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INFLUENCE OF APPLICATION MINERAL SORBENTS IN SOIL CONTAMINATED WITH NICKEL ON THE CONTENT OF SOME ELEMENTS IN INDIAN MUSTARD

***Maja Radziemska*[1], *Zbigniew Mazur*[2], *Joanna Fronczyk*[3]**

[1] Faculty of Civil and Environmental Engineering, Department of Environmental Improvement, Warsaw University of Life Sciences
[2] Faculty of Environmental Management and Agriculture, Department of Environmental Chemistry, University of Warmia and Mazury in Olsztyn
[3] Faculty of Civil and Environmental Engineering, Department of Geotechnical Engineering, Warsaw University of Life Sciences

K e y w o r d s: halloysite, Indian mustard, macroelements, Ni-contamination, zeolite.

A b s t r a c t

The effects of increasing nickel contamination of soil on selected macroelement uptake by Indian mustard (*Brassica juncea* L. Czern) and application of natural zeolite, raw and modified halloysite were investigated in this experiments. In a vegetative-pot experiment, four different level of nickel contamination, i.e., 0 (control), 80, 160, 240, 320 $mg \cdot kg^{-1}$ were applied in an analytical grade $NiSO_4 \cdot 7H_2O$ solution mixed thoroughly with the soil. The content of nitrogen, phosphorus, sodium, calcium, potassium and magnesium in Indian mustard depended on the dose of nickel and type of neutralizing substance. The average accumulation of tested elements in Indian mustard grown in nickel contaminated soil were found to follow the decreasing order Na>P>Ca>Mg>K>N. The application of MH turned out to be most advantageous, resulting in a small increase in the average content of Na and Mg. Addition of RH and NZ led to the highest increase in the average content of the P.

Address: Maja Radziemska, Faculty of Civil and Environmental Engineering, Department of Environmental Improvement, Warsaw University of Life Sciences – SGGW, ul. 159 Nowoursynowska, 02-773 Warsaw, Poland, phone: +48 (22) 593 53 70,
e-mail: maja_radziemska@sggw.pl

WPŁYW DODATKU SORBENTÓW MINERALNYCH DO GLEBY ZANIECZYSZCZONEJ NIKLEM NA ZAWARTOŚĆ WYBRANYCH MAKROELEMENTÓW W GORCZYCY SAREBSKA

***Maja Radziemska*[1], *Zbigniew Mazur*[2], *Joanna Fronczyk*[3]**

[1] Wdział Budownictwa i Inżynierii Środowiska, Katedra Kształtowania Środowiska, Szkoła Główna Gospodarstwa Wiejskiego w Warszawie

[2] Wydział Kształtowania Środowiska i Rolnictwa, Katedra Chemii Środowiska, Uniwersytet Warmińsko-Mazurski w Olsztynie

[3] Wdział Budownictwa i Inżynierii Środowiska, Katedra Geoinżynierii, Szkoła Główna Gospodarstwa Wiejskiego w Warszawie

Słowa kluczowe: haloizyt, gorczyca sarepska, zeolit, zanieczyszczenie niklem, makroelementy.

Abstrakt

Przedmiotem badań było określenie wpływu wzrastającego zanieczyszczenia gleby niklem oraz dodatku naturalnego zeolitu, modyfikowanego i surowego haloizytu na zawartość wybranych makroelementów w gorczycy sarebska (*Brassica juncea* L. Czern.). W doświadczeniu wazonowym zastosowano cztery wzrastające dawki niklu 0 (kontrola), 80, 160, 240, 320 mg · kg^{-1} wprowadzonych w formie związku $NiSO_4 \cdot 7H_2O$ cz.d.a, który wymieszano z glebą. Zawartość azotu, fosforu, sodu, wapnia, potasu i magnezu w gorczycy sarepskiej zależała od wielkości dawki niklu oraz typu substancji neutralizujących. średnia zawartość badanych makroelementów w gorczycy sarepskiej rosnącej na glebie zanieczyszczonej niklem przyjmowała następującą kolejności Na>P>Ca>Mg>K>N. Aplikacja MH okazała się najbardziej korzystna, powodując wzrost średniej zawartości Na i Mg. Dodanie RH i NZ wywołały największy wzrost średniej zawartości w przypadku P.

Introduction

Nickel (Ni) has two main oxidation states (+2 and +3) and five natural isotopes. Especially dangerous for living organisms as well as plants is the cation Ni^{2+}, occurring in many salts of mineral and organic acids. Nickel has been listed as a priority control pollutant by the United States Environmental Protection Agency (US EPA), and is present in plant tissues in the range of 0.05–10 μg · g^{-1} of dry matter (JAIME et al. 2012, Singh and PRASAD 2015). This element is sorbed by hydrated Al and Fe oxides, organic substance and clay minerals, which results in its accumulation in soil. What is more, nickel from a natural source, in contaminated soils, is less soluble and moves less readily than the remaining elements. Mineral fertilizers, especially phosphorus ones, organic fertilizers, compost, organic waste, mineral waste used to lime soils can be a source of nickel in the natural environment; moreover, volcanic eruptions and wind-blown dust also play a role (BUEKERS et al. 2015, ESTRADE et al. 2015). Additionally, nickel can also be derived from anthropogenic activities, e.g. metal mining, vehicle emissions, industrial wastes, paint manufacturing, coal combustion (ELOUEAR et al. 2009, VAVERKOVÁ and ADAMCOVÁ 2014, SCHORNÍK et al. 2015).

Another effect of the harmful influence of nickel are disturbances in the proportions of the chemical composition of plants, which can lead to the excessive, toxic accumulation of this element and reduction in the general sum of cations in plant tissues and an increase in the amount of calcium and phosphorus (RADZIEMSKA et al. 2013).

The effectiveness of phytoremediation methods largely depends on the choice of an appropriate plant species, characterized by a high tolerance and high accumulation capability of heavy metals in its mass (LIU et al. 2010). *B. juncea* (L.) is a high biomass crop species – at least 10 times higher than other hyper accumulators, able to tolerate and accumulate high concentrations of potentially toxic trace elements (NOVO and GONZÁLEZ 2013, NOVO et al. 2013a, 2013b, KARAK et al. 2013). MINGLIN et al. (2005) reported that it is the most promising and interesting plant model for phytoremediation, as it accumulates more than 400 $\mu g \cdot g^{-1}$dry weight (DW) of cadmium in its shoot. *B. juncea* (L). might also be a plant suitable for phytoremediation of Pb-contaminated soils and waters in spite of high mercury phototoxicity (SHIYAB et al. 2009).

The present research objective was to investigate the effects of mineral sorbents, i.e. raw halloysite (RH), modified halloysite (MH), and natural zeolite (NZ) on the content of selected elements in above-ground parts of Indian mustard *B. juncea* (L.) grown in Ni-contaminated soil.

Materials and Methods

Experimental system description

The experiment was assessed under the conditions of a pot experiment in an acclimatized greenhouse, with two factors and fourfold replication. The first factor was the addition of increased doses of Ni to soil (0, 80, 160, 240, and 320 $mg \cdot kg^{-1}$), introduced in the form of chemically pure aqueous solutions of nickel sulphate heptahydrate ($NiSO_4 \cdot 7H_2O$) (Sigma-Aldrich). The second factor consisted of the addition of three mineral adsorbents, i.e., raw halloysite, modified halloysite, and natural zeolite (3.0% w/w). Soils without nickel and amendments (0.0%) were designated as the control. Non-polluted soil for the pot experiment were collected at a depth of 0–20 cm from farmland in the vicinity of Olsztyn, Poland, (53°35'45''N, 19°51'06'E). The chemical properties of the soil are shown in Table 1. The soil was air-dried, passed through a 1-cm sieve and packed into 20-cm-diameter and 26-cm-height experimental pots

Table 1

Physical and chemical parameters of the experimental soil

Soil chemical parameters	
pH	4.80
Hydrolytic acidity ($mmol \cdot kg^{-1}$)	33.75
Sum of exchangeable bases Ca^{++}, Mg^{++}, K^{+}, Na^{+} ($mmol \cdot kg^{-1}$)	62.20
Cation exchange capacity ($mmol \cdot kg^{-1}$)	95.95
Base saturation (%)	64.80
Organic matter	
Organic carbon ($g \cdot kg^{-1}$)	7.13
Total N ($g \cdot kg^{-1}$)	1.04
Carbon:Nitrogen	6.85
$N\text{-}NH_4^{+}$ ($mg \cdot kg^{-1}$)	21.18
$N\text{-}NO_3^{-}$ ($mg \cdot kg^{-1}$)	9.88
Soil texture (%)	
Fractions 2.0–0.05 mm	86.6
Fractions 0.05–0.002 mm	11.2
Fractions 0.002 mm	2.2
Trace metal ($mg\ kg^{-1}$)	
Nickel	4.05
Copper	8.49
Chromium	10.95
Zinc	24.21
Lead	16.33
Manganese	210.9
Available forms ($mg\ kg^{-1}$)	
Phosphorous	46.6
Potassium	8.20
Magnesium	33.9

(10 kg soil per pot); it was then used for physical and chemical analysis, as well as N, P, K, Mg, Ca, Na concentration analysis.

The polyethylene pots were maintained under natural day/night conditions; during the day (14h), the air temperature was 26±3°C and approximately ten degrees lower (16±2°C) at night (10h), with a relative humidity of 75±5%. The plants were watered every other day with distilled water to 60% of the maximum water holding capacity of the soil. The plants were harvested after 100 days, and soil and sorbents were collected.

The seeds of *B. juncea* (L.) cv. Małopolska, were obtained from an authorized Seed Production Centre in Olsztyn, Poland (OLZNAS-CN Sp. z o.o.), and were planted at the quantity of $n=5$ per pot. Soil was fertilized with a macro- and micronutrient fertilizer mixture ($g\ kg^{-1}$) containing N-26%, K_2O-26%, B-0.013%, Cu-0.025%, Fe-0.05%, and Mn-0.025%. The above-part of Indian mustard was harvested in the flowering phase and plant material samples were collected for laboratory tests.

Sample preparation and element content analysis

In the laboratory, plant samples were thoroughly rinsed, first with tap water and then with deionized water to remove dust and soil particles. After oven drying (60°C, 48h) the plants were weighed (DW) and powdered using an analytical mill (A11 IKA, Germany) preceding the chemical analyses. The samples were kept at an ambient temperature until analysis. All reagents were of analytical reagent grade unless otherwise stated. Ultra-pure (UP) water (Millipore System, USA) of 0.055 $\mu S \cdot cm^{-1}$ resistivity was used for preparing the solutions and dilutions.

Total nitrogen content was tested for by means of Kjedahl's method after mineralization in concentrated sulfuric (VI) acid using hydrogen peroxide as a catalyst (BREMNER 1965). Phosphorus (P) