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Dynamics of Soil pH after Utilization of By-products of Industrial Rock Processing as a Calcareous Material in Acid Soils

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ABSTRACT

Production of crushed stone from carbonate rocks intended for road construction produces a huge amount of screening fractions, which accumulate in landfills. To date, in the Leningrad region 70 million tons of waste from the production of crushed stone has been accumulated. Its use as an ameliorant can significantly reduce the severity of acid soil problems in the region and at the same time solve an important environmental problem – landfill disposal. In a 6-year 10-variant microfield experiment, a comparative study of the reclamation properties of the screening dolomite dropouts without separation into fractions and the particles of 5–7 and 7–10 mm in size used for liming in increasing doses was conducted on a medium-acid sod-podzolic light loamy soil. These dolomite screenings neutralized soil acidity already in the first year, while their positive aftereffect lasted for at least 6 experimental years. The use of a natural mixture of the screenings in high doses was not effective because of high losses of bases from soil. Clustering of individual experimental variables was carried out by their effect on the pH_{KCl} value over the entire experiment period.

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Dolomite screenings; soil acidification; liming; clustering

Introduction

Utilization of human waste products and industrial wastes is one of the global problems of human development (Amaizah et al. 2013; Basu et al. 2009; Buntić et al. 2018; Corradini et al. 2020; Malik and Thapliyal 2009). The landfills of the industrial rock processing occupy huge areas of potentially arable lands, especially the location where calcareous rocks are processed for urban infrastructure purposes (Szczepańska and Twardowska 2004). At the same time soil acidification is one of the urgent soil degradation problems. Acid soils limit obtaining high and stable yields due to decreased availability of some plant nutrients (Xu et al. 2020) from the one hand and promote transition of some potentially toxic elements into plant available forms (e.g., Blake and Goulding 2002; Fei et al. 2020; Xu et al. 2019). Soil acidity is prone to rapid changes due to agronomic practices, particularly to liming. In this regard, particularly attractive is the use of calcareous industrial by-products in agriculture as a liming material (Barrow 2012; Lehman and Joseph 2015; Velichko and Kuzmich 1986). In previous years, the main focus in liming in Russia was the use of high-quality industrial lime fertilizers specially produced for the reclamation of acid soils. Later, the difficult economic conditions forced us to use the cheapest local calcareous materials and industrial wastes for neutralization of naturally acid soils of north-west

Russia. These ameliorants have different chemical composition, density, and porosity, and as such, their dissolution rate and duration of action in soils vary greatly.

Production of crushed stone from carbonate rocks intended for road construction produces a huge amount of screening fractions of sizes less than 10 mm, which are disposed in landfills. These dropout fractions consist of the sizes: < 0.25 mm – 11.4%; 0.25–1 mm – 11.5%; 1–3 mm – 28.6%; 3–5 mm – 16.5%; 5–7 mm – 11%; 7–10 mm – 11%; > 10 mm – 16% CaCO_3 content – 46.1%; MgCO_3 – 38.4%. To date, in the Leningrad region 70 million tons of waste from the production of crushed stone is accumulated. Therefore, the problem of their utilization is long overdue. There are no factories for grinding dolomite rocks into dolomite flour in this region. However, these waste by-products have a potential to be used as soil liming material, especially to reduce the severity of acid soil problems in the region and at the same time to solve an important environmental problem of landfill disposal, thus opening new land areas for agricultural production. Hitherto, it was believed that ameliorant particles greater than 1 mm are ballast in soil because generally they are not suitable for liming due to their slow solubility. Litvinovich et al. (2016), (2017)) studied the particle-size distribution and fertilization value as well as liming characteristics and duration of dissolution of the screening fractions of dolomite stone of the sizes less than 5 mm (0.25; 0.25–1; 1–3 and 3–5 mm). Their studies proved a high efficacy of these fractions when limed the sod-podzolic soils (Umbric Albeluvisol Abruptic). To continue studying the possibility to use the larger fractions of the screenings as a liming material a new experiment using the coarser particles of dolomite to see its behavior in soil and effects on plant was initiated. Since, irrespective of the chemical composition of the calcareous material used, the soil loses its base and gradually oxidizes. Because the rate of acidification depends on the chemical composition of the liming material, its chemical composition should be well compiled (Litvinovich et al. 2018).

