

## CALCIUM CONTENT IN POST-MINING GROUNDS IN THE ŁĘKNICA REGION

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**Summary:** The work contains results of a study on the calcium content – subtotal and soluble in 0.1M HCl – in post-mining grounds in Łęknica area. Subtotal content of calcium in the level of forest litter, regardless of the used fertilization during reclamation and the location of the study, was apparently the highest (av. 4108 mg·kg<sup>-1</sup>). With the increasing depth of sampling the calcium content decreased (up to av. 80 mg·kg<sup>-1</sup> in the parent material). Similar dependence has been noted to the calcium extracted by 0.1M HCl (av. 382 mg·kg<sup>-1</sup> in litter vs. 7 mg·kg<sup>-1</sup> in parent rock). Very significant correlation between the content of the both identified forms of calcium in the test soils has been observed.

**Key words:** calcium, soil, post-mining areas reclamation

### 1. INTRODUCTION

Many of the reclaimed post-mining grounds, relating to lignite mining, shows defective chemistry. Many authors describe low pH and low fertility of ground material as the main limiting factors for the effective implementation of plants on such areas [Greinert 1988, Krzaklewski et al. 1997, Drab et al. 2005, Greinert et al. 2009].

Regulating the pH of soil rich in iron sulphides encountered many problems associated with the need of using large masses of neutralizer and also repeating the procedure. The first of these problems leads to a high cost, indicating the need for a substantial commitment of public funds. The second is highly problematic because of land development – complementary forest liming is in most cases unrealistic. However, reclamation with use of high doses of lime is generally accepted and performed practice to remove the soils phytotoxicity caused by low pH value [Ulrich et al. 1984, Krzaklewski et al. 1997, Greinert et al. 2009]. Some authors describe also the negative behaviour of liming for different elements of the natural environment. Murach and Schünemann [1985] pointed out,

that in the limed grounds otherwise runs the rooting of trees – the fine roots engaged surface soil layers (humus and the upper 5 cm of mineral layer). This observation was further narrowed in the research Hahn and Marschner [1998], who observed plant roots growth after liming only in the humus layer. This causes the exposure of plants to desiccation in dry seasons and tipping of trees in strong winds. Liming may also cause the nitrification increase [Arnold et al. 1994], and nitrate leaching from the soil [Wenzel and Ulrich 1988] (resulting in a deficit of nitrogen for plants [Derome et al. 1986]) and heavy metals mobilisation [Schierl and Kreutzer 1991].

In addition to pH regulation, liming is the primary agrotechnical treatment, enriching the forest and reclaimed soils in calcium. Most of the soil researchers and specialists of environment engineering indicates the role of calcium as a nutrient, as well as the main element determining the cation base saturation (and as an buffering soil acidification factor) [Akselsson et al. 2007, Drouet and Herbauts 2008]. Akselsson et al. [2007] have observed in forest soils of northern Europe, a negative balance of Ca – the similar observations have been made in Poland and other Central European countries as well [Salminen (Ed.) 2005].

The increase of calcium content in the surface layers of soils is noted usually for a period of several years after liming, what has been observed in the cultivation of forest by Smallidge and Leopold [1997]. With this experience, however, these authors observed 10-20-fold increase of the Ca content in organic layer (O) and just 50-100% in the underlying transition layer (OA).

The aim of work was to demonstrate the behaviour of calcium in reclaimed post-mining grounds under coniferous forest vegetation.

## 2. DESCRIPTION OF RESEARCH OBJECT

Described area is located in south-western Poland, in the southern part of Lubuskie province, northeast from the Łęknica town, in the triangle formed by villages: Nowe Czaple - Pustków - Chwaliszowice (Fig. 1). Lignite Mine "Friendship of Nations" led opencast operation in the area of Łęknica from the early seventies of the XXth Century. After exploitation the extensive excavation, partially filled with water and heaps of varying degrees of external technical conformation have arose.

Investigation plots were established on the outer mine-dumps, formed by technical reclamation from the rock material, extracted from the overburden layers.

