $\cdot$ kg^{-1}

P_2O_5 at many-year application of these fertilizers should lead to maintaining this metal content in soil on an equal level or even its decrease.

The fertilizers presented in this paper, were manufactured in 2005. Average content of cadmium in the tested fertilizers containing phosphorus was 50.1 mg · kg^{-1} P_2O_5, which was by 21.6 mg · kg^{-1} P_2O_5 lower in comparison with this metal content in a similar group of fertilizers manufactured in Poland in 1993–1994 [9]. The level of cadmium pollution 60 mg · kg^{-1} P_2O_5 was exceeded in 7 analysed fertilizers (Table 3). A three-component mixed fertilizer with magnesium supplement was most burdened with this metal (102.4 mg Cd · kg^{-1} P_2O_5). Pollution of the studied fertilizers with mercury and lead, with reference to their phosphorus contents, was visibly lower in comparison with cadmium, whereas the contents of chromium, copper and zinc in these fertilizers should not raise objections.

The analyzed nitrogen fertilizers and concentrated potassium salt contained lower (and in some cases trace) amounts of the assessed metals (Table 4). Lead was the exception, as it occurred in these fertilizers in concentrations on average over twice higher in comparison with the fertilizers containing phosphorus (see Table 1). Only trace amounts of the analysed metals, particularly cadmium and lead, were detected in the analysed urea and ammonium nitrate. Nitro-chalks, mainly those containing magnesium, had slightly elevated contents of the studied metals.

Table 4

Heavy metals content in investigated nitrogen and potassium fertilizers

No. of sample	Chemical composition of fertilizer	Cd	Cr	Cu	Hg	Ni	Pb	Zn
		[mg · kg^{-1}]						
1	N = 46	0.03	1.71	0.65	0.031	0.63	0.59	42
2	N = 46	0.01	0.30	0.69	0.032	0.40	0.30	22
3	N = 34	0.01	0.35	1.49	0.023	0.78	0.38	26
4	NCa = 32 – 2	0.08	1.67	0.82	0.021	0.47	3.79	58
5	NCaMg = 30 – 4 – 2	0.13	1.09	0.57	0.024	0.56	7.58	64
6	NMg = 27 – 4	0.05	0.51	1.37	0.022	0.54	6.54	49
7	NCaS = 27 – 7.5 – 4.5	0.03	0.23	1.65	0.024	0.43	2.82	14
8	NCaMg = 27.5 – 6 – 4	0.11	0.57	0.95	0.021	0.48	6.45	41
9	NCaMgB = 27.5 – 6 – 4 – 0.2	0.10	0.69	0.67	0.022	0.52	6.53	55
10	NCaS = 27.5 – 6 – 4	0.08	0.44	1.07	0.019	0.41	2.81	34
11	NCaMg = 27.5 – 6 – 4	0.14	0.67	0.95	0.023	0.68	7.09	47
12	NCaMg = 27 – 6 – 3	0.28	1.73	0.72	0.006	0.87	15.05	114
13	NCaMgB = 27 – 6 – 3 – 0.2	0.20	1.07	0.62	0.005	0.67	10.91	71
14	N = 21	0.03	1.07	0.35	0.002	0.73	0.39	21
15	K = 60	0.01	0.29	1.17	0.011	0.55	1.31	20
Mean content		0.09	0.83	0.92	0.019	0.58	4.84	45

Also small content of the assessed metals in the concentrated (60 % K_2O) potassium salt should be emphasized (Table 4). This fertilizer is composed almost exclusively of potassium chloride releasing from the potassium bearing minerals, when the components not containing potassium are rejected [1]. Most probably, less concentrated potassium salts, with greater share of ballast would have been more polluted with heavy metals. Concentrated potassium salt added in production of mixed and complex multicomponent fertilizers (PK and NPK) may be the source of potassium, since it may slightly dilute the contents of heavy metals in these fertilizers. It seems to be confirmed also by negative values of all correlation coefficients describing the dependencies between the content of potassium and individual heavy metals in the tested two- and threecomponent fertilizers (Table 2).

Conclusions

1. Considering the analyzed fertilizers, significant contents of heavy metals were assessed only in phosphorus and multicomponent fertilizers. In nitrogen fertilizers and potassium salt only small content of lead was detected, whereas the other metals occurred in trace amounts.

2. In the group of phosphorus and multicomponent fertilizers, zinc occurs in the greatest amounts, then chromium and nickel, whereas mercury and lead in the smallest.

3. The main sources of cadmium, chromium and zinc in fertilizers containing phosphorus is phosphorite raw material, while nickel in multicomponent fertilizers comes from the used admixtures of ground dolomite.

4. Permissible contents of cadmium (50 mg $\cdot$ kg^{-1}), lead (140 mg $\cdot$ kg^{-1}) were exceed in none of the analyzed fertilizers and mercury (2 mg $\cdot$ kg^{-1}) in mineral fertilizers not used for liming.

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ZANIECZYSZCZENIE NAWOZÓW MINERALNYCH METALAMI CIĘŻKIMI

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Abstrakt: Celem prezentowanych badań było porównanie zawartości metali ciężkich w nawozach mineralnych stosowanych w Polsce oraz określenie źródeł pochodzenia tych metali w nawozach. Metodami przyjętymi jako referencyjne w analizie nawozów, określono zawartość Cd, Cr, Cu, Hg, Ni, Pb i Zn w 44 próbkach nawozów, w tym 29 zawierających fosfor (2 superfosfatach i 27 nawozach wieloskładnikowych), w 14 nawozach azotowych i 1 soli potasowej.

Znaczące zawartości metali ciężkich stwierdzono tylko w nawozach fosforowych i wieloskładnikowych. W nawozach azotowych i w soli potasowej oznaczono jedynie niedużą zawartość ołowiu, a pozostałe metale najczęściej wykrywano w ilościach śladowych. W grupie nawozów fosforowych i wieloskładnikowych, w największych ilościach występuje cynk, chrom i nikiel, a w najmniejszych rtęć i ołów. Głównym źródłem kadmu, chromu i cynku w nawozach zawierających fosfor jest najprawdopodobniej surowiec fosforytowy, a niklu w nawozach wieloskładnikowych stosowane domieszki zmielonego dolomitu. W żadnym badanym nawozie nie zostały przekroczone dopuszczalne zawartości kadmu (50 mg · kg^{-1}), ołowiu (140 mg · kg^{-1}) i rtęci (2 mg · kg^{-1}) w nawozach mineralnych nie służących do wapnowania.

Słowa kluczowe: nawozy mineralne, metale ciężkie, źródła zanieczyszczenia metalami ciężkimi