Soils of northwest Russia are naturally acidic and require repeated liming. Liming has a long and comprehensive influence on all components of the soil (Bakina 2012; Bennett et al. 2014; Goulding 2016; Litvinovich et al. 2017; Paradelo, Virto, and Chenu 2015). However, the rate of acidification in limed soils that are in use is even higher (Goulding 2016) than of adjacent non-limed soil. There is still no consensus on the duration of the action of liming material and the terms of re-liming in the literature (e.g., Anderson et al. 2013; Hardy, Tucker, and Carter 2014; Higgins, Morrison, and Watson 2012). Furthermore, studies on the utilization of large-sized dolomite screenings for land reclamation are scarce. Another aspect of this study is cost-effectiveness of the use of waste materials for improving soil fertility, especially for the low-resource farms (Ilves, Smolina, and Danilov 2020).

The main hypothesis was that the industrial by-products from dolomite stone production can be used as long-lasting ameliorants for maintaining soil acidity. The aim of this study was comparison of reclamation characteristics of the dolomite stone screenings without fractionation (natural mixture) and the screening particles of the 5–7 and 7–10 mm in a six-year 10-variable micro-field experiment. The objectives included 1) studying dynamics of the changes of pH_{KCl} value in sod-podzolic soil reclaimed with increasing doses of coarse dolomite screenings, 2) clustering of individual treatments limed with different sized particles according to their effect on the pH_{KCl} value for the entire experiment period and 3) developing linear trends of the acidification process for individual groups throughout the study period.

Materials and methods

The objects of the study were dolomite screenings of 5–7 and 7–10 mm sizes as well as natural mixture of the total screening fractions. The fractions of the 5–7 and 7–10 mm sizes composed 11% of the total screening. For the comparison, dolomite powder (DP) prepared from the screenings with 0.25 mm sieve was used. The screening contained 46.1% of CaCO_3 and 38.4% of MgCO_3 . The experiment was designed as a 10-variable microfield trial established on sod-podzolic soil at the Agrophysical Research Institute, Saint-Petersburg, Russia and lasted 4 years with the six terms sampled and studied (Table 1, Figure 1). Each of four experimental replication was set in polyethylene vessels without a bottom

Table 1. Dynamics of pH_{KCl} changes over 6 years of the experiment.

No	Treatment	Test years and terms						SumS _i	Average \bar{S}_i
		2015	2016		2017		2018		
		1 peas	2 mustard	3 pea	4 mustard	5 pea	6 mustard		
1	Background (NPK)	4.6	4.2	4.2	4.1	4.2	3.8	25.1	4.2
2	Background (NPK) + dolomite powder 1 Ha*	5.7	5.6	5.7	5.9	5.8	5.6	34.3	5.7
3	Background + (5–7 mm) 1 Ha	5.0	4.3	4.5	4.5	4.4	4.3	27.0	4.5
4	Background + (5–7 mm) 3 Ha	5.5	4.5	5.0	4.8	4.7	4.9	29.4	4.9
5	Background + (5–7 mm) 5 Ha	6.0	4.9	5.5	5.3	5.2	5.5	32.4	5.4
6	Background + (7–10 mm) 1 Ha	5.3	4.2	4.5	4.4	4.3	4.4	27.1	4.5
7	Background + (7–10 mm) 3 Ha	5.5	4.3	4.7	4.6	4.7	4.8	28.6	4.8
8	Background + (7–10 mm) 5 H	6.0	5.0	5.0	5.0	5.0	5.2	31.2	5.2
9	Background + mixture 1 Ha	5.7	5.4	5.1	5.0	5.0	5.1	31.3	5.2
10	Background + mixture 3 Ha	6.3	4.8	6.1	5.9	5.9	6.4	35.4	5.9