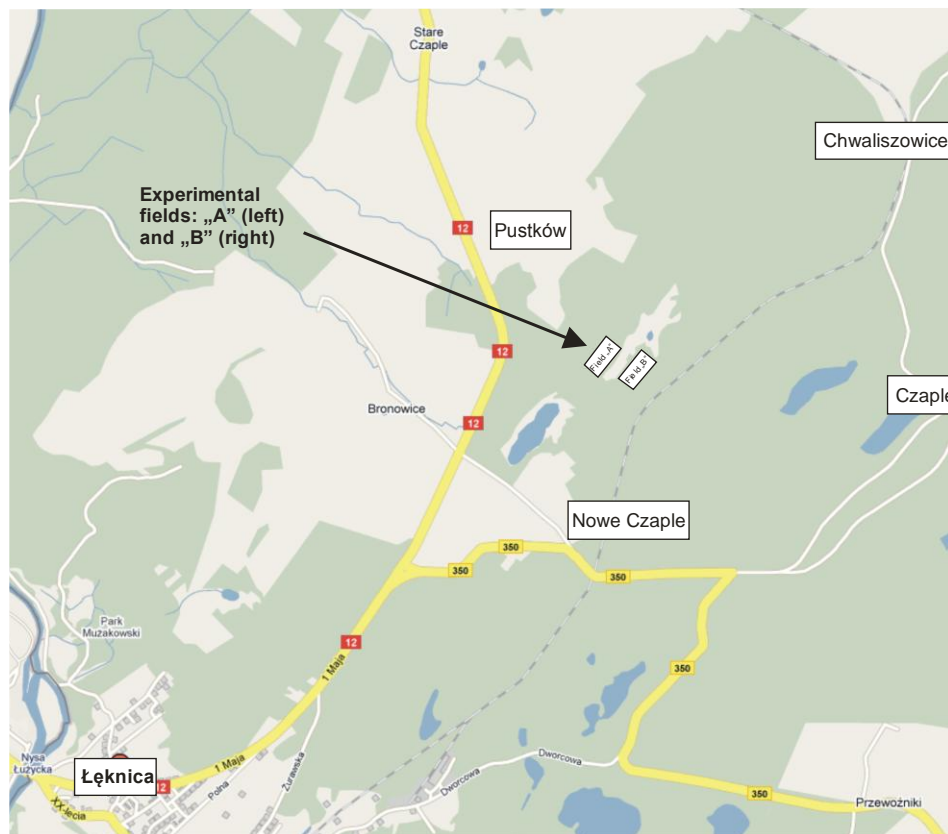


Fig. 1. Location of study area

Post-mining grounds, have been characterized by extremely unfavorable properties in terms of plant growth and development [Greinert 1988, Greinert et al. 2009]. Dump material is dominated by Miocene sands with a large admixture of brown coal dust and pieces and pyrite ( $\text{FeS}_2$ ). Other properties were characterized as follows:

- low water content (below 30%<sub>weight</sub>)
- organic matter content between 1.2 and 6.3%;
- wide C:N ratio (often greater than 100:1);
- low macronutrients total content (excluding potassium);
- very low pH (below 3.0);
- low of available nutrients content.

Described land have been reclaimed in the forest direction. Heaps plateau and the bottom of the pit have been leveled, giving slope gradient 1:3 to 1:4. After the relief shaping, phosphorite meal has been applied at a dose of  $5 \text{ Mg} \cdot \text{ha}^{-1}$  and

magnesium scrap lime from "Miasteczko Śląskie" Ironworks in a dose of 50 Mg·ha<sup>-1</sup>. Then the surface was afforested with Scots pine (*Pinus sylvestris* L.).

### 3. RESEARCH METHODS

The trees grew very poorly, showing symptoms of nutrient deficit. Much of the plantings fell. To explain reason and determine the remedial steps, in 1986 the field experiments have been found – an object "A", on which six-year-old trees grew, and object "B", with two years old planting. Both fields have been fertilised the same way:

1. – without fertilization (A-1, B-1)
2. – magnesium lime 8 Mg·ha<sup>-1</sup> (A-2, B-2)
3. – N – 100, P<sub>2</sub>O<sub>5</sub> – 70 kg·ha<sup>-1</sup> (A-3, B-3)
- 4 – N – 100, K<sub>2</sub>O – 160 kg·ha<sup>-1</sup> (A-4, B-4)
5. – N – 100, P<sub>2</sub>O<sub>5</sub> – 70, K<sub>2</sub>O – 160 kg·ha<sup>-1</sup> (A-5, B-5)
6. – N – 200, P<sub>2</sub>O<sub>5</sub> – 140, K<sub>2</sub>O – 320 kg·ha<sup>-1</sup> (A-6, B-6)
7. – magnesium lime 8 Mg·ha<sup>-1</sup>, N – 100, P<sub>2</sub>O<sub>5</sub> – 70 kg·ha<sup>-1</sup> (A-7, B-7)
8. – magnesium lime 8 Mg·ha<sup>-1</sup>, N – 100, K<sub>2</sub>O – 160 kg·ha<sup>-1</sup> (A-8, B-8)
9. – magnesium lime 8 Mg·ha<sup>-1</sup>, N – 100, P<sub>2</sub>O<sub>5</sub> – 70, K<sub>2</sub>O -160 kg·ha<sup>-1</sup> (A-9, B-9)
10. – magnesium lime 8 Mg·ha<sup>-1</sup>, N – 200, P<sub>2</sub>O<sub>5</sub> – 140, K<sub>2</sub>O 320 kg·ha<sup>-1</sup> (A-10, B-10).

Lime was applied once in November 1986. Nitrogen, phosphorus and potassium were applied in experimental combinations as following fertilizers:

- N – ammonium nitrate
- P – simple dusty superphosphate
- K – potassium chloride, 50% salt

used in spring of 1986.

In 1987 additional mineral nitrogen fertilization o half-part of plots 6 and 10 has been added, in both experimental facilities. This resulted in separation of plots, respectively: A-6a (N - 200 P<sub>2</sub>O<sub>5</sub> - 140 K<sub>2</sub>O - 320 kg·ha<sup>-1</sup>), A-6b (N - 400 P<sub>2</sub>O<sub>5</sub> - 140 K<sub>2</sub>O - 320 kg·ha<sup>-1</sup>), A-10a (N - 200 P<sub>2</sub>O<sub>5</sub> - 140 K<sub>2</sub>O - 320 kg·ha<sup>-1</sup>), A-10b (N - 400 P<sub>2</sub>O<sub>5</sub> - 140 K<sub>2</sub>O - 320 kg·ha<sup>-1</sup>) and, by analogy: B-6a, B-6b, B-10a, B-10b. So in any combination, plot "a" was treated according to the scheme for 1986, and the plot "b" – fertilized with additional nitrogen at 200 kg·ha<sup>-1</sup>. Variants of fertilizer divided the research facilities to plots 35x8 meters each (1-5 and 7-9, individual size 280 m<sup>2</sup>) and 35x4 meters (6 and 10, the surface of individual 140 m<sup>2</sup>).

In autumn 2004, soil profiles have been made on individual plots. The average soil profile samples have been collected from depths: 0-3, 3-8, 8-15, 15-25, 25-50 and 50-75 (plot "B"), 50-100 (plot "A") cm below surface level.

In the samples were determined, among others the calcium soluble in 0.1M HCl (universal extractor in the U.S. analyses) [Page et al. 1982], and subtotal content of calcium after sample digestion with aqua regia [Mc Grath & Cunliffe 1985]. The soil reaction was determined potentiometrically in aqueous, and 1M KCl extracts.

The results were analyzed statistically by calculating Pearson correlation coefficients [Łomnicki 2003, Drab 2007].

#### 4. RESEARCH RESULTS

Calcium subtotal content in samples of forest litter (0-3 cm), irrespective of the fertilizer variation field and the experiment location (both fields) was significantly higher than levels in samples from mineral horizons (Table 1). Mean subtotal calcium content in the litter layer throughout the experiment was 4108 mg·kg<sup>-1</sup>, while in the parent rock (dump material), only 80 mg·kg<sup>-1</sup>. With the increasing depth of soil sampling, the content of described forms of calcium decreased. It should be noted, that decreases of calcium content in layers of 3-8 cm in comparison to forest litter was 5-10 times. Differences in calcium content of less than 8 cm below surface level were much lower.

The average Ca subtotal content from the field "B" was higher by about 340 mg·kg<sup>-1</sup> compared to the calculated for the field "A".

Variants of fertilizers applied in years 1987 to 1989 clearly differentiated calcium subtotal content in the tested ground materials. An increase of calcium in the additionally limed plots, compared to plots fertilized with NPK without calcium has been noted.

Calcium soluble in 0.1M HCl have had a similar tendency like the subtotal form. Mean content of soluble calcium from the whole experiment in the litter layer was 382 mg·kg<sup>-1</sup>, while in the parent rock only 7 mg·kg<sup>-1</sup>. Both experimental fields showed a highly significant correlation between the two forms of calcium (Fig. 2.) Variants of fertilizer did not differentiate clearly the relationship between calcium content in 0.1M HCl and aqua regia extracts (Table 2).

