ISSN 1064-2293, Eurasian Soil Science, 2013, Vol. 46, No. 6, pp. 697–703. © Pleiades Publishing, Ltd., 2013.
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DEGRADATION, REHABILITATION, AND CONSERVATION OF SOILS

Ecological Evaluation of Artificial Soils Treated with Phosphogypsum

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Received March 17, 2012

Abstract—An attempt to set up ecologically acceptable concentrations of toxic components contained in phosphogypsum was made for soils of different land uses. For this purpose, an experimental ecological evaluation of a standard soil mixture (model artificial soil ISO 11268-1) treated with phosphogypsum was performed. Both positive and negative effects of the phosphogypsum components were found. Thus, a significant increase in the biomass of lawn grasses was observed in the model soil with the phosphogypsum content of less than 3.3%. In the soil containing more than 6.8% phosphogypsum, the concentrations of Sr and F exceeded the maximum permissible values and adversely affected the living organisms. According to the basic ecological norms, the allowable content of phosphogypsum should be ≤2.0% for the soils of specially protected natural areas; ≤6.8% for agricultural and urban soils; and ≤9.6% for the soils of forest, water management, and transport lands.

Keywords: soil mixture, biomass, fluoride, strontium

DOI: 10.1134/S1064229313060124

INTRODUCTION

The natural soils in some Russian territories are subjected to heavy anthropogenic loads and have become unfit for use; they cannot perform their environmental functions. In particular, this has led to the necessity to apply artificial soils for landscaping, gardening, and rehabilitation of urban territories.

An artificial soil is an artificially constructed soil mass possessing fertility or the fertile soil layer removed from the surface of a land plot (Moscow Law No. 31, July 4, 2007). It is assumed that the processes taking place in the artificial soils are the same as those in the natural soils, so that artificial soils can completely replace natural soils in large cities (Regulation No. 514-PP of the Moscow Government, June 17, 2008). As a rule, artificial soils represent a mixture of three major components: mineral (clay, blanket loam, glaciofluvial sand, alluvial loam, etc.), organic (manure, peat, tree leaves, wood dust, etc.), and nutrient additives (mineral fertilizers, humin preparations, lime materials, etc.). The available literature data suggest that the effects of some traditionally applied nutrient additives introduced into artificial soils on the soil biota are not always favorable [17].

Phosphogypsum (PG) is a by-product of the production of phosphoric acid obtained as a result of the decomposition of raw phosphate materials or apatite concentrate with a mixture of sulfuric and phosphoric acids. It finds diverse applications in agriculture, the construction industry, and the pulp and paper industry. The presence of important nutrients, such as potassium, phosphorus, and sulfur, makes it possible to apply PG as a fertilizer. It is also used as an ameliorant of slightly solonetzic and solonchakous soils and solonetzes [3, 7, 25, 26]. According to the available literature data, the recommended rates of PG application range from 2 to 35 t/ha depending on the soil type, the crops grown, the application method, and the applied fertilizers [12, 13].

The effects of PG, which contains, together with the main component (gypsum), considerable amounts of admixtures in the form of compounds of stable strontium, fluorine, cadmium, and other elements, on the ecological state of soils are studied insufficiently [6, 21–23].

To determine the allowable levels of the quality of soils and artificial soils, the following characteristics should be taken into account: (1) the loss of the bioorganic potential[1], (2) the threshold values of the contamination and degradation of the soils and artificial soils under which the active transfer of contaminants and the soil mass into the adjacent natural media is still impossible, (3) the tolerance of the soils and artificial soils to anthropogenic impacts, and (4) the type of land use [19]. We examined the range of the excessive contents of PG in relation to its possible negative effects on the basic ecological norms applied to soils and artificial soils under different land uses (Table 1).

[1] The bioorganic potential represents the sum of the living and humified organic matter.

Table 1. Ecological requirements (norms) for the soils of different land use categories (according to [19])

Soil characteristics	Land categories					
	specially protected natural territory	agricultural land	urban land	forest land	industrial and transport land	water land
Chemical	Background	MPC/PPC		The transfer of pollutants into the adjacent natural media is not allowed		
Physical	″	The self-restoration capacity of soil ecosystems is preserved (the loss of no more than 30% of the soil bioorganic potential)				
Biological	″					

Biological methods based on the response of living organisms toward external impacts (including the presence of xenobiotics) are widely applied, in combination with chemical methods, for the integrated assessment of the environmental quality and the measure of the xenobiotic effects [2, 5, 10, 15, 20]. It is reasonable to study the responses of living organisms of different trophic levels: producers, consumers, and reducers [16, 24].