*Ha – Hydrolytic acidity; DM – dolomite powder; NPK – fertilizer (16:16:16)

**Figure 1.** Microfield experiment in studying the reclamation properties of screening of crushed stone production.

(S = 1 m², depth 25 cm, weight 300 kg soil per vessel). Before setting the experiment, soil was extracted from each plot (vessel) to the depth of the arable layer (25 cm). A plastic film was placed around the perimeter of the plots. The prepared vessels were filled in with the soil preliminary limed and fertilized. The increasing doses of liming (Table 1) were calculated by hydrolytic acidity (Ha), where a full dose (1 Ha) is calculated as: $\text{CaCO}_3, \frac{t}{\text{ha}} = \frac{\text{Ha} \times 10 \times 50 \times 3000000}{10^9} = \text{Ha} \times 1.5$, where Ha is a hydrolytic acidity, mmol (eq.) 100⁻¹ g soil; 10 is conversion into mmol (eq) kg⁻¹; 50 is the amount of CaCO₃ required to neutralize 1 mmol (eq) of H⁺, mg; 3000000 – mass of arable layer per 1 hectare, kg; 10⁹ – conversion from mg ha⁻¹ into t ha⁻¹). The hydrolytic acidity was determined according to Kappen (1934): the samples were treated with 0.5 M dm⁻³ Ca-acetate solution adjusted to pH 8.2 in the ratio of 1:2.5. The suspensions were shaken at room temperature for 1 hour then filtrated. The filtrates were titrated with 0.1 M dm⁻³ NaOH solution in presence of phenolphthalein indicator and the hydrolytic acidity values were calculated from the amount of alkali consumed (0.1 M dm⁻³ NaOH cm³ for 50 g soil).

Effect of liming was tested on the crops sensitive to liming and with high requirements for Ca and Mg: In 2015 – peas; in 2016 and 2017 – mustard and pea, and in 2018 – mustard. Crops were harvested

in the flowering phase. Fertilizers were applied annually. At the start of the experiment 60 g of nitrogen, phosphorus, and potassium fertilizer (NPK in ratio of 16:16:16, respectively) was applied into each vessel. In the second and fifth years 18 g of NPK, and in the third, fourth, and sixth years 30 g of NPK were applied. Soil was sampled annually after harvesting. Statistical calculations were done according to the Bure (2007).

Results and discussion

The pH_{KCl} value of a soil is a universal soil indicator reflecting the diversity of soil properties and chemical soil processes. It depends primarily on the content of organic matter in the soils, mobile aluminum, exchangeable hydrogen, manganese, and iron. Determination of pH_{KCl} is one of the mandatory analyzes in the study of agrochemical parameters and is absolutely necessary for judging the dynamics of changes in acid-base properties of reclaimed soils. The initial soil characteristics were as follows: pH_{KCl} 4.6; H_a – 4.9 mmol.eq/100 g soil; content of humus – 2.16%; particles < 0.01 mm – 21.4%. The dynamics of the changes in pH values in the studied soils are presented in Table 1.

Cultivation of peas without liming (the 1st year) didn't change the pH value after a year of the experiment. However, already after the second year, the soil pH in this treatment decreased and by the end of the experiment reached the value of 3.8 (Table 1). This suggests that arable sod-podzolic soil that is fertilized but not limed is constantly acidifies thus gradually degrading. Similarly, Danilov, Yakovleva, and Nikolaeva (2020) on the soils of northwest Russia confirmed that sod-podzolic soils intensively lose their cations and acidifies due to application of mineral fertilizers. In podzolic soil types calcium is highly mobile due to weak retention by soil (Bakina 2012). Main causes of acidification in arable soils are nutrient mining by crops, losses of basic cations due to leaching with downward water movement (Karlen et al. 2008; Xu et al. 2020), especially of Ca (Pavlova et al. 2019a, 2019b). According to Nebolsin and Nebolsina (2010) annual uptake of Ca by peas can reach 60–80 kg ha⁻¹ and by mustard 300–500 kg ha⁻¹, calculated as per CaCO₃. While annual losses of Ca due to leaching can reach 400 kg ha⁻¹ (Zelenov et al. 2010) and Mg 12–42 kg ha⁻¹ per year (Mazaeva 1977).

