

MICROSTRUCTURES AND SELECTED PROPERTIES OF RECLAIMED ZINC AND LEAD POST-FLOTATION WASTES AND SOIL OF ADJACENT NATURAL ECOSYSTEM

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The studies were conducted on the shelf of the spoil formed from material disposed after zinc and lead ores flotation carried by Mining and Metallurgy Plant «Bolesław» in Bukowno near Olkusz (Southern Poland) and on the adjacent forest soil. Presented studies aimed at analysis of selected properties and microstructure development of dumping ground which underwent reclamation treatments or was formed as a result of spontaneous plant succession, and compared them with microstructures and properties of soil from the adjacent natural forest. Reclamation treatments carried out on the heaps made of post-flotation material resulted in the increase of organic carbon accumulation and in biological activity stimulation in comparison with the properties of the ground covered by spontaneous succession vegetation and lead to the development of microstructures and micromorphometric parameters more similar to these of the adjacent natural forest soil than those observed in non reclaimed stand.

Keywords: zinc and lead ores mining, soil microstructures, reclamation, natural succession.

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МИКРОСТРУКТУРНЫЕ И ПРОЧИЕ СВОЙСТВА ПОЧВ ЕСТЕСТВЕННОЙ ПРИРОДНОЙ ЗОНЫ, А ТАКЖЕ ПОЧВ, ВОССТАНОВЛЕННЫХ ПОСЛЕ ВОЗДЕЙСТВИЯ ОТХОДОВ, СОДЕРЖАЩИХ КОМПОНЕНТЫ СВИНЦА И ЦИНКА

Проведен анализ пластов отвалов, сформированных из продуктов постобработки цинковых и свинцовых руд металлургического завода «Болеслав» города Буковно (Южная Польша). Подобные исследования нацелены на изучение определенных свойств, а также микроструктурных процессов почв в местах сброса отходов, которые в одном случае подвергались мелиорации, а в другом – нет (являющихся прямым результатом техногенных сукцессий). Полученные результаты сравнивались с микроструктурой и свойствами почв естественного леса, находящегося неподалеку. Проведена мелиорация отвалов, возникших в результате чрезмерного накопления органического углерода, а также из-за интенсивной биологической активности. В случае мелиорации микроструктура и микроморфометрические параметры почвы оказались более схожими с теми же характеристиками земель соседней естественной природной зоны, чем с характеристиками участков, не подвергнутых восстановлению.

Ключевые слова: добыча цинковых и свинцовых руд, микроструктура почвы, мелиорация, естественная сукцессии.

In the Olkusz district located on the border of the areas of Kraków-Częstochowa Jura and Silesia Upland (southern Poland) mining and metallurgical engineering of zinc and lead ores have been carried out since the 18-th century and have led to the environmental degradation of these areas. During open-cast zinc and lead ores mining large amounts of spoil material were excavated and deposited in heaps and post-flotation materials were accumulated in settling ponds covering the surface of 100 ha (Grodzińska et al., 2000; Grodzińska, Szarek-Lukaszewska, 2002; Ciarkowska, Gambuś, 2005). Such areas contain high levels of Pb, Zn and Cd compounds and are characterised by disturbed water regimes, high susceptibility to wind erosion, low humus accumulation capacities which together with nutrient deficiency make them an inappropriate substrate for plant growth and development.

Thus, in these sites reconstruction of soil is necessary for ecosystem restoration. (Frouz et al. 2001, Frouz, Novakova 2005, Prach, Pysek 2001). To achieve that target two way treatments were performed on settling ponds formed of post flotation materials. A part of the pond was submitted to land reclamation processes while another part was left for spontaneous succession of vegetation. A criterion commonly used to evaluate restoration successes of such areas is comparing them with nearby undisturbed sites that are representative of pre-disturbance conditions (Mummey et al. 2002).

Biological processes which reflect viability and stability of newly created ecosystems, consisting in processes of decomposition of organic matter and formation of soil structure are best studied in thin sections. The most objective way of evaluating the soil microstructure is a computer-aided image analysis of photographs taken of thin sections (Protz, Vandenbygaart 1998, Ciarkowska, Gambuś 2005).

Presented studies aimed at analysis of selected properties and microstructure development of dumping ground which underwent reclamation treatments or was formed as a result of spontaneous plant succession, and compared them with microstructures and properties of soil from the adjacent natural forest.

STUDY AREA

The studies were conducted in 2007 on the shelf of the spoil formed from material disposed after zinc and lead ores flotation carried by Mining and Metallurgy Plant «Bolesław» in Bukowno near Olkusz (N 50° 16,856' E 19°30,204') and on the adjacent forest soil (N 50° 17,726' E 19°29,150'). For the study, undisturbed and representative ground samples of surface (0–10 cm) and subsurface (10–20 cm) layers were sampled from the three sites. On stands where the substrate samples were taken floristic research was also conducted.

The first site was located on the part of the shelf where reclamation was performed in 2002. The process started with the application of hydroseeding containing sewage sludges with the addition of straw, cotton threads and cellulose and a grass mixture, followed by birch (*Betula pendula* Roth), plum *Prunus* sp. (*Prunus domestica* L.: 'Dąbrowicka purple plum', *Prunus domestica* L. 'Common purple plum', *Prunus cerasus* L. 'Tabel® Edabriz') and yew (*Taxus baccata* L.) trees planting. The second site was situated on shelf covered by the 30 year old vegetation which resulted in the process of natural succession. Grass community containing species characteristic for galmanic flora (*Armeria maritima* (Mill.) Willd. ssp. *halleri* (Wallr.) Á. Löve & D. Löve, *Silene vulgaris* (Moench.) Garcke, *Gypsophila fasigiata* L.), birch (*Betula pendula* Roth) and pine (*Pinus sylvestris* L.) trees amounted to 15 % of the surface. On this site natural revegetation follows a pattern in which several pioneer tree species became established, and the deposition of organic matter has begun on the spoil surface. Revegetation proceeded from the adjacent forest of mixed stand of conifer and deciduous trees (*Pinus sylvestris* L., *Betula pendula* Roth, *Fagus sylvatica* L.) where the third study site was located. The undergrowth of the site was characterized by the presence of *Juniperus communis* L.. In the field layer *Vaccinium myrtillus* L., *Vaccinium vitis-idaea* L., *Deschampsia flexuosa* (L.) Trin., *Deschampsia caespitosa* (L.) P. Beauv., *Calamagrostis arundinacea* (L.) Roth) constituted a notable portion of vegetation and *Pleurosium scheberi* Wildt. was also noticed there.

METHODS

In the representative ground samples the following analysis were performed: the soil reaction by potentiometer in 0,01 mol·dm⁻³ CaCl₂ suspension, the level of total nitrogen, organic and inorganic carbon with the use of TOC–TN 1200 Thermo Euroglas, the total content of Cd, Pb and Zn was determined after soil digestion in the mixture of nitric and perchloric concentrated acids. Contents of the elements in solutions were assessed with the use of the atomic emission spectrometer with inductively coupled argon plasma ICP–AES JY 238 ULTRACE.

Slides were prepared from the ground samples of intact structure and fixed with Araldite epoxy resin in Epovac apparatus. Observations of soil microsections were conducted using Nikon Eclipse E400 POL polarizing microscope in light passing through parallel

nicols, with magnifications of 20× and 40×. Slide images were subjected to computer morphometric analysis using Aphelion ADCIS S.A., Aai inc programme. The results were statistically reworked with the use of STATISTICA 7.1, ANOVA analysis and a posteriori Fisher's test was used to study differences between respective variants at the significance level $\alpha=0,05$.

RESULTS

Selected physico-chemical and chemical properties of studied stands are shown in Table 1. Ground samples taken from both sites situated on the shelf of heap formed from post-flotation materials, the reclaimed site (stand I, samples 1, 2) and the site with natural succession (stand II, samples 3, 4) revealed alkaline reaction with *pH* values higher than 7,7 in surface and subsurface horizons together with high content of inorganic carbon. The fact resulted from the numerous dolomite fragments occurring in the substrate as a waste rock accompanying zinc and lead ores. Only the soil of the adjacent natural forest (stand III, samples 5, 6) had slightly acid reaction caused by the impact of acid bedrock sands and the influence of conifer trees. In the soil under the natural forest the highest level of organic carbon was accumulated which amounted to 24,8 g·kg⁻¹ and, what is typical for forest soil, decreased rapidly to 2,2 g·kg⁻¹ in subsurface layer. Grounds of both sites situated on post flotation material heaps were characterised by considerably lower amounts of organic carbon (varied within the range from 1,4 to 2,0 g·kg⁻¹) which did not differ much between surface and subsurface layers. In the reclaimed ground a higher level of organic carbon was stored than in the ground left for natural plant succession mainly on the account of the carbon supplied to the ground together with sewage sludges. The substrate of that site was characterised by the lowest nitrogen content of all studied sites, and in consequence the highest C:N ratio, which were effects of straw, cellulose and cotton threads introduced into the ground during reclamation processes.

Table 1

Physico-chemical and chemical properties of studied grounds (mean values)

Specification	Site number/depth in cm					
	I/0-10	I/10-20	II/0-10	II/10-20	III/0-10	III/10-20
Number of sample	1	2	3	4	5	6
Organic C content, g·kg ⁻¹	1,81	2,02	1,43	1,62	24,82	2,2
Total N content, g·kg ⁻¹	0,10	0,08	0,16	0,18	2,21	0,2
Inorganic C content, g·kg ⁻¹	1,92	1,85	2,18	1,97	0,6	0,5
C _{org} :N ratio	18,1	25,3	8,9	9,0	11,2	12,3
pH	8,0	7,8	8,4	7,7	5,6	5,6
Cd content, mg·kg ⁻¹	66,21	85,58	23,41	18,0	7,87	0,5
Pb content, mg·kg ⁻¹	2877,6	3902,0	958,7	774,7	407,5	19,4
Zn content, mg·kg ⁻¹	7196,0	9277,4	4050,6	2481,6	388,7	23,0

Studied substrates coming from the heaps were characterized by a very strong contamination with cadmium, lead and zinc which exceeded limiting levels, defined by the Ministry of the Environment in the directive for industrial lands (Law Gazette 02.165.1359). In both sites higher amounts of the three metals were determined in subsurface layers than in surface ones. The sites were considerably diversified considering contents of the studied heavy metals, which were about 3 times for cadmium and 2 times for *Pb* and *Zn* higher in the reclaimed ground (stand I, sample 1, 2) than in stand II (sample 3, 4) left for spontaneous succession. The fact can be explained by two reasons. Firstly, much longer time passed from the moment of storage of post-flotation material in case of the site with natural succession (30 years) than in case of the reclaimed ground (5 years). Secondly,

the occurrence of specific galmanic flora species growing as the the result of spontaneous succession acted as the phytoextractors of heavy metals. Only in soil overgrown with natural forest (stand III, samples 5, 6) the limiting levels of heavy metals were not exceeded but in case of this soil higher amounts were determined in surface layers than in subsurface, which indicates the input of heavy metals with the atmospheric dust fall.

The results of micromorphological studies and micromorphometric analysis demonstrated differences in structure, voids, the form and content of organic matter among the studied stands (Table 2, 3). In all stands, but for the surface layer of soil from natural forest, the biggest part of image was occupied by voids ranging from 60,9 % (sample 2) to 81,4 %, in sample 3, of the total image area. Depending on of the microstructure and the presence of organic aggregates the types of voids changed from simple packing to complex

Table 2

Results of micromorphological studies (dominant soil features observed in thin sections)

No. of sample	Form of organic matter	Mineral parts	Microstructure	Type of voids
1	organ residues, aggregates, rare macrofauna fecal pellets	dolomite, quartz, iron sulphur, marcasite	intergrain micro-aggregates, few crumbs*	complex packing, rare compound packing**
2	few aggregates, rare fecal pellets, organic punctuations	metal ores, dolomite, quartz, iron sulphur, marcasite	single grains, few intergrain micro-aggregates	simple packing, complex packing
3	red-brown hyphes, few small aggregates, small fecal pellets, organic punctuations	dolomite, quartz, iron sulphur, marcasite	single grains, few intergrain micro-aggregates	simple packing, few complex packing
4	red-brown hyphes, organic punctuations	metal ores, dolomite, quartz, iron sulphur, marcasite	single grains, bridged grains	simple packing
5	yellow organ residues, roots, red-brown hyphes, fecal pellets, earthworm casts organic punctuations	few quartz	crumb aggregates	compound packing, channels
6	few dark brown organ residues, brown hyphes, organic punctuations	quartz	single grains, pellicular grains	simple packing

