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NFM

Remediation in conditions of an operating copper-nickel plant: results of perennial experiment

UDC 504.064+669.3+669.24

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The paper presents the results of a long-term experiment on remediation of depressed territories (technogenic barrens) in Subarctic (the Kola Peninsula, Russia). The main negative factors, hampering processes of natural restorative succession of such territories, are the inclement environmental conditions and permanent aerotechnogenic exposure, which leads to high acidity level and high concentrations of metals in atmospheric precipitation and soil solutions.

An innovative “mild” remediation technique of soils in an impact zone of a copper-nickel plant, using different types of mining wastes, has been put forward. It has been shown that these wastes serve as effective ameliorants due to high content of calcium and magnesium carbonates and/or silicates and have a prolonged beneficial effect on nutrient regime of soils. The vegetable cover of grain crops established on the above mentioned wastes without resorting to soil and peat, scarce in the region, has been proven to be productive and resistant to aerotechnogenic burden.

In order to estimate favorability of technosoil during the process of monitoring research of artificial phytocenosis evolution, there has been suggested an integral index — module of toxicity, defined as a ratio of phytoavailable copper and nickel aggregate content to calcium and magnesium, expressed in mole units. It has been found that sungulite wastes of phlogopite mining (city of Kovdor, Murmansk region) appear to be a sort of mining wastes which have considerable promise with relation to effectiveness of land-improvement and economic expediency of their use in large-scale work on remediation of depressed territories in subarctic conditions.

Key words: remediation, grass, industrial barren, mining waste, Subarctic, copper-nickel plant, aerotechnogenic exposure, impact zone, ameliorant, pollutant, state of ecosystem, toxicity module.

DOI: *10.17580/nfm.2017.02.04*

Introduction

Operating enterprises of non-ferrous metallurgy exert an adverse effect on environment mainly by pollutant emission into atmosphere with their consequent falling over adjacent territories, which leads to pollution of all vital environments and degradation of ecosystems [1, 2]. In the impact zones of large-scale enterprises, technogenic barrens may arise. These are the open landscapes with contaminated and eroded soils, completely or almost completely (>90%) lacking in vegetation [2]. Arising of 36 technogenic barrens were observed in the world, most of which are situated in northern hemisphere (Central Europe, USA and Russia) [2, 3].

At present, the plant biomass in an impact zone of the copper-nickel plant near the city of Monchegorsk (Murmansk region, Russia) forms 1% of biomass of uncontaminated forests, which is connected with emissions of heavy metals and sulfur dioxide [3]. Conflagrations, disafforestations, harmful activity of destructive insects, changes in moisture nature of the territory introduce an additional contribution into digression of ecosystems [3, 4].

The limiting factors of recovering the growth on technogenic barrens of the Kola Peninsula are such soil parameters as extreme copper and nickel concentrations, shortage of nutrient elements (K, Ca, Mg, P, Mn), high acidity, low moisture as well as scanty seed bank and lack of plant renewal underground organs [4, 5]. Progressive successions in the impact zone on this territory are impossible without human intervention even after termination of the plant operation [6]. At present, as aerotechnogenic emissions of the Kola Mining and Metallurgical company JSC are decreasing in comparison with the level of 1990-ies, secondary pollution owing to erosion of soils and migration of heavy metals have a pronounced effect on ecological situation [7, 8]. In this connection, establishment of stable vegetation cover in combination with stabilization of heavy metals is the main objective of the plant's impact zone remediation.

Establishment of high-quality artificial phytocenosis in unfavourable climate environment of the Kola Peninsula is connected with the number of problems, such as short vegetative period, low spring-and-fall temperatures, frequent and precipitation, strong winds, deficiency and poorness of the soil resources, lack of peatery as a source of fill-up soil in the region, labour-intensiveness and expensiveness of measures intended to increasing the fertility of local soils with initially low nutrient status [9].

For vast territories, a "mild" remediation technique in situ by way of applying ameliorants, able to stabilize pollutants and to serve as a source of nutrients has been accepted as one of the promising and economically sound technologies of restoring the soils, being polluted in consequence of the functioning of enterprises of non-ferrous metallurgy [9, 10].