Application of dolomite powder (DP) resulted in a decrease of soil acidity in the first year of liming. After harvest of peas the pH value was 5.7, and further throughout the experiment it ranged between 5.6 and 5.9. According to modern concepts, the effect of lime on soil acidity over time can be divided into three phases. In the first phase, in spite of the removal and washing out of the bases, the soil acidity does not increase, since the processes of soil interaction with the remains of unreacted lime go on simultaneously and the number of bases in the soil is replenished. Litvinovich et al. (2019) showed that dissolution of DP lasted three years, during which the unreacted amount of carbonates was detected. The second phase is increasing acidification of soil due to crop removal and leaching down the soil profile. The third phase is return to the initial state (Nebolsin and Nebolsina 2010). In our study, application of DP maintained the value of soil pH at the first year level throughout six years of the experiment.

Application of the screening of the 5–7 mm size in a dose of 1 Ha resulted in a decreased soil acidity in the first year of application from 4.6 to 5.0. After 2 experimental years, the value of pH decreased to 4.3, and further throughout the experiment it ranged between 4.3 and 4.5, i.e., was lower than initial value. However, important is that throughout the entire experimental years the value of pH in this treatment was higher that of the control (non-limed) soil. This suggests that the 5–7 mm size screenings showed weak but positive effect on soil acidity. An increase of the dose of these fractions up to three full doses (3 Ha) resulted in a regular increase in the reclamation effect. After a year of liming (after harvest of peas) the value of pH approached the value of that in DM treatment (Table 1). In the year of aftereffect, the pH_{KCl} decreased to 4.5 and then, over the entire study period, ranged from 4.7 to 5.0, thus retaining its positive effect. The maximum reclamation effect from the use of particles of the 5–7 mm size was achieved in the treatment with five full doses (5 Ha). The maximum shift to pH 6.0 was recorded in the year of liming. In the second year it decreased to 4.9, and starting

from the 3rd year until the end of the experiment, pH_{KCl} ranged from 5.2 to 5.5. Thus, with an increase in the dose of dolomite screenings of 5–7 mm size, the reclamation effect intensified. Using five full doses allowed reducing the soil acidity from very acid to a slightly acid reaction.

The effect of liming with dolomite screenings of 7–10 mm size on the value of pH_{KCl} in the year of liming didn't significantly differ from the treatment with 5–7 mm screenings. The reclamation effect from the use of these fractions in equal doses was the same. The effect of the particle size, applied in an equivalent amount, on the pH_{KCl} of the soil after two experimental years, has also not been established. In the 3rd, 4th, 5th and 6th experimental years, a certain (insignificant) advantage of the 5–7 mm fraction was noted compared with the 7–10 mm.

In general, the dynamics of changes in pH_{KCl} in the treatments reclaimed by large-sized particles of dolomite screening differ significantly from the dynamics of changes in soil acidity in the treatment with dolomite powder. According to modern concepts, this can be explained by the nature of interaction of calcareous materials with soil that occurs with the participation of two mechanisms:

- (1) due to the gradual transition of the bases into the soil solution, followed by a reaction with a soil-absorbing complex. This mechanism is fully consistent with the dissolution of DP in the soil, and
- (2) due to the contact exchange of the surface of the ameliorant particle and soil. The exchange process does not involve the inner layers of granules (Nebolsin and Nebolsina 2010).