*Intergrain micro-aggregates are fine material micro-aggregates between the sand grains; crumbs are formed from aggregate material with a crumbly shape; bridged grains are sand grains joined by bridges of fine organic material, pellicular grains are sand grains covered with a pellicle of fine organic material;

**Complex packing are the voids between single grains and between the aggregates, simple packing voids are between skeleton grains, compound packing voids are the result of packing the aggregates.

and compound packing voids (Table 2). Simple packing voids were characteristic of subsurface layers of all studied sites, but in surface layers the complication of void organization ranged from single packing with few complex packing voids in sample 4, through complex packing with few compound packing voids in sample 1, to compound packing voids in sample 5. In stand II (samples 1, 2) large voids with diameter bigger than 200 μm prevailed while the amount of small (till 50 μm) and medium (50–200 μm) sized voids were low and the differences were statistically significant (Table 3A). In both layers of the ground of the stand II (samples 3, 4) medium voids dominated, which indicated better water retention capacities of the ground of this site when compared with the ground of the stand I and even with natural stand III. As far as shape indexes of voids are concerned there are not significant differences between stand I (samples 1, 2) and II (samples 3, 4), and only

voids in soils of natural forest (samples 5, 6) have less regular shapes as they are mostly located among aggregates or undecomposed residues of plants and not among mineral grains as it is in case of the other two sites. The second component when considering the

Table 3

Results of micromorphometric analysis: A, B, C, D

A) Selected characteristics of shapes and measures of soil voids

No. of soil	Mean indexes of void shapes with dia. in μm						% share of voids with dia. in μm		
	to 50	50-200	over 200	to 50	50-200	over 200	to 50	50-200	over 200
	circularity			compactness					
1	0,14 ^{a*}	0,46 ^b	0,24 ^{ab}	0,14 ^a	0,37 ^{ab}	0,20 ^a	0,05 ^a	10,8 ^a	89,2 ^d
2	0,16 ^a	0,42 ^{ab}	0,33 ^{ab}	0,16 ^a	0,40 ^b	0,29 ^{ab}	0,07 ^a	37,8 ^{bc}	62,2 ^{abc}
3	0,13 ^a	0,44 ^{ab}	0,40 ^b	0,11 ^a	0,41 ^b	0,36 ^b	0,01 ^a	58,6 ^c	41,4 ^a
4	0,32 ^b	0,46 ^b	0,39 ^b	0,32 ^b	0,41 ^b	0,33 ^{ab}	0,29 ^a	50,2 ^c	49,5 ^{ab}
5	0,15 ^a	0,37 ^a	0,22 ^a	0,12 ^a	0,34 ^a	0,21 ^a	1,18 ^b	28,4 ^{ab}	70,5 ^{bcd}
6	0,13 ^a	0,44 ^{ab}	0,32 ^{ab}	0,11 ^a	0,40 ^b	0,28 ^{ab}	0,01 ^a	26,7 ^{ab}	73,3 ^{cd}

B) Selected characteristics of shapes and measures of decomposed organic components

No. of soil	Mean indexes of shapes of organic components with dia. in μm						% share of organic components with dia. in μm		
	to 50	50-200	over 200	to 50	50-200	over 200	to 50	50-200	over 200
	circularity			compactness					
1	0,76 ^b	0,66 ^c	0,47 ^c	0,60 ^c	0,52 ^c	0,37 ^c	2,3 ^a	34,1 ^b	63,6 ^{ab}
2	0,56 ^a	0,32 ^a	0,31 ^b	0,44 ^{ab}	0,25 ^a	0,25 ^b	3,9 ^a	27,7 ^{ab}	68,5 ^{ab}
3	0,55 ^a	0,38 ^{ab}	0,38 ^c	0,43 ^a	0,30 ^{ab}	0,31 ^{bc}	11,3 ^b	33,0 ^b	55,7 ^a
4	0,66 ^{ab}	0,47 ^b	0,46 ^c	0,52 ^{bc}	0,38 ^b	0,37 ^c	6,1 ^{ab}	35,2 ^b	58,7 ^a
5	0,63 ^a	0,36 ^{ab}	0,12 ^a	0,50 ^{ab}	0,29 ^{ab}	0,09 ^a	2,7 ^a	9,9 ^a	87,5 ^b
6	0,64 ^a	0,43 ^{ab}	0,27 ^b	0,51 ^{ab}	0,34 ^{ab}	0,22 ^b	9,0 ^{ab}	32,8 ^b	58,2 ^a