Experimental work on remediating the territory of technogenic barren has been earlier implemented by the staff of the Institute of North Industrial Ecology Problems, Kola Science Center, Russian Academy of Sciences. The remediation has involved utilization of displaced fertile stratum of soil, lime or dolomite with planting of deciduous and needle-leaved trees and sowing of perennial grasses [11, 12]. Subsequent planting monitoring has revealed that notwithstanding liming, soils have been characterized by high acidity and has been short of some macro- and microelements [13].

In 2008, group of researches of institutes of the Kola Science Center has implemented an experiment on phytoremediation with use of roll grass-plot, grown hydroponic vermiculite substrate. Such a way was efficient on plots of a tailing pit of apatite-nepheline concentrating mill [14], but in conditions of a technogenic barren, the vegetation cover falling out has taken place within 1–2 vegetation seasons [15]. In 2010, an approbation of mining wastes as land-improvement materials has started in the field experiments on soil remediation in impact zones of the Kola Mining and Metallurgical Company JSC. The Kola Peninsula possesses the highly developed mining complex, in which connection there are placed a lot of wastes of the ore mineral resources extraction and processing. Use of these wastes in nature-conservative technologies assists with their utilization and thereby with lowering the technogenic burden in the places of storage. To establish vegetation cover, the cereals has been used due to their high adaptability and effectiveness for creating a primary plant community [16, 17].

The work has targeted on initialization of restoration processes on ecosystem level by means of soil remediation on technogenic barrens with a simultaneous formation of a primary plant community.

Research objects and procedure

Site description. Remediation has been implemented on the plot of technogenic barren near the city of Monchegorsk (Murmansk region, Russian Federation). Experimental plot is located at a distance of 0.7 kilometres from the border line of industrial zone of the Kola Mining and Metallurgical Company JSC, Monchegorsk site (67°55.783 'N, 32°51.535' E). Annual total pollutant emissions of the enterprise in 2011–2016 have changed slightly and have amounted to 37–43 thousand tons [18].

The plot is depressed and represents a "drained" low-moor cotton grass bog [19]. The soil is a peat eutrophic one [20]. The native vegetation on the plot has been absent; turf horizon ("tow") has been absent as well. A pH value of an aqueous extract of upper 5-cm soil layer has come to 3.6–4.0, ash content was 24%, and total carbon content was 30%.

Experimental design. A plot with plane surface and homogeneous top-soil has been chosen for experiment. Square of each replication was 1 m^2; distance between areas was 0.5 m.

The cereal plants *Agropyron intermedium* (Host.) Beauv., *Festuca rubra* L., *Lolium perenne* L., *Phleum pratense* L. mixed in the ratio 1:1:2:2 by mass has been used for the vegetation cover growing.

Establishment of vegetation cover has been implemented in three series: 1) sowing the seeds directly to peat soil; 2) sowing the seeds to a vermiculite substratum layer 1 cm thick; 3) sowing the seeds to a vermiculite substratum layer 1 cm thick, being deposited onto the surface of a mineral material layer 5 cm thick.

Hydroponic vermiculite substratum is a patented material obtained of vermiculite [21]. It ensures high pace of development and production of cereal plants early in the ontogenesis, which is important in conditions of a short vegetation period in Subarctic [22]. Every year, combined NPK-fertilizer has been applyed once in a vegetative season on the basis of 60 $g \cdot m^{-2}$.

Substratum of technogenic surface formations, incorporated into the group of naturfabricats, the sub-group of lithostrats (serpentine-magnesit, sungulite) and the group of artifabricats, the sub-group of artiindustrats (carbonatite, sungulite and phlogopite wastes) [20, 23] have been used as mineral materials. Ameliorants have been chosen by criterion of presence in their content the minerals, able to actively interact with acid liquors. Among these minerals are calcium and magnesium carbonates and magnesium hydrosilicates, namely calcite, magnesite, dolomite, serpentine minerals, vermiculite, phlogopite. Sand has been used as a reference mineral material. Variant details of the 3-rd series of the experiment are represented in Table 1.