The dynamics of changes in pH_{KCl} in the treatments with the use of 5–7 and 7–10 mm fractions, regardless of the dose, was of a similar nature and showed the following: a sharp increase in pH after the 1st test year; decrease in soil pH_{KCl} after harvest of the 2nd year crop; increase in the 3rd test year; and then during the 4th, 5th and 6th test years, the fluctuations of the value of pH were generally insignificant. Such dynamics is explained as follows. The dolomite screenings in the landfills is subject to physical destruction (weathering the surface of the granules). In the process of weathering, the crystallization structural bonds between the carbonate molecules are destroyed. The outer layer acquires certain friability and dissolves quickly when it enters the soil. In the year of application of large particles, a sharp increase in the soil pH occurs. Further, the dissolution is slowed down, since the inner layers located under the weathered layer are more durable. In our experiments the decomposition proceeded according to the second mechanism. At the same time, plants are able to absorb cations from local foci of soil adjacent to the surface of the ameliorant (Nebolsin and Nebolsina 2010). The use of large-sized fractions of dolomite screenings in high doses creates a supply of carbonates in the soil for a long time, which gradually dissolving enriches the soil solution with calcium and magnesium cations necessary for plants. In this regard, it should be emphasized that after the experiment was completed, a significant amount of undecomposed dolomite particles remained in the soil of the lime vessels. This implies that one can expect further longer aftereffect.

The use of a natural mixture of the screening fractions of the dolomite in a dose corresponding to 1 Ha decreased soil acidity. After the 1st test year, the pH didn't differ from the value established in the treatment with dolomite powder (DP). In the subsequent test years the soil acidity gradually increased, and after the completion of the experiment, pH_{KCl} corresponded to 5.1, i.e., was higher than at the start of the experiment. The reclamation effect over the entire study period was inferior to DP treatment used in an equivalent dose. Increasing the dose of mixed fractions to an amount corresponding to 3 Ha enhanced its effect on soil pH. At the end of the experiment, the pH_{KCl} value corresponded to the value established after the 1st year of application of the ameliorant. However, when using a natural mixture of dolomite screenings as a calcareous material, the use of the doses exceeding the dose higher than 1 Ha is not recommended due to an increase in unproductive losses of Ca and Mg as a result of migration. So, Litvinovich et al. (2019) showed that the eluvial losses of bases at using a mixture of fractions at a dose of 3 Ha was higher than the base losses when applying dolomite powder in a one full dose of 1 Ha. Therefore, the use of screening dolomite in high doses without fractionation is not effective.

The mathematical processing of the data consisted in clustering (grouping of individual experimental treatments by the magnitude of the reclamation effect obtained as a result of liming) (Table 2). To do this, the sums of pH were calculated in each treatment for the experimental years in the penultimate column, and the arithmetic means in the last column in Table 1.

For the sums, the following equations of inequality were performed:

$$S_1 = 25, 1 < S_3 = 27, 0 < S_6 = 27, 1 < S_7 = 28, 6 < S_4 = 29, 4 < S_8 = 31, 2 < S_9 = 31, 3 < S_5 = 32, 4 < S_2 = 34, 3 < S_{10} = 35, 4$$

Given the proximity of the sums to each other, it is advisable to consider the following clustering (the difference between the sums from different groups is more than 1.5 units; and within the groups less than 1.5 units):

- (1) the group 1 includes only treatments of the first experiment (background (NPK))
- (2) the group 2 includes treatments no 3 and 6 (background+5-7 mm at 1 Ha and background +7-10 mm at 1 Ha)
- (3) the group 3 includes treatments no 4 and 7 (background+7-10 mm at 3 Ha and background +5-7 mm at 3 Ha)
- (4) the group no 4 includes treatments no 5, 8 and 9 (background+7-10 mm at 5 Ha, background +5-7 mm at 1 Ha and background+5-7 mm at 5 Ha)
- (5) the group no 5 includes treatments no 2 and 10 (background+DP and background+mixture at 3 Ha)

In order to verify the validity of the clustering for each group, average dynamics of the pH value for 6 experimental years were constructed. The linear trends of averaged dependencies are given in Table 3. The graphical display of the models is given in the Figure 2.