C) % share of the component area in the total component area

No. of soil	Undecomposed organic matter with dia. in μm			Mineral parts with dia. in μm		
	to 50	50-200	over 200	to 50	50-200	over 200
1	33,3 ^b	66,7 ^c	0,0 ^a	2,6 ^a	55,8 ^b	41,6 ^b
2	0,0 ^a	0,0 ^a	0,0 ^a	10,8 ^b	39,3 ^a	50,0 ^b
3	100,0 ^c	0,0 ^a	0,0 ^a	13,7 ^b	55,3 ^b	31,0 ^{ab}
4	100,0 ^c	0,0 ^a	0,0 ^a	8,7 ^{ab}	58,5 ^b	32,8 ^{ab}
5	14,3 ^{ab}	37,0 ^b	48,7 ^b	34,4 ^c	61,5 ^b	4,1 ^a
6	42,5 ^b	57,5 ^{bc}	0,0 ^a	12,4 ^b	61,6 ^b	26,1 ^{ab}

D) % share of studied components in the total image area

No. of soil	Decomposed organic matter	Undecomposed organic matter	Mineral parts	Soil voids
1	15,0 ^{ab}	0,02 ^a	7,7 ^b	77,3 ^{cd}
2	29,2 ^c	0,0 ^a	9,8 ^{bc}	60,9 ^b
3	8,2 ^a	0,01 ^a	10,4 ^{bc}	81,4 ^d
4	9,3 ^a	0,01 ^a	11,3 ^c	79,5 ^{cd}
5	43,5 ^d	23,61 ^b	3,1 ^a	29,8 ^a
6	17,2 ^b	0,16 ^a	11,8 ^c	70,9 ^c

*The same letters indicate a lack of statistically significant differences among mean values; circularity – 1 for a circle and smaller for less circular objects, compactness – 1 for the perfect square and smaller for shapes with irregular boundaries.

area occupied in the total image was in most cases decomposed organic matter which covered from 8,2 % (sample 3, stand II) to 43,5 % (sample 5, stand III) of the total image area. The site covered with plant resulting from natural succession was characterised by the low area covered with decomposed organic matter in both layers. In this site organic matter occurred mainly in the form of small aggregates and punctuations and micromorphological observations were conformed by micromorphometric analysis showing that in this site the lowest percentage of the largest aggregates from all sites and the highest percentage of the smallest aggregates were counted (Table 2, 3B). The fine excrements originated from soil mesofauna such as springtails oribatid mites and enchytraeid worms while the coarse organo-mineral excrements indicated macrofauna excrements in the studied soil layers. The oribatid mites and enchytraeids pellets were observed in lower amounts in all studied grounds as they are supposed to be more sensitive to heavy metal pollution than population of springtails which was not easily affected by high heavy metal concentrations (Gillet, Ponge 2002).

In both layers of the reclaimed stand there was a slightly larger area occupied by the biggest aggregates and similar areas are occupied by medium size aggregates but a significantly smaller area is occupied by small aggregates. That fact together with the observed intergrain microstructure demonstrated more developed forms of the accumulated organic matter, which is probably due to beneficial effect of sewage sludges applied to the ground which created in such a short time sufficiently suitable conditions for the development of biological life entailing in the formation of stable aggregates of macrofauna origin. The most pronounced microstructure can be observed in soil under natural forest (stand III, sample 5) where in surface horizon crumb microstructure is formed, and big aggregates of irregular shapes prevail, left by mesofauna and macrofauna representatives. In this natural forest stand a high amount of undecomposed organic matter in the form of plant residues is also accumulated, which is typical for forest soil upper horizon (Table 2). Grounds of all studied sites contained similar areas occupied by mineral parts, most of them were medium sized (50–200 μm – Table 3C).