Plants in series 1 and 2 either didn't grow through or have perished during the first vegetation season (2011); the projective cover on corresponding test areas in 2016 has been 0%. In this connection, the present paper concerns only the results obtained in variants of the series 3.

Sampling design. Sampling of revegetative layer has been implemented by monolith method of 10×10 cm size at the depth of 10 cm; the monolith has comprised plants, mineral material and a layer of peat soil under it [24]. Values of pH and concentrations of chemical elements in soil and mineral materials has been determined in two layers (the mineral material and the soil ones) of monoliths, picked from each area. The monoliths have been picked out in June and July of 2016. The biomass of the abovegound organs has been determined by hay-crop of a quarter of each test area with consequent drying up to air-dry condition. Sampling with a view to determine production (mass of the aboveground organs) and to measure height of the plants has been carried out on June 5, July 27 and September 22, 2016.

Chemical analysis. Chemical analysis of the soil and vegetable specimens has been fulfilled in a specialized laboratory of the Institute of Chemistry and Technology of Rare Elements and Mineral Raw Materials, Kola Science Center, Russian Academy of Sciences (Apatity, Russia) under the supervision of S. V. Drogobuzhskaya, Senior Researcher, Candidate of Engineering Sciences. The bulk content of components in the plants has been determined in DAK 100 autoclaves (Berghof, Germany) after autoclave microwave decomposition in a SW4 system. The detection of available forms of elements in the soil and ameloirants has been implemented after exhaustion by ammonium-acetate buffer solution (pH = 4.65) [25]. Obtained solutions have been analyzed by method of mass-spectrometry with inductively coupled plasma on ELAN 9000 DRC-e (Perkin Elmer, USA), pH — by potentiometric measurements at a soil:solution ratio of 1:10 [26] for mineral assays and of 1:25 for the peat ones.

Data processing and statistical analysis. Simple mean and error in mean have been calculated for each version of experiment. Number of sizing the biomass of plants, content of chemical elements and pH value correspond to the number of replications (Table 1), for the height of aboveground organs of the plants $n = 15–45$. Chemical composition of the plants in the versions of experiment has been defined for an averaged sample. Statistical analysis was performed in Excel 2016 and One-way ANOVA with post-hoc Tukey HSD Test. The data sets were found to be significantly different when the probability value was <0.05.

Results and discussion

Description of mineral and soil complexes. Mining wastes used for establishment of recultivating cover differ by content of phytoavailable Ca and Mg. Content of phytoavailable calcium in mineral materials (without taking reference experiment with sand into consideration) diminishes in the following raw: CW >> PW ~ SW > > SG > SM, content of phytoavailable magnesium diminishes in the order: SG > SM > SW > CW > PW (Table 2). These elements are spent both in the process of migration into an underlying layer of peat soil and during formation of aboveground and underground organs of plants.

The most significant increase of calcium in peat under the layer of mineral materials is registered for a version with CW, and magnesium — for a version with SG. Maximal calcium concentration in the upper peat layer are observed in a variant with CW of 19 $g·kg^{-1}$, magnesium — in a variant with SG of 5.5 $g·kg^{-1}$, which is a sequent of the highest content of these elements in the mentioned substrates. Minimal content of the macrocomponents in question is observed under QS: 0.7 g/kg of calcium and 0.3 of magnesium. The peat under SW has contained 3 $g·kg^{-1}$ of calcium and 2.8 $g·kg^{-1}$ of magnesium. This proves that mining wastes have a pronounced land-improvement effect. Correlation analysis has revealed close interrelation between an aggregate content of macrocomponents (Ca + Mg) in a fill-up layer and the peat layer ($R = 0.96$). Molar Ca/Mg ratio in the peat changes in accordance with the content of mobile macrocomponents in substrates; the highest values of this parameter are noted in the version with CW.

In the period from June to July, lowering of calcium and magnesium content in CW and SW is being observed, while in SM content of bioavailable macrocomponents doesn't lessen, which testifies quick filling up a macrocomponent pool in an SM layer as a result of dilution of its mineral phases [27].