Calcium solubility rate was small, especially in samples taken from the field "A" (Table 1), where it was 8.2% on average. In samples from the "B" field the rate was higher and averaged 21%. It should be noted that in most plots this rate was relatively higher in samples taken from a depth of 3-8 cm and 8-15 cm (layers lying directly beneath the forest litter).

Soil samples from the field "A" showed reaction levels of the forest litter (0-3 cm) within the range of 4,0-4,9 (in H<sub>2</sub>O) and 3,5-4,2 (in 1M KCl). Underlying layer (3-15 cm) showed a pH within 3,5-6,8 (in H<sub>2</sub>O) and 3,1-6,2 (in 1M KCl). Layers containing the rock material (dump material) were characterized by pH between 3,3 and 5,1 (in H<sub>2</sub>O) and between 2,9 and 4,5 (in 1M KCl).

Soil samples from the field "B" had pH of the forest litter (0-3 cm) within range of 4,4-5,6 in (H<sub>2</sub>O) and 3,9-5,0 (in 1M KCl). Layer lying directly under litter (3-15 cm) showed a pH between 3,9 and 5,9 in H<sub>2</sub>O and between 3.5 and 5.5 (in 1M KCl). Layer of the rock material (dump material) was characterized by reaction within the range of 2,9-4,8 (in H<sub>2</sub>O) and 2,4-4,3 (in 1M KCl).

Table 1. Subtotal and soluble in 0.1M HCl calcium content (mg·kg<sup>-1</sup>), and the relation between analysed forms (%)

Field number	Depth (cm)	Ca <sub>HCl</sub>	Ca <sub>subt.</sub>	Ca <sub>HCl</sub> : Ca <sub>subt.</sub> ratio	Field number	Depth (cm)	Ca <sub>HCl</sub>	Ca <sub>subt.</sub>	Ca <sub>HCl</sub> : Ca <sub>subt.</sub> ratio
A-1 „0”	0-3	6520	118	1,7	B-1 „0”	0-2	5672	748	13,2
	3-8	834	60	7,2		2-4	652	472	72,4
	8-15	200	24	12,0		4-6	208	70	33,7
	15-25	188	14	7,4		6-15	180	40	22,2
	25-50	176	23	13,1		15-25	96	20	20,8
	50-100	128	22	17,2		25-50	32	10	31,3
average		1341	44	9,8	50-75	32	10	31,3	
A-2 „0”Ca	0-2	5100	54	1,1	average		982	196	32,1
	2-8	760	44	5,8	B-2 „0”Ca	0-2	5428	616	11,3
	8-15	826	14	1,7		2-6	416	320	76,9
	15-25	143	4	2,8		6-15	96	76	79,2
	25-50	104	5	4,8		15-25	168	20	11,9
	50-100	44	4	9,1		25-50	32	10	31,3
average		1163	21	4,2		50-75	52	3	5,8
A-3 NP	0-2	4758	74	1,6	average		1032	174	36,1
	2-8	714	32	4,5	B-3 NP	0-2	5428	640	11,8
	8-15	273	31	11,4		2-4	1876	508	27,1
	15-25	104	5	4,8		4-15	96	75	78,1
	25-50	104	5	4,8		15-25	96	2	2,1
	50-100	72	4	5,6		25-50	28	2	7,1
average		1004	25	5,5		50-75	28	1	3,6
A-4 NK	0-3	964	98	10,2	average		1259	205	21,6
	3-8	260	31	11,9	B-4 NK	0-3	5848	740	12,7
	8-15	176	9	5,6		3-8	1776	488	27,5
	15-25	104	8	7,7		8-15	180	20	0,6
	25-50	226	6	2,7		15-25	168	40	23,8
	50-100	56	5	8,9		25-50	108	15	13,9
average		298	26	7,8		50-75	58	4	6,9
A-5 NPK	0-3	3172	106	3,3	average		1356	218	14,2
	3-8	524	33	6,3	B-5 NPK	0-2	5116	600	11,7
	8-15	128	10	7,8		2-4	912	440	48,2
	15-25	83	10	12,0		4-8	328	80	24,4
	25-50	83	5	6,0		8-15	124	25	20,2
	50-100	83	5	6,0		15-25	108	20	18,5
average		679	28	6,9		25-50	80	4	5,0
A-6 2NPK	0-3	3586	120	3,3	50-75	68	3	4,0	
	3-8	250	24	9,6	average		962	167	18,9
	8-15	104	13	12,5	B-6 2NPK	0-2	6828	840	12,3
	15-25	56	8	14,3		2-4	928	290	31,3
	25-50	44	5	11,4		4-8	500	204	40,8
	50-100	128	14	10,9		8-15	336	102	30,4
average		695	31	10,3		15-25	80	15	18,8