To characterize the limits of the allowable quality of soils of different land uses treated with PG, the changes in the concentrations of some potentially dangerous admixtures were studied, the effects of PG on the capacity of artificial soils to support the growth and development of higher plants, the effects of PG on the test functions of organisms of different trophic levels, and some bioindication parameters of the complex of soil microorganisms were examined.

OBJECTS AND METHODS

Model experiments on the effects of PG on the state of soil cenoses were performed with well reproducible conditions of artificial soils. The model artificial soil was prepared according to international standard ISO 11268-1 using specialized equipment of the Biogrunt company. It had the following composition: kaolin, 20%; transitional (mesotrophic) peat, 10%; and building sand (0.2–0.4 mm), 70%. Artificial soils of such composition are applied to study the maximum permissible concentrations of pesticides and the effect of different tillage operations on the soil status [18]. The model artificial soil had the following properties: the water content, 15.7 ± 0.9%; the pH_{KCl} 4.95 ± 0.06; the pH_{H_2O} 5.61 ± 0.06; the organic matter content, 10.9 ±1.4%; the P_2O_5 (available), 133 ± 38 mg/kg; and the K_2O (available), 38 ± 8 mg/kg.

The model artificial soil is much more homogeneous in its properties than any natural soil, and this makes it possible to solve some important problems typical of agrochemical experimentation, such as the minimization of the effect of the soil cover's heterogeneity with a significant increase in the representativeness of the obtained results and the minimization of the dependence of the results on the previous history of the plot.

The phosphogypsum used in the experiments represented the product of processing of apatite from the Kirov Field in the Khibiny Mountains. We studied seven variants of the model artificial soil with different PG concentrations: 0 (control), 1.1, 3.3, 7.5, 14.7, 25.0, and 100% of the soil mass. The artificial soil without PG served as the control.

The study was performed using pot and laboratory experiments. The methods of the chemical analysis, bioindication, and biotesting were used to evaluate the environmental effects of the PG. The chemical analyses included the determination of the pH of the soil water extract (by potentiometry), the concentrations of fluoride ions in the soil water extract by the method of ion-exchange chromatography, and the concentrations of the mobile forms of Ca and Sr in the ammonium acetate extract using atomic absorption spectrophotometry. The effects of PG on the communities of microorganisms and on the plants grown in the model soil were studied in pot experiments. For this purpose, 2 kg of the artificial soil mixture with different concentrations of PG were placed into trays 13 × 37.5 × 6 cm in size. The bioindication study was performed after a month of the exposure of these substrates in an open greenhouse.

The following parameters were determined in the samples: the intensity of the basal soil respiration (the CO_2 emission) with a 3700 gas chromatograph with a thermal conductivity detector [8]; the structure of the community of the soil micromycetes and their synecological characteristics by the method of inoculation of soil suspensions into Czapek's medium [9] with the evaluation of the total number of colony-forming units, the portion of dark-pigmented fungal species resistant to unfavorable factors, and the portion of quick-growing species utilizing easily available organic substrates; the Shannon and Pielou indices of the diversity; the biomass of the spores and the biomass of the mycelium of the soil micromycetes by the method of luminescent microscopy in preparations of soil suspensions stained with Fluorescent Brightener 28 (SIGMA) [8]; and the structure of the community

Table 2. Characteristics of the test systems and the methods of the evaluation of the ecotoxicity of the contaminated soil samples

Trophic level	Test organisms	Test function	Exposure time, h	Analytical method
Producers	Higher plants *Sinapis alba*	Germinating capacity/root length	96	ISO 11269-1
	Microalgae *Chlorella vulgaris*	Optical density of the cultural liquid	22	PND FT 14.1:2:3:4.10-04
	Microalgae *Scenedesmus quadricauda*	Change in the number of cells	72	FR 1.39.2007.03223
Consumers	Crustacean *Daphnia magna*	Mortality	96	PND FT 14.1:2:4.12-06
	Protozoan *Paramecium caudatum*	"	24	PND FT 14.1:2:3.13-06
	Mammal *Boa taurus*	Mobility of sex cells	2	PND FT 14.1:2:4:15-09
Reducers	Bacterium *Escherichia coli*	Change in the bioluminescence	0.5	PND FT 14.1:2:3:4.11-04
	Micromycete *Fusarium oxysporum*	Rate of the colony radial growth	120	[14]