In accordance with the migration of macrocomponents process rate, an actual acidity of the contaminated peat is changing. Values of pH of peat under QS and PW has amounted to 4.9–5.0 at pH of substrates of 5.5 (QS) and 7.8 (PW). Other

Table 1

Description of variants of experiment with mineral materials

Designation	Name	Origin	Mineral availability	Repeat count *n*	Year of experiment start
CW	Carbonatite waste	Wastes of the ore secondary processing, Kovdor Mining and Processing Plant JSC, Murmansk region	Calcite	8	2011
SM	Serpentinite-magnesite	Overburden rock, Khalilovskoye magnesite deposit, Orenburg region	Chrysotile, magnesite, dolomite	9	2011
SG	Sungulite	Surrounding rock, Khabozero olivinite deposit, Murmansk region	Lizardite, chrysotile, calcite, dolomite	2	2011
SW	Sungulite waste	Overburden rocks, phlogopite deposit, Kovdor, Murmansk region	Lizardite, calcite, vermiculite	3	2013
PW	Phlogopite waste	Wastes of phlogopite processing, Kovdor, Murmansk region	Phlogopite, calcite	6	2013
QS	Quarry sand	Monchegorsk, Murmansk region	Antacid minerals are absent	8	2011

types of substrates have had more appreciable alkalization effect. Values of pH of CW, SM, SG, SW materials lays in an alkalescent range (8.0–8.5), and pH value of peat under them has amounted to 6.1 for SW, 6.3 for CW, 6.6 for SM and 7.3 for SG. Thus, the most efficacious sort of mining wastes with relation to increasing pH values of peat is SG material.

In the variant using SG there has been also observed the highest concentrations of mobile copper and nickel in the peat layer, contacting with substrate. Content of mobile form of metals in the peat layer is for certain lower under sand (40 mg·kg^{-1} of Ni and 970 mg·kg^{-1} of Cu) in comparison with the other variants (110–390 mg·kg^{-1} of Ni and 2080–3410 mg·kg^{-1} of Cu), in other words, all types of substrates with the exception of the reference one (sand) promote accumulation of pollutants in the upper soil horizon and decrease of their radial migration.

Molar Cu/Ni ratio reflects an influence of physico-chemical environmental conditions of the soil layer on immobilization of metals. Both metals are fastened on alkaline barriers, however nickel in contrast to copper is referred to elements-silicatophils, which are included into the lattice of laminated silicates [28]. The serpentine minerals are also referred to such silicates [29–32]. Molar Cu/Ni ratio has changed from largest to smallest value in the following order: QS > CW ~ PW > SW ~ SM > SG. Analisys of Cu/Ni changes depending on substrate type shows that presence of serpentine minerals in the mineral layer assists to nickel accumulation.

Since the vegetation and microorganism condition in the impact zone of the copper-nickel plant are predominantly influenced by such factors as content of macroelements and heavy metals in soil, there have been suggested criteria for the toxicity level evaluation in the form of a ratio of aggregated content of calcium and magnesium exchange forms to copper or nickel content [33].

