

Influence of natural zeolite on nitrogen dynamics in soil

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Abstract: Sorption of the cations NH_4^+ , Al^{3+} , Fe^{2+} , Mn^{2+} , and Mg^{2+} in powdered zeolite and its mixture with soil was monitored. More than 90% of the observed cations were fixed in the zeolite in the first few minutes of the laboratory experiment, which is a very fast speed of sorption, and the ammonia cations reacted fastest. Consequently, the dynamics of the nitrate and ammoniac nitrogen in soil after application of zeolite in different amounts was monitored. A decreased ammonium content was apparent in the variants with zeolite 1 month after zeolite application in comparison with variants without zeolite owing to the specific fixation of NH_4^+ cations in the zeolite lattice (92.5 mg in the control variant and 77.2–81.0 mg per kg of soil in the tested variants). Three months later, there was an increase in ammonium content in the variants with zeolite of 24% to 59% in comparison with the control variant owing to gradual ammonium release from the zeolite lattice. Six months after the zeolite application, statistically significant differences were found not only between the control variant and the variants with zeolite, but also between individual variants with various zeolite doses ($H = 14.201$; $P = 0.003$ according to the Kruskal–Wallis test). The nitrification process in the soil was less intense under the influence of applied zeolite. The nitrate nitrogen contents in the soil decreased by 66% to 78% in comparison with the control variant in the autumn period; therefore, the amount of nitrate leaching from the soil horizons to the groundwater was less. In summary, zeolite can be considered to be a slow-releasing nitrogen fertiliser.

Key words: Ammonium, nitrate, soil nitrogen dynamics, sorptive speed, zeolite

1. Introduction

Zeolites are crystalline hydrated aluminosilicates. Their basic building unit is a crystalline lattice of silicon tetrahedrons and aluminium octahedrons. Zeolites, as individual rocks, were discovered by the Swedish scientist Alex Frederik Cronstedt in 1756 (Cronstedt, 1756). Regardless of their impressive physicochemical properties, zeolites did not find practical application for a long time because investigations concentrated exclusively on zeolites of volcanic origin. The number of zeolites in the total volume of rocks rarely exceeded a few per cent. The discovery of zeolites of sedimentary origin allowed their use to be investigated seriously. Because of their extremely high sorptive capacity, which depends on the dominant mineral, zeolites can be utilised in different fields of national economy, including agriculture.

The utilisation of zeolites in agriculture as a fungicide, herbicide, or carrier of plant nutritional elements is feasible simply because of the high sorptive capacity of this rock. When nutrients are introduced into the soil in this way, their consumption is reduced, so there is no need

for redundant delivery of raw materials and consequently fewer nutrients (mostly nitrogen, which causes eutrophication of water sources) are leached into ground and surface waters. Zeolites from Italy (Ciambelli et al., 1985) and New Zealand (Nguyen and Tanner, 1998) have been used for ammonium removal from wastewater. After researching Turkish zeolites, Işildar (1999) confirmed that nitrification decreased after zeolite application to soil, albeit not in all observed soil types. Zeolites from eastern Slovakia were used to protect the groundwater at waste disposal sites (Fronczyk and Garbulewski, 2013). Another use for natural zeolites is to decrease the heavy metal content of plants (Petkova et al., 2000) or reduce the soil contamination risk of heavy metals (Michalik and Missik, 1995).

2. Materials and methods

2.1. Natural zeolite

The zeolite used in this work was obtained from the Nizny Hrabovec deposit (48°51'36"N, 21°45'54"E) in Slovakia. Its chemical composition was as follows: SiO_2 , 70.9%; Al_2O_3 ,

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1.1%; Fe_2O_3 , 1.4%; CaO , 2.9%; K_2O , 3.3%; MgO , 0.80%; Na_2O , 0.54%; H_2O^+ , 4.6%; H_2O^- , 4.3%; and ignition loss, 2.78%. The rest was created by traces of other elements (titanium, manganese, phosphorus, sulphur, and others). The content of heavy metals in the zeolite was no higher than was permissible for waste application to agricultural soils in Slovakia (Table 1).

Zeolite with the dominant mineral clinoptilolite was from the Nizny Hrabovec deposit and its granulometric composition was as follows: fraction 0.06–0.02 mm, 11.10%; fraction 0.02–0.01 mm, 16.36%; and fraction <0.01 mm, 72.54%.