DISCUSSION

Mining of zinc and lead ores results in severe disturbance of large land areas and spoil material is accumulated in heaps which contain toxic level of heavy metals. Thus, the reclamation of such lands creates serious problems from both the ecological and economic point of view (Prach, Pysek 2001, Frouz, Novakova 2005, Frouz et al. 2006, Mummey et al. 2002). At post-mining sites, various reclamation technologies are applied to accelerate ecosystem development and soil formation. In some sites soil reestablishment is accelerated by the spread of topsoil. In other sites, including the sites where this study was conducted, no top soil is added and soil develops directly from the raw spoil material. In such cases the application of hydroseeding containing sewage sludges rich in organic matter and other nutrients together with grass mixture plays a crucial role in the process of the organic matter accumulation and stabilization of plant species introduced in the process of ground reclamation (Bradshaw 1997).

Recently spontaneous succession of vegetation receives increasing attention in various restoration projects as it represents an interesting object of ecological studies for it increases biodiversity and enhances wildlife and natural values (Frouz et al. 2006, Grodzinska et al. 2000, Frouz, Novakova 2005, Prach, Pysek 2001). Nevertheless, in the sites left for the spontaneous succession the processes of soil formation usually proceed slowly, with the amount of stored organic matter increasing gradually with time, and newly formed soil microstructure characteristics are often worse than in the reclaimed grounds (Frouz et al. 2006). Bradshaw (1997) suggests that natural succession can eventually restore even heavily degraded mineland but it often takes 40–100 years if unaided, the biggest problem being immigration of species.

In the presented study we compared some chemical properties and microstructures of the ground of the reclaimed site (5-years after reclamation) with the unreclaimed site overgrown with plants (mainly metallophytes) originating from natural 30-year succession us-

ing the adjacent, natural mixed forest site as a benchmark for those properties. The findings indicated that the properties of the ground formed as a result of reclamation such as organic carbon accumulation, aggregation and microstructure were better developed and more similar to those observed in soil overgrown with natural forest than in the stand left for natural succession, which is consistent with results obtained by Frouz et al. (2005, 2006) and Prach, Pysek (2001). However, we observed rather little evidence of animal activity such as the accumulation of fecal material in both the reclaimed and the non reclaimed grounds in comparison with the natural forest site. It may be explained by a toxic effect of heavy metals and poor water holding capacities of the sites situated on spoil material heaps. Toxic metals exert a selective pressure on soil organisms, reducing the diversity of the functional groups when a resilience threshold is reached. Some animal species can adapt, becoming more resistant to heavy metals but there are limits to this process (Balabane et al. 1999, Gillet, Ponge 2002). Nevertheless, the lack of undecomposed organic matter observed in the present study indicates that either adapted or tolerant fauna and microflora insure some level of biological activity even in highly polluted substrata or alkaline reaction of the studied grounds promoted retrogradation processes of heavy metals and their turn into insoluble thus, intoxic forms.

In the unreclaimed site we observed a beneficial effect of vegetation cover mainly in the form of metallophyte grasslands which consisted in the decrease of heavy metal concentration in comparison with the reclaimed site, and a higher porosity with the bigger share of small voids. The latter properties were the result of a major presence of small aggregates originating from the activity of mesofauna. These characteristics indicate better water holding capacities of this site when compared not only with the reclaimed one but also with soil under natural forest. According to these findings the site covered with more diverse plant communities as a result of spontaneous succession will be more resistant to drought, which proves the ecosystem stability.

CONCLUSIONS

1. Reclamation treatments carried out on the heaps made of post-flotation material resulted in the increase of organic carbon accumulation and in biological activity stimulation in comparison with the properties of the ground covered by spontaneous succession vegetation
2. Microstructures and micromorphometric parameters of the reclaimed stands were more similar to these of the adjacent natural forest soil than those observed in non reclaimed stand
3. Grass vegetation and metallophytes communities overgrowing the spoil heap as a result of natural succession contributed to the decrease of the heavy metal level and the increase of water retention capacities of the substrate, which indicates that the stable and resilient ecosystem is gradually created.

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