The performed clustering confirmed the correctness of the earlier suggestion that there were no significant differences in the effect of fractions of 5–7 and 7–10 mm, applied in the same doses, on the pH_{KCl} value. The justification of the cluster analysis of individual treatments into groups is confirmed by the trends of the averaged dynamics of pH_{KCl} . Trends show different patterns, and the average data don't match. The trend lines for groups 1 to 4 are almost parallel and there is a significant shift in the trends relative to each other.

Table 2. Averaged dynamics of pH_{KCl} value change within individual groups over 6 test years.

Group number	Test-year					
	1	2	3	4	5	6
1	4.60	4.20	4.20	4.10	4.20	3.80
2	5.15	4.25	4.50	4.45	4.35	4.35
3	5.50	4.40	4.85	4.70	4.70	4.85
4	5.90	5.10	5.20	5.10	5.07	5.27
5	6.00	5.20	5.90	5.90	5.85	6.00

Table 3. Linear trends of averaged dependencies.

group No (model No)]	The equation of the linear trend
1	$y_1 = 4,593 - 0,117 \cdot t$
2	$y_2 = 4,883 - 0,107 \cdot t$
3	$y_3 = 5,08 - 0,07 \cdot t$
4	$y_4 = 5,609 - 0,096 \cdot t$
5	$y_5 = 5,6 + 0,056 \cdot t$

t — time (test-year).

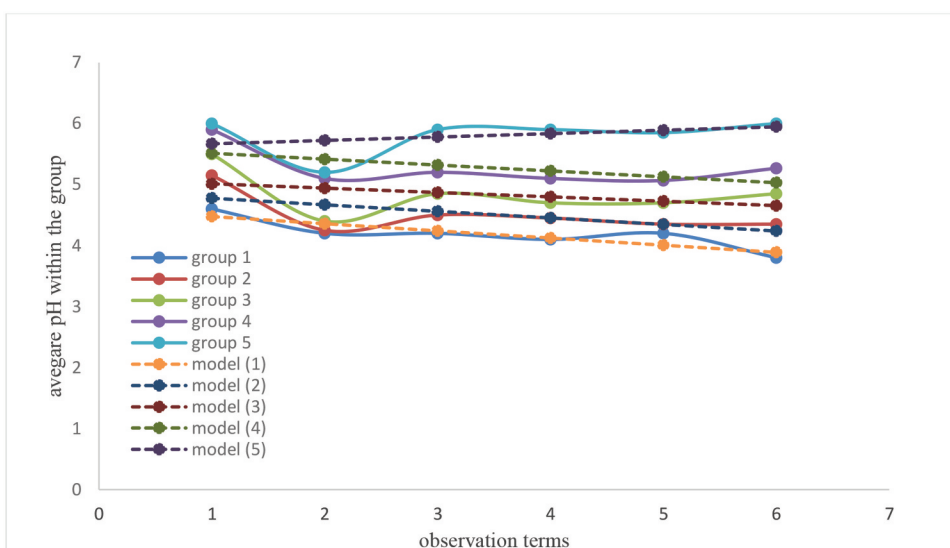


Figure 2. The averaged dynamics of soil pH under use of dolomite screenings in a long-term microfield experiment.

Conclusions

Dolomite fractions of 5–7 and 7–10 mm in size are valuable calcareous material, since they neutralize soil acidity already in the first year of reclamation with positive aftereffect lasting for at least six years. The reclamation effect was greater, the higher the dose of application. However, the use of the mixture of natural dolomite screenings in a deliberately overestimated dose was not effective due to high migration losses of the bases. In practical terms, the results obtained make it possible to choose such a dose of these fractions at which the annual plant demand for calcium and magnesium will be fully satisfied, and unproductive losses due to migration will be minimized.

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