This powdered zeolite, containing particles smaller than 45 μm , contains on average 57.2% clinoptilolite (Singliar, 1992). In this work, the chemical composition of the powdered zeolite used was determined after sample analysis of a mixture of inorganic acids (Na, K) or after melting with Na_2CO_3 (Al, Ca, Fe, and Mg), and eventually zeolite heavy metal contents were gravimetrically (SiO_2 , unbounded and bounded water) determined in lixivium of 2 M HNO_3 .

2.2. Sorption of selected cations in zeolite powder

The sorption speed of NH_4^+ , Ca^{2+} , Mg^{2+} , Fe^{2+} , and Mn^{2+} cations from a water solution in the powdered zeolite was evaluated first. The following procedure was chosen: to 1 L of solution (prepared from NH_4Cl , FeSO_4 , MnSO_4 , CaCl_2 , and MgCl_2 salts) that contained 50 mg NH_4^+ , 30 mg Fe^{2+} , 10 mg Mn^{2+} , 200 mg Ca^{2+} , and 100 mg Mg^{2+} , 100 g of powdered zeolite was added and it was mixed for 1, 2, 4, 10, 20, 40, 80, 160, 320, 540, and 960 min. Ca, Mg, Mn, and Fe concentrations were determined by atom absorptive spectrophotometry, and ammonium cations were determined photometrically using the Nessler and Jendrasik method (Hanes, 1999). To determine cation sorptive capacity, a methodology based on direct displacement of the saturated salt NH_4Cl was used, whereby an adsorbed cation NH_4^+ was directly displaced by KNO_3 solution. The cation NH_4^+ was identified in the KNO_3 extract after rinsing with an acidified solution of NaCl by way of distillation and titration or photometry (Hanes, 1999).

2.3. Influence of natural zeolite on the mineral nitrogen dynamics in soil

In consequence of the results of the laboratory experiments, the effect of natural zeolite on the dynamics of mineral nitrogen in soil with a depth of 0.0–0.3 m was verified in field experiments. Selected chemical soil properties were determined using the obligatory methods for soil analysis (Fiala, 1999).

There were 4 variants of the experiment: K, control variant without any fertilisation; Z_1 , 600 kg of zeolite per hectare; Z_2 , 900 kg of zeolite per hectare; and Z_3 , 1200 kg of zeolite per hectare.

All variants were carried out 4 times. The experimental field was 50 m^2 (10 \times 5 m). The experimental crop was medium-ripening white cabbage (*Brassica oleracea* L., Slava variety) grown with the usual agricultural techniques. Seedlings were planted on 26 April and harvested on 25 August 2011. Average soil samples were taken and analysed for pH, nitrogen (ammonia and nitrate), and the contents of available phosphorus, potassium, magnesium, and organic carbon before the experiment was set up and after its completion on each site. In addition, once a month, soil samples from a depth of 0.0–0.3 m were taken from each site in order to monitor the dynamics of ammonia and nitrate nitrogen in the soil.

The statistical processing assumed that the zeolite dose influenced both ammonium and nitrate content in the soil. The Kruskal–Wallis test was chosen for verification of this hypothesis. In the case of rejection of the null hypothesis, the nonparametric Mann–Whitney test was used.

3. Results

3.1. Cation sorption speed in zeolite powder

The exchangeable zeolite cations Ca^{2+} , Mg^{2+} , K^+ , and Na^+ are already present in natural deposits. These cations can exchange zeolite for NH_4^+ and other metal cations, depending on the pH value of the solution and their concentrations.

The time-dependent development of NH_4^+ sorption from solution in zeolite is shown in Figure 1, where the curve reflects the time dependence of the sorption of the initial NH_4^+ concentration.

Table 1. Content of heavy metals in zeolite (in mg kg^{-1} of solids in lixivium 2 M HNO_3).

	Pb	Cd	Cr	Cu	Zn	Ni
Zeolite	16.8	0.15	3.0	15.0	111	1.9
Maximum allowed content*	750	10.0	1000	1000	2500	300

*: According to the Act of the National Council of the Slovak Republic No. 188/2003 about waste application into agricultural soils.

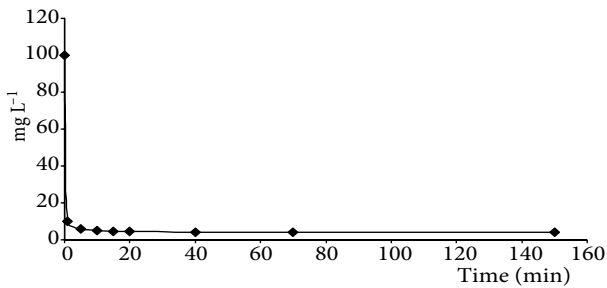


Figure 1. Sorption rate of ammonia cations (NH_4^+) on powdered zeolite (0.1 kg L^{-1} , fraction under $45 \mu\text{m}$).