Table 2

Chemical characterization of mineral and soil complex of recultivating cover

Sample features				pH_{H_2O}	Acetate-ammonium extractable, mg·kg^{-1}				Ratio, mol/mol		Mt$^{3)}$
Variant	Month	Layer$^{1)}$			Mg	Ca	Ni	Cu	Ca/Mg	Cu/Ni	
CW	June	0–5	M$^{2)}$	8.5	1545	61080	8	22	24	2.4	0.03
			SE$^{4)}$	8.2–8.8	97	4565	1	7	–	–	–
		5–10	M	6.3	932	18621	112	2624	12	22	8
			SE	6.1–6.6	70	5342	28	240	–	–	–
	July	0–5	M	8.1	819	37852	9	57	28	5.7	0.11
			SE	7.8–8.3	26	2010	3	22	–	–	–
SM	June	0–5	M	8.1	7069	1583	35	18	0.1	0.5	0.26
			SE	8.0–8.2	1038	36	8	8	–	–	–
		5–10	M	6.6	3035	2450	205	2083	0.5	9.4	19
			SE	6.2–6.9	272	194	80	176	–	–	–
	July	0–5	M	7.8	7192	1858	41	26	0.2	0.6	0.32
			SE	7.6–8.0	1461	332	9	7	–	–	–
SG	June	0–5	M	8.2	21322	5467	22	5	0.2	0.2	0.05
			SE	–	3198	820	3	1	–	–	–
		5–10	M	7.3	5453	2217	338	3414	0.2	9.3	20
			SE	–	818	333	51	512	–	–	–
SW	June	0–5	M	8.5	3997	8746	20	13	1.3	0.6	0.14
			SE	8.4–8.6	102	100	0	2	–	–	–
		5–10	M	6.1	2846	3032	201	2430	0.6	11	21
			SE	5.9–6.3	193	375	82	77	–	–	–
	July	0–5	M	8.1	3020	6129	24	56	1.2	2.2	0.45
			SE	8.0–8.1	126	71	3	14	–	–	–
PW	June	0–5	M	7.8	524	9234	62	238	10	3.6	1.9
			SE	–	79	1385	9	36	–	–	–
		5–10	M	4.9	336	1885	162	3062	3.4	18	81
			SE	–	50	283	24	459	–	–	–
QS	June	0–5	M	5.5	58	187	7	69	2.0	9.5	17
			SE	5.3–5.8	7	38	1	16	–	–	–
		5–10	M	5	114	692	41	968	3.7	22	70
			SE	4.8–5.3	30	131	7	140	–	–	–
	July	0–5	M	5.5	177	299	9	82	1.0	8.8	10
			SE	5.4–5.8	10	8	0	4	–	–	–

Notice 1. Layer 0–5 corresponds to the layer of mineral substrates, layer 5–10 — to the upper layer of peat soil.

Notice 2. M — simple mean, SE — error in mean.

Notice 3. Module of toxicity was calculated as (Cu + Ni)·100/(Ca + Mg) (mol/mol).

Notice 4. In case of pH, range of values is pointed out.

In order to estimate favourability of the substrates when monitoring experiments on remediation of the technogenic barren soil, we have modified the mentioned criterion into the toxicity module, which is defined as a ratio of aggregated content of copper and nickel to calcium and magnesium in the form available for plants and expressed in molar units: Mт = (Ni + Cu)·100/(Ca + + Mg). For the convenience of data analysis, the sum of copper and nickel concentrations is increased 100-fold. It has been earlier shown that a hundredfold exceeding of the macrocomponents content in reference to metals (Mт = 1) neutralizes their biological impact on plants [34].

It has been found that the sand toxicity module (checkup) exceeds the index under review for material out of mining wastes by 2—3 orders (Table 2). Migration of components from the mineral layer into underlying peat leads to lessening of its Mт in all variants, with the exception of sand. Relying on the data on Mт one may ascertain that mining wastes have improved agrochemical peat behaviour, in such a way producing land-reclamation effect. This thesis is confirmed by mass penetration of the plant roots into technogenic peat in versions of experiment with the use of CW, SM, SG and SW. At the same time, it has been established that PW has had no land-reclamation effect in consequence of relatively low migration rate of macrocomponents as well as presence of high content of pollutants in peat under substrate.

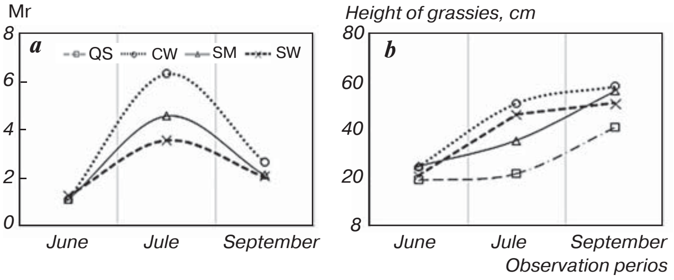

Fig. 1. The change in Mr index (ratio of biomasses of plants on mineral materials and on sand) (*a*) and height (*b*) of the aboveground part of plants during the vegetation period