A-7 CaNP	0-3	6344	108	1,7	B-7 CaNP	25-50	108	10	9,3
	3-8	619	51	8,2		50-75	68	4	5,9
	8-15	176	23	13,1		average	1264	209	14,9
	15-25	128	16	12,5		0-2	7456	968	12,9
	25-50	250	20	8,0		2-4	1236	422	34,1
	50-100	202	5	2,5		4-8	280	76	27,1
average	1286	37	7,7	8-15	180	46	25,6		
A-8 CaNK	0-2	3516	115	3,3	15-25	180	15	8,3	
	2-8	428	34	7,9	average	1558	305	21,6	
	8-15	238	21	8,8	0-2	5220	688	13,2	
	15-25	250	20	8,0	2-4	2400	568	23,7	
	25-50	83	8	9,6	4-8	500	214	42,8	
	50-100	56	5	8,9	8-15	296	60	20,3	
average	762	34	7,8	15-25	180	15	8,3		
A-9 NPK	0-2	4344	106	2,4	25-50	180	3	1,7	
	2-8	786	52	6,6	50-75	96	5	5,2	
	8-15	631	54	8,6	average	1267	222	16,4	
	15-25	273	27	9,9	0-2	5324	550	10,3	
	25-50	56	11	19,6	2-4	1440	468	32,5	
	50-100	44	6	13,6	4-8	488	300	61,5	
average	1022	43	10,1	8-15	60	25	41,7		
A-10 Ca2NPK	0-3	6275	117	1,9	15-25	68	22	32,4	
	3-8	666	52	7,8	25-50	44	5	11,4	
	8-15	297	34	11,4	50-75	44	3	6,8	
	15-25	154	11	7,1	average	1067	196	28,1	
	25-50	104	6	5,8	B-10 Ca2NPK	0-2	4088	224	5,5
	50-100	83	6	7,2	2-15	234	5	2,1	
average	1263	38	12,2	15-25	68	10	14,7		
field average	951	33	8,2	25-50	80	4	5,0		
				average	1118	61	6,8		
				field average	1292	195	21,1		

Table 2. Sequence correlation ratios ( $r_s$ ) for the soluble and subtotal calcium forms

	Field „A”	Field „B”
Control	0,95 <sup>xx</sup>	0,87 <sup>xx</sup>
Ca	0,80 <sup>x</sup>	0,90 <sup>xx</sup>
NP	0,92 <sup>xx</sup>	0,92 <sup>xx</sup>
NK	0,98 <sup>xx</sup>	0,94 <sup>xx</sup>
NPK	0,99 <sup>xx</sup>	0,98 <sup>xx</sup>
2 NPK	0,99 <sup>xx</sup>	0,97 <sup>xx</sup>
CaNP	0,94 <sup>xx</sup>	0,96 <sup>xx</sup>
CaNK	0,98 <sup>xx</sup>	0,93 <sup>xx</sup>
CaNPK	0,92 <sup>xx</sup>	0,82 <sup>x</sup>
Ca 2 NPK	0,93 <sup>xx</sup>	0,99 <sup>xx</sup>

<sup>x</sup> – important relation ( $\alpha = 0.05$ )

<sup>xx</sup> – very important relation ( $\alpha = 0.01$ )