The development of the sorption indicates a very rapid response and, in the first minute, a sorption of more than 90% of the maximum potential ammonia concentration in zeolite on an infinite timescale can be observed. During the next period from 2 min up to several hours, the concentration of NH_4^+ does not change significantly and is almost constant. Figure 2 presents the rate of sorption of the other observed cations. Similar to the sorption rate of the ammonia cations, the iron cation sorption (Fe^{2+}) is very fast. A significant decrease in Fe^{2+} concentration could also be caused by Fe^{2+} oxidation to Fe^{3+} , and it is coagulated at filtration. Manganese (Mn^{2+}) sorption is also fast in the first minute, but as manganese has less sorption selectivity as regards zeolite, no significant reduction in concentration occurs, only approximately 40%. The calcium (Ca^{2+}) concentration in solution with time dependence did not have a tendency to increase. The concentration of Mg^{2+} was initially slightly decreased and then increased slightly later.

On the basis of experiments in which the time dependence of NH_4^+ , Fe^{2+} , Mn^{2+} , Ca^{2+} , and Mg^{2+} sorption in the zeolite powder was measured, it can be concluded that the sorption of the cations is extraordinarily fast. This is illustrated by the fact that in the first minute sorption in zeolite reached more than 90% of the maximum possible concentration of equilibrium, which would stabilise in

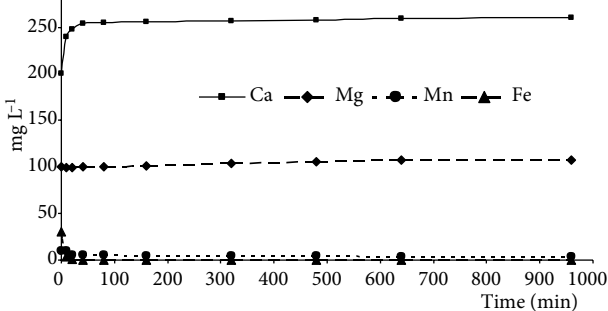


Figure 2. Sorption rate of selected cations Mg^{2+} , Fe^{2+} , Mn^{2+} , and Ca^{2+} on powdered zeolite (0.1 kg L^{-1} , fraction under $45 \mu\text{m}$).

infinite time. It can be assumed, however, that this process is dependent on the sizes of the zeolite particles, so when they increase, the stabilisation of sorption equilibrium, and especially that of NH_4^+ , will be slower.

3.2. The effect of zeolite application to soil on mineral nitrogen dynamics

The stabilisation of ionic exchange balance in several minutes or hours can be considered as fast because the zeolite will be in the soil for several years after its application. This knowledge can be applied to liquid nitrogen-based plant nutrition on the basis that zeolite applied to the soil would prevent the escape of redundant ammonium ions, as these would be bound to zeolite in moist soil very quickly and would be gradually released into the soil solution. The observed cation solution was verified by a field experiment based on clay eutric Cambisols, the basic chemical properties of which are shown in Table 2 (nutrient analyses according to the Mehlich method, organic carbon according to the Tjurin method).

In Figure 3, the dynamics of NH_4^+ in the soil is presented. It can be seen that after 1 month of zeolite application to the soil, the eutric Cambisol topsoil contained 14% to 20% more ammonia nitrogen (92.5 mg kg^{-1} of soil) in the control variant compared with the experimental variants ($77.2\text{--}81.0 \text{ mg kg}^{-1}$ of soil). There was a change in this subsequent period; the content of ammonia nitrogen in the topsoil in the variants with applied zeolites increased and after 3 months reached 24% to 59% higher values (depending on the amount of zeolite) than the control variant. At the end of the field experiment (5 months after the application of zeolite), the experimental variants reached $68.5\text{--}86.9 \text{ mg}$, and the control variant reached only 55.2 mg per kilogram of soil.

From the calculated values of the Mann–Whitney test, we can conclude that statistically significant differences in mean NH_4^+ concentrations in the soil were confirmed not only between the control variant and the observed ones, but between all observed variants, as well (Table 3).

Statistical analysis of the results according to the Kruskal–Wallis test confirmed the significant differences between the mean values of all observed variants ($H = 14.201$ with $P = 0.003$). The Mann–Whitney test results are given in Table 4.

After the zeolite application, the nitrate nitrogen content also changed during the observed period (Figure 4). All variants kept the spring maximum in April ($24.3\text{--}27.5 \text{ mg kg}^{-1}$ of soil) and the summer minimum in June and July ($1.4\text{--}4.9 \text{ mg kg}^{-1}$ of soil). At the time of crop harvest (in August) there was an apparent increase in nitrate nitrogen (12 mg kg^{-1} of soil) in the control variant, which corresponded to the so-called autumn maximum. This was not confirmed in the variants with zeolite because the nitrification process of ammonia nitrogen, owing to the

Table 2. Selected chemical soil parameters and clay content in the soil (eutric Cambisols).

Depth	pH	N-NO ₃ ⁻	N-NH ₄ ⁺	N _{an}	N _{total}	P	K	Mg	C _{org}	Clay
(m)		mg kg ⁻¹							%	
0.0–0.3	7.2	34.3	26.8	61.1	2043	118	411	166	1.62	65.4
0.3–0.6	7.3	24.6	27.8	52.4	1717	89	294	173	1.31	58.6

presence of zeolite in the soil, becomes slower. Trapping and keeping nitrogen in the soil after a zeolite application was explained by Barbarick and Pirela (1984): NH₄⁺ cations penetrate the zeolite channels, the small size of which do not allow nitrifying bacteria to penetrate. From this perspective, the application of zeolite holds back the chemical change of ammonium cations into nitrate anions, thereby decreasing the amount of nitrate leaching into the groundwater. In this way, it is possible to significantly reduce nitrogen loss, which reaches, according to several authors, 25%–35% of the total amount of applied nitrogen in mineral fertilizers.

In our case, the content of nitrate nitrogen in the soil in the experimental variants was 66% to 78% lower in comparison with the control variant. Statistical analysis of the results according to the Kruskal–Wallis test confirmed the significant differences between the mean values of all observed variants ($H = 13.708$ with $P = 0.003$). The Mann–Whitney test results are given in Table 5.

4. Discussion

We found very fast cation sorption in zeolite but it should be noted that the particle size of the zeolite was 45 µm. The speed of sorption would be very different for larger particles. This is confirmed by Horvathova and Kachnak (1987), who measured the time dependence of heavy metal sorption in zeolite fraction of 0.2–3.2 mm. Stabilization of the exchange equilibrium was reached in only a few hours.

Our results showed a reduction of ammonia nitrogen content in the soil a short time after zeolite application. This reduction of the contents in the experimental variants can probably be attributed to fixation of ammonium ions by specific areas in the zeolite crystal lattice, because they

bind to the zeolite with the greatest intensity of all cations (Cicisvili and Andronikasvili, 1988). Similar findings were reached in our previous works (Torma and Chimic, 1992).

During the 5 months of the experiment, we found higher contents of ammonia nitrogen in the variants with zeolite, which means ammonia nitrogen is gradually released from zeolite and more is available for cultivated plants. Chelischeva and Chelischev (1984) and Tukvadze (1984) found a similar effect after zeolite application, although their experiments contained significantly higher zeolite amounts. Application of 2 t of zeolite in heavy soils increased the hydrolysable nitrogen content twice, and when they used 4 and 6 t of zeolite, it increased by 4 and 5 times, respectively. Sopkova et al. (1993), Ming and Alein (2001), Rehakova et al. (2003), and Uher and Balogh (2004) suggested that fertiliser with the addition of zeolite became a 'slow-releasing fertiliser', although Ruzek and Kovanda (1996) debated the use of the term in relation to zeolite.

The fact that zeolite can relatively strongly fix the ammonia nitrogen leads to the reduction of nitrogen leaching. This is confirmed not only by our results, but also by researchers in many countries who have had positive results with zeolite application decreasing nitrogen loss due to leaching (e.g., Işildar, 1999; Sepaskhah and Yousefi, 2007; Gholamhoseini et al., 2009; Bernardi et al., 2010; Ippolito et al., 2011; Aghaalikhani et al., 2011). Zeolite is also used to remove nitrates from water (Mazeikiene et al., 2010).

Similar results were obtained by Bajrakov and Shevchenko (1984). In their experiments there was an observed decrease of the N-NO₃⁻ in the soil only to the level of 41%–48% after the zeolite application compared with the nitrate nitrogen content in the control variant.

Whereas at the beginning of the experiment statistically significant differences were confirmed only between the control variant and observed variants, in the last month of the experiment they were also confirmed between all observed variants. This was confirmed by a Kruskal–Wallis test with a relatively high level of significance ($H = 13.780$ and $P = 0.003$).