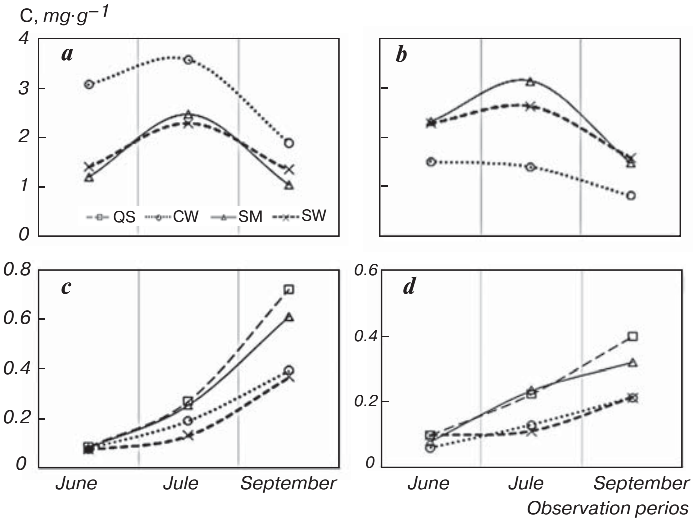

Fig. 2. The change of the calcium (*a*), magnesium (*b*), copper (*c*) and nickel (*d*) content in the aboveground part of plants (C, mg·g^{-1})

Evaluation of the vegetation cover condition

Analysis of vegetation cover has showed that state of phytocenosis, being formed during three (SW, PW) and five years (QS, CW, SM, SG), may be described as very good (in variants with the use of CW, SM, SG), good (SW, PW) and poor (QS). In all experimental variants, the projective cover of vegetation cover has amounted to 100%, while it had not exceeded 70% on sand. The structure and assortment of plant communities, being formed on mining wastes, have been gradually become complicated due to natural colonization of pioneer vegetation with establishment of population communities, characterized by intensive growth and development, powerful rootage, high production, which are a forage reserve or an ecotope for insects, mammals and other living organisms. Hense, they can be described as an environmentally stable primary meadow community.

To give prove of the selection of the most promising type of mining with relation to implementation of a large-scale works on remediation of this territory, a biometrics analysis of four substrates (CW, SM, SW and QS) has been implemented. At that, the SW material has the best prospects for application in Murmansk region with relation to its reasonable cost and favourable mineral content.

Mass of aboveground part of plants depends on the mineral material type, on which they grow (Fig. 1, *a*). In the beginning of a vegetative season (June) it has been the same for all variants, but in July biomass of a herbage, developed on mining wastes has been already significantly greater than on sand. The largest parameter values has been registered in variants with the use of CW (6.5 times higher than on sand), which may be stipulated by the highest calcium content in this substrate.

It is known that Ca/Mg ratio in the soil should be more than unity (by mass) to establish favourable conditions for plants development. The sole exception are serpentine soils, where high magnesium content with sufficient calcium content doesn't prevent from absorption of the latter by the rootage of plants [34]. Among the studied types of ameliorants, the above-mentioned ratio is less than unity for SM, but grass cover in this variant is characterized by quite high biomass (4.5 times larger than on sand) (Fig. 1, *a*).

Height of aboveground organs has been also maximal for plants in case of CW usage, the minimal one — on sand and in September it was equal to 58 and 41 cm correspondingly. For SM and SW this index has amounted to 56 and 51 cm respectively (Fig. 1, *b*). Height of plants in the sand variant was statistically significantly ($p < 0.01$) less in comparison with the other variants (Tukey HSD-test), at the same time, there have not been found any statistically significant differences between the height of plants in variants with CW, SM and SG.

Concentration of both macroelements and pollutants in aboveground organs of plants has varied during vegetation period (Fig. 2). An intensive calcium accumulation has been observed in the variant with CW (Fig. 2, *a*), and that of magnesium in experiments with SM (Fig. 2, *b*), which is closely connected with chemical composition of substrate.

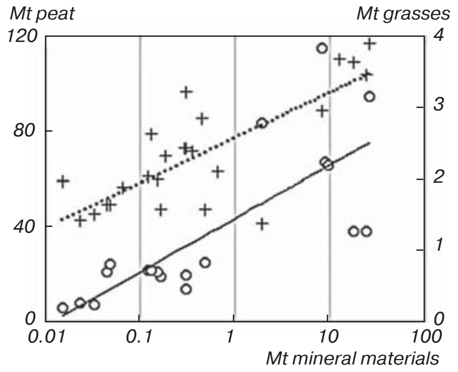

Fig. 3. Correlation between the toxicity module (Mt) of mineral materials (the X axis) and peat (___, the main Y axis), Mt of mineral materials and plants (····, an additional Y axis)

Towards the end of vegetation period (September) content of macroelements has lowered, while accumulation of microcomponents (copper and nickel) has continued (Fig. 2, *c*, *d*). Plants on QS and SM have accumulated metals to a greater extent than on CW and SW. According to the data of Table 2, CW and SW distinguish thereselves by heightened content of calcium as compared with QS and SM. The obtained results have showed that increased calcium content in substrates is an aid to lessening penetration of metals into plants.

Correlation between values of Mt, an integral chemical composition index of substrate layer, peat under the substrate layer and plants on substrate, is shown in Fig. 3, where the data for all types of substrates are represented. Correlation between Mt (substrate) and Mt (peat), Mt (substrate) and Mt (plants) takes place; credibillity in logarithmic approximation for a set of data varies from 0.62 and 0.59 correspondingly.

The substrate toxicity module is specified by its mineral composition. The higher calcium and magnesium carbonates and/or silicates content in substrate, the smaller its toxicity module and the higher an extent of its assistance in lessening the contaminated peat toxicity. Moreover, high content of bioavailable calcium and magnesium stimulates growth and development of plants as well as an establishment of stable phytocenosis on substrates. Thus, the key factor influencing on effectiveness of the elaborated technology of the depressed territories remediation in conditions of high pollution level is selecting the substrate for making a fill-up layer on the ground of the resolved plot.

When choosing mining wastes, one should also take into account an expediency of their utilization by phytoremediation technology throughout the territory of Murmansk region. From this standpoint the most promising material appears to be singulite wastes, which feature by reasonably large content of phytoavailable calcium and magnesium on the one hand, and by lack of prospects as secondary raw materials for extracting any valuable component, on the other.

Conclusion

1. Mining wastes (carbonatite waste, serpentinite-magnesite, sungulite waste) may be successfully used in the process of "mild" remediation on highly contaminated territories in Arctic conditions [36]. Possessing an alkalescent reaction of nutrient medium and high pool of the most of essential elements, they are effective ameliorants, promoting optimization of edaphic ground conditions of technogenic barren. The waste usage also assists in solving the problem of utilization of the stored wastes and lessening their negative impact on the environment.

2. The vegetation cover established on the basis of the complex harnessing local populations of perennial grasses (*Agropyron intermedium, Festuca rubra, Lolium perenne, Phleum pratense*), ameliorants out of mining wastes and swollen vermiculite, without resorting to soil and peat, scarce in the region, has been proven to be productive and resistant to aerotechnogenic burden, capable of longstanding self-dependent existence and providing an initial stage of progressive succession in the impact zones of copper-nickel enterprises.

3. In order to estimate favorability of technosoil and mineral materials during the process of monitoring research of artificial phytocenosis evolution, there has been suggested an integral index — toxicity module.

4. It has been found that sungulite wastes of phlogopite mining (city of Kovdor, Murmansk region) appear to be a sort of mining wastes which have considerable promise with relation to effectiveness and economic expediency of their use in large-scale work on remediation of depressed territories in Arctic conditions (Murmansk region).

V. V. Lashchuk and S. V. Drogobuzhskaya are also participated in the work (I. V. Tananaev Institute of Chemistry and Technology of Rare Elements and Mineral Raw Materials, Kola Science Center of the Russian Academy of Sciences, Apatity, Russia).

The work has been implemented owing to organizational and financial support of the Kola Mining and Metallurgical Company JSC. The work is included into the program of the Presidium of the Russian Academy of Sciences "Implementation of basic research in the interests of the Arctic region development", and has also received the support of the Russian Foundation for Basic Research, project мол_а_дк 16-35-60022.

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