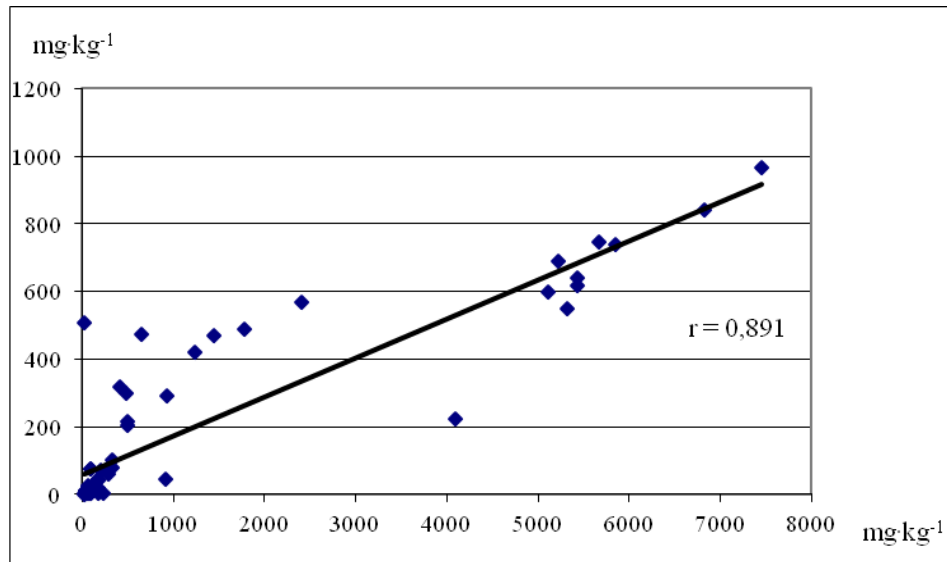


Fig. 2. Correlation between subtotal and 0.1M HCl soluble Ca forms content – experimental field "B"

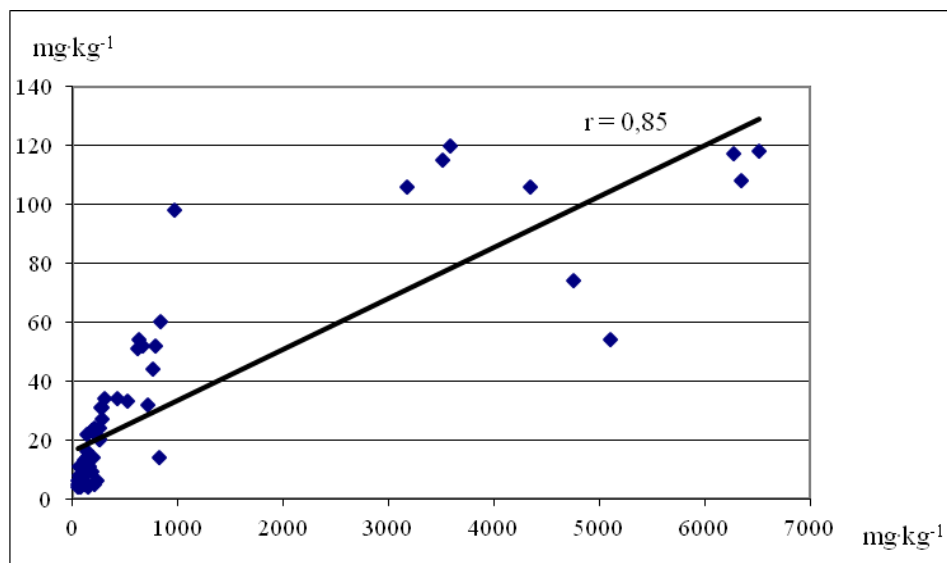


Fig. 3. Correlation between subtotal and 0.1M HCl soluble Ca forms content – experimental field "A"



## 5. DISCUSSION OF RESULTS

Post mining grounds from the Łęknica region contain pyrite ( $\text{FeS}_2$ ) in their composition. Iron sulfide oxidation processes are the cause of the strong ground masses acidity, even to the pH below 3.0 [Krzaklewski et al. 1997, Greinert et al. 2009]. For the neutralization of acidity on the surface of dumps in the Łęknica region lime from "Miasteczko Śląskie" ironworks has been used within a dose of  $50 \text{ Mg} \cdot \text{ha}^{-1}$ . Comparing this fact with the German researches, it was not an extremely high dose. Schaaf and Hüttl [2006] described the use for post-mining grounds neutralization in the former GDR lime in doses up to  $200 \text{ Mg} \cdot \text{ha}^{-1}$ . Applied lime resulted in increased soil reaction. However, this phenomenon was not too long lasting, and after a few years reaction returned to the state before liming [Drab et al. 2005].

The results of this study suggest that lime used to neutralize ground acidity has been systematically leached, and the part of calcium was incorporated into the biological circulation – it was absorbed by vegetation and then returned to the land with drooping needles.

Most of the calcium has been found in the layer of forest litter. The calcium subtotal content corresponds with the results of Gonet et al. [2007] in soils of forest area in Młynarze (Slovakia) and the Rogów, near Warsaw. It is a form of calcium strongly bound by organic matter – the so-called raw-matter. In the layers beneath the forest litter (3-8 and 8-15 cm below the surface), significant calcium decrease was found as compared to forest litter. The content of available calcium in organic soils layers under a pine trees Mätkönen et al. [1999] identified (depending on the fertilization and location) as:  $391\text{-}6929 \text{ mg} \cdot \text{kg}^{-1}$  at pH 3,1-4,5 (measured after fertilization), and from 1915 to  $1997 \text{ mg} \cdot \text{kg}^{-1}$  with pH 3,7-4,3 (measured after 5 years). In this context, the content shown near Łęknica (mean 382, range  $54\text{-}968 \text{ mg} \cdot \text{kg}^{-1}$  with  $\text{pH}_{\text{KCl}}$  3,5-5,0; 22 years after application) has to be assessed as comparable to this described by quoted authors. Uncommon low Ca content in this form has been reported only in the plots A-2 (control + lime) and A-3 (NP).

The layers of 3-15 cm characterised higher percentage of calcium in 0.1 M HCl in a subtotal form. This can be explained by larger amount of organic compounds at higher degree of humification and increasing solubility of most minerals induced typically by podsolization process. Similar conclusions presented Gonet et al. [2007]. About release of Ca from decomposing leaf litter as important Ca source for the forest plants wrote also Dijkstra [2003] and Hobbie et al. [2010]. Dijkstra pointed out, however, that low soil reaction affects slowing down calcium release from the organic matter due to inhibition of microbial decomposition. Featured experiment in Łęknica region seems to confirm this thesis, especially in the context of the low rate of solubility of calcium in soil.

Fahey et al. [1988] pointed out also another source of calcium as an effect of fine roots decomposition. Hobbie et al. [2010] noted a significant correlation between the Ca and K content in soil, and the fine roots development of numerous species of trees. Unfortunately, in the case of toxic acidity of the soil, forest vegetation is growing very poorly, which significantly limits the role of this source of calcium. Such situations were recorded in all plots of the presented experiment.

It was noted, that in the layers below 25 cm from soil surface calcium content is low and corresponds to the content of this element in the raw material before reclamation. Also reaction of these materials did not display any significant response to applied fertilizers. Similar results (pH 3.7-5.5 in layers approximately 20 cm and pH below 2.9 for these lying 100 cm below the surface) are reported by Hüttnl and Schaaf [2006].

## 7. CONCLUSIONS

The obtained results permit the formulation of the following conclusions:

- The subtotal calcium content in forest litter layer was significantly higher than the Ca content in samples taken from deeper layers of the soil profile;
- Subtotal calcium content in samples taken on the limed plots was higher as compared with not limed plots at the beginning of the experiment.
- The content of calcium soluble in 0.1M HCl in post-mining dump grounds follows a similar pattern to subtotal calcium.
- Significant correlation between content of subtotal and acid-soluble form of calcium has been noted in soils under experiment.

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#### ZAWARTOŚĆ FORM WAPNIA W GRUNTACH POKOPALNIANYCH Z REJONU ŁĘKNICY

##### Streszczenie

Praca zawiera wyniki analiz zawartości wapnia ogólnego oraz rozpuszczonego w 0,1M HCl w gruntach pokopalnianych z rejonu Łęknicy. Zawartość wapnia ogólnego w poziomie ściółki leśnej, bez względu na zastosowane w trakcie rekultywacji nawożenie oraz lokalizację badań, była zdecydowanie najwyższa (śr. 4108 mg·kg<sup>-1</sup>). W miarę wzrostu głębokości poboru próbek zawartość wapnia zmniejszała się (do śr. 80 mg·kg<sup>-1</sup> w skale macierzystej). Podobne zależności odnotowano wobec wapnia wyekstrahowanego 0,1M HCl (śr. 382 mg·kg<sup>-1</sup> w ściółce vs. 7 mg·kg<sup>-1</sup> w skale macierzystej). Wystąpiła wysoce istotna korelacja pomiędzy zawartością obu oznaczonych form wapnia w badanych gruntach.