Regarding the obtained results, it is possible to conclude that sorption of cations NH₄⁺, Fe²⁺, Mn²⁺, Ca²⁺, and Mg²⁺ in zeolite with a grain size of less than 45 µm takes place very quickly. More than 90% of the maximum

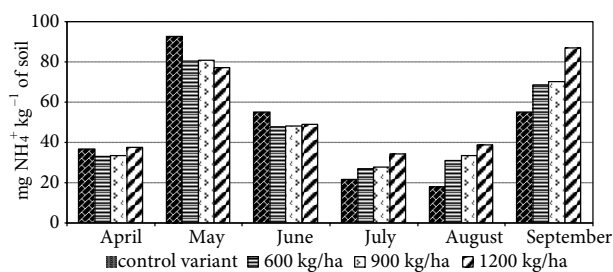


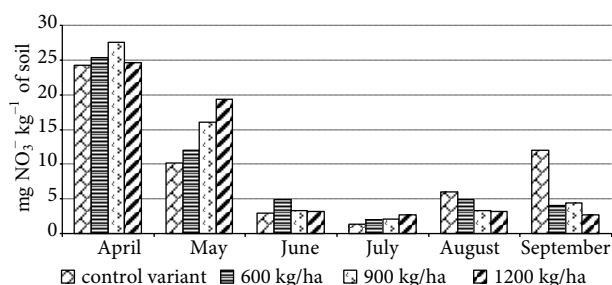
Figure 3. Dynamics of ammonia nitrogen in the soil horizon 0.0–0.3 m after the application of different amounts of zeolite.

Table 3. The main statistical parameters from the last measurement at the end of the field experiment (ammonia nitrogen).

	Control variant	600 kg zeolite ha ⁻¹	900 kg zeolite ha ⁻¹	1200 kg zeolite ha ⁻¹
Minimum	54.50	67.30	69.40	86.10
Maximum	56.10	68.90	70.50	87.40
Median	55.20	68.90	70.25	87.05
Arithmetic mean	55.25	68.50	70.10	86.90
Standard deviation	0.733	0.800	0.483	0.572

Table 4. Mann–Whitney test results of the observed variants (regarding ammonium nitrogen).

	600 kg zeolite ha ⁻¹	900 kg zeolite ha ⁻¹	1200 kg zeolite ha ⁻¹
Control variant	0.000 + (0.029)	0.000 + (0.029)	0.000 + (0.029)
600 kg zeolite ha ⁻¹		0.000 + (0.029)	0.000 + (0.029)
900 kg zeolite ha ⁻¹			0.000 + (0.029)

**Figure 4.** Dynamics of nitrate nitrogen in the soil horizon 0.0–0.3 m after the application of different amounts of zeolite.

possible concentration of those cations is sorbed in the first few minutes. As zeolite has an effect on soil for several years after its application, this can be considered to be a significant fact in the prevention of nitrogen loss from soil.

The dynamics of ammonia nitrogen in the soil during the 5 months of observation justified zeolite's description as a slow-releasing nitrogen fertiliser. A short time after zeolite application, the nitrogen content in the soil decreased as a result of NH_4^+ fixation by zeolite, but after 3 or 4 months the opposite was true, and the increase was

probably caused by the gradual release of nitrogen from the zeolite crystal lattice. Therefore, the zeolite works in the soil as a sort of slow-releasing nitrogen fertiliser with NH_4^+ ions and indirectly limits the intensity of nitrification in the soil. Cultivated plants are thus provided a sufficient amount of nitrogen throughout the vegetation period. On the other hand, the presence of zeolite in the soil inhibits the process of nitrification to a certain extent, so that the nitrate leaching into deeper soil horizons is not so intensive.

Zeolite application to soil favourably affects the environment by preventing the leakage of mineral nutrients (especially nitrogen) into groundwater and increasing the efficiency of nutrients occurring in soil, and it does not negatively affect the quality of the cultivated products. It is fair to say that natural zeolite plays a positive role in plant nutrition.

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Table 5. Mann–Whitney test results of the observed variants (regarding nitrate nitrogen).

	600 kg zeolite ha ⁻¹	900 kg zeolite ha ⁻¹	1200 kg zeolite ha ⁻¹
Control variant	0.000 + (0.029)	0.000 + (0.029)	0.000 + (0.029)
600 kg zeolite ha ⁻¹		1.500 - (0.570)	0.000 + (0.029)
900 kg zeolite ha ⁻¹			0.000 + (0.029)

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