Table 3. Changes in some chemical parameters of the model soil in dependence on the phosphogypsum content

Phosphogypsum content, %	pH_{H_2O}	P_2O_5 (available)	F^-	Sr^{2+}	Ca^{2+}	Ca : Sr
		mg/kg				
0 (Control)	5.80	132	0.75	3.5	1106	316
1.1	5.65	163	1.02	131	2450	19
3.3	5.22	275	1.55	394	7315	19
7.5	5.03	325	2.35	676	14751	22
14.7	4.85	405	4.93	1267	29348	23
25.0	4.26	970	6.85	1946	41564	21
100.0	3.39	2860	25.34	5647	152453	27

of the soil microorganisms by the molecular method of gas chromatography–mass spectrometry (according to the composition of the fatty acids of the cell walls) [1].

The ecotoxicity of the model soil with different concentrations of PG was evaluated by the methods of biotesting according to the responses of organisms of different trophic groups (Table 2). The toxicometric parameters—the EC_{50} (the semi-effective concentration of the PG causing a 50% decrease in the studied test function relative to the control) and the innoxious concentration of the PG (causing no observed effect level (NOEL) on the test organisms)—were calculated using the method of probit analysis [11].

The evaluation of the effect of the PG on the capacity of the model artificial soil to support the growth of higher plants was performed in an open greenhouse with mustard plants (*Sinapis alba* L.) and the universal mixture of lawn grasses as the test cultures. The germinating capacity and root length of the mustard plants and the dry biomass of the first cut of the lawn grasses were determined. An effect causing the decrease of the test function by more than 20% was considered as a toxic effect.

The statistical treatment of the experimental data was performed using the MS Office, Statistica 6.0, and Probit Analysis programs.

RESULTS AND DISCUSSION

Effects of the phosphogypsum on the physicochemical properties of the model soil. The analysis of some chemical properties of the model soil shows their changes with the addition of phosphogypsum. Thus, the pH of the soil water extract changes from 5.80 (control) to 3.39 (pure PG) (Table 3). This undoubtedly affects the mobility of the toxicants in the soil.

Table 4. Concentrations of phosphogypsum in the model soil (% of the mass) affecting the bioindication parameters EC_{50} and $NOEL_{20}$

Parameter	SB/MB ratio	CO_2 emission	N of microorganisms	
			by GC–MS	inoculation into Czapek's medium
EC_{50}	34.3 ± 3.7	46.7 ± 7.5	47.3 ± 6.3	Not det.
$NOEL_{20}$	9.6 ± 1.0	10.8 ± 1.5	17.3 ± 2.0	

Note: SB is the biomass of the spores, and MB is the mycelium biomass of the soil micromycetes; GC–MS is the method of gas chromatography–mass spectrometry (applied to fatty acids of cell walls).

The contents of the available phosphorus and some potentially dangerous chemical pollutants (Sr and F) in the artificial soil also depend on the concentration of PG; this dependence is described by the following equations:

$$[P_2O_5]_{(available)},\ mg/kg = 27 \times [PG, \%] + 146; \quad (R^2 = 0.92), \quad (1)$$

$$[Sr^{2+}],\ mg/kg = 77 \times [PG, \%] + 75; \ (R^2 = 0.95), \quad (2)$$

$$[F^-],\ mg/kg = 0.25 \times [PG, \%] + 0.80; \ (R^2 = 0.94). \quad (3)$$

The high value of the squared correlation coefficient ($R^2 = 0.92–0.95$) attests to the significant linear relationships between these parameters. It follows from eqs. (1)–(3) that an increase in the PG content in the model soil by 1% increases the content of extractable phosphorus, strontium, and fluoride ions by 27, 77, and 0.25 mg/kg, respectively.

The concentration of radioactively stable Sr is a characteristic of the soil contamination under the impact of PG application. As seen from Table 3, even an insignificant application of PG (1.1%) resulted in a 37-fold increase in the Sr content relative to the control. The effect of PG on the content of extractable Ca is much smaller. In the model soil with 1.1% PG, the extractable Ca content increased by 2.2 times. As a result, the Ca : Sr ratio is subjected to considerable changes under the impact of PG. In the model soil with 1.1% PG, the Ca : Sr ratio decreases by 17 times in comparison with the control. It is known that Ca and Sr ions can replace one another in the tissues of living organisms, so the Ca : Sr ratio serves as an index of the environmental danger and bioavailability of Sr [6]. According to the literature data [4], the soil concentrations of Sr exceeding 600 mg/kg can cause bone diseases in humans because of the disturbance of the calcium metabolism.

Fluorine is another pollutant contained in PG in great amounts. The concentration of its water-soluble forms can reach 25 mg/kg, whereas the maximum permissible concentration of this toxicant in soils is 2.8 mg/kg.

The contents of PG under which the maximum permissible levels of Sr (600 mg/kg) [25] and fluoride (2.8 mg/kg according to Sanitary Standard 2.1.7.1287-03) occur were calculated on the basis of eqs. (2) and (3) and comprised 6.8 and 8.0%, respectively.

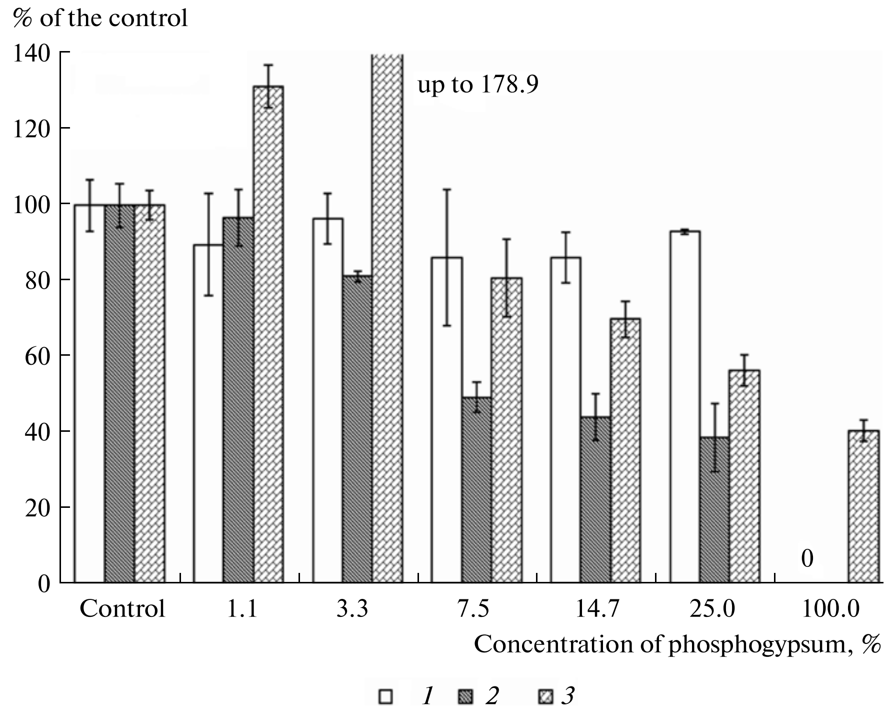
Thus, it follows from the obtained data that the addition of PG into the model soil in amounts up to 6.8% (by mass) does not cause an increase in the concentrations of the potentially dangerous pollutants above the permissible level.

Effect of phosphogypsum on the capacity of the model soil to support the growth of higher plants. The results of our study of the growth of higher plants under the influence of PG are presented in the figure. These data suggest that the ameliorant can have both stimulating and depressing effects on the higher plants. The maximum tolerance toward the influence of PG was found for the germination capacity of *S. alba* seeds. Of all the studied variants, only pure PG caused a decrease in this parameter by more than 20% relative to the control. A similar decrease (by 20%) of the length of the roots of *S. alba* and of the phytomass of the lawn grasses was observed upon the PG concentrations of 7.5 and 14.7%, respectively. It should be noted that the addition of PG in amounts of up to 3.3% resulted in a significant (by 80%) increase in the phytomass of the lawn grasses relative to the control.

Responses of the communities of soil microorganisms to the application of phosphogypsum into the model soil (bioindication experiments). The biotic responses to the influence of PG on the soil microbial communities were judged from the changes in their structural and functional parameters. Table 4 contains data on the concentrations of PG that caused changes in these parameters by 50 and 20% in comparison with those in the control (by analogy with the toxicometric parameters EC_{50} and $NOEL_{20}$).

The proportion between the mycelial and spore biomasses of the micromycetes was the most sensitive bioindication parameter. It was found that the concentration of PG above 9.6% had a negative effect on the communities of the soil microorganisms manifested by a rise in the portion of the spore biomass in the structure of the micromycetes.

The response of the standard test organisms of different trophic levels to the application of phosphogypsum into the model soil (biotesting). The analysis of the ecotoxicity for the samples of the model soil with PG in the biotest systems was based on the responses of organisms of different taxonomic categories belonging to the major trophic groups. This method reflects the



Effects of different concentrations of phosphogypsum on the growth and development of higher plants: (*1, 2*) germinating capacity and root length of *Sinapis alba* and (*3*) the phytomass of the universal mixture of lawn grasses.

systems approach and allows one to identify the trophic levels that are most susceptible to the impact of unfavorable factors.

The results of the biotesting were used to calculate the toxicometric parameters EC_{50} and $NOEL_{20}$ and to determine the most sensitive test organisms. The obtained data are presented in Table 5.

The following test functions proved to be most sensitive to the impact of PG: at the level of producers, the change in the growth rate of the cells of *Scenedesmus quadricauda* algae; at the level of consumers, the survival of *Daphnia magna* crustacea; and, at the level of reducers, the luminescence of *E. coli* luminescent bacterial strain in the Ecolum preparation. The semi-effective concentration (EC_{50}) of PG reached 5.1% for *S. quadricauda*, 7.7% for *D. magna*, and 15.3% for *E. coli*. The harmless concentrations of PG (NOEL) for these biotest systems were 2.0, 2.8, and 5.3%, respectively.

The obtained data suggest that the addition of PG into the model soil in concentrations of more than

Table 5. Concentrations of phosphogypsum in the model soil (% of the mass) affecting the test functions (EC_{50} and NOEL) of the organisms of different trophic levels

Test function	EC_{50}	NOEL
Producers		
Root length of *S. alba*	8.7 ± 1.2	3.8 ± 0.7
Growth rate *C. vulgaris*	79.6 ± 8.3	23.4 ± 2.7
S. quadricauda	5.1 ± 0.8	2.0 ± 0.3
Consumers		
Survival of *P. caudatum*	12.7 ± 1.5	4.4 ± 0.8
D. magna	7.7 ± 1.0	2.8 ± 0.4
Mobility of sex cells of *B. taurus*	23.8 ± 2.9	10.3 ± 1.2
Reducers		
Bioluminescence of the bacterium *E.coli*	15.3 ± 1.6	5.3 ± 0.7
Growth rate of *F. oxysporum*	Not found	

2.0% (by mass) results in the soil toxicity with respect to the *S. quadricauda* microalgae. On the whole, the analysis of the responses of the standard test organisms of the main trophic groups showed that they can be arranged into the following order according to their sensitivity to the PG: reducers < consumers < producers.

CONCLUSIONS

The application of chemical methods, together with bioindication and biotesting methods, allows us to obtain relatively reliable estimates of the effect of PG on soils. Phosphogypsum as a waste product of processing of natural minerals exerts both ameliorative and harmful effects on soils related to the changes in the soil acidity and to the presence of phosphate, fluoride, strontium, and calcium ions in the phosphogypsum. This circumstance points to the necessity to include the chemical, bioindication, and biotesting parameters into the system of soil monitoring for the soils treated with phosphogypsum.

The nonlinear character of the changes in the environmental quality upon uniformly increasing anthropogenic loads is examined in the theory of catastrophes. This phenomenon is described by Richards' function (the generalized logistic curve). The response of the soil biota and vegetation to the environmental loads characterized by the 20–30% loss of the biodiversity can be considered the boundary criterion [12]. We assume that the limit of the tolerance of the soil ecosystem corresponds to the 20% deviation of the controlled bioindication parameters from their values in the control soil; among these parameters, the structural–functional characteristics of the eukaryotic and prokaryotic (fungal and bacterial) microbial communities play the major role. On the basis of the obtained experimental data, we can determine the permissible concentrations of phosphogypsum in soils with due account for the ecological norms developed for different land categories. Thus, the permissible concentration of phosphogypsum in the model artificial soil should be less than 2% for the specially protected natural areas, no more than 6.8% for the agricultural and urban lands, and no more than 9.6% for the lands of forest and water land funds and for the lands of industry and transport. This corresponds to the application of phosphogypsum at the rates of 45, 150, and 210 t/ha, respectively (taking into account the bulk density of the model soil of 1.1 g/cm^3 and the thickness of the soil layer of 20 cm (the maximum thickness of the artificial soil layer in city gardens according to Regulation of the Moscow Government no. 743 from September 12, 2002).

ACKNOWLEDGMENTS

This study was supported in part by the Russian Foundation for Basic Research and by the Federal targeted program Scientific and Pedagogical Personnel for Innovation in Russia (project no. GK 14.740.11.0796).

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Translated by T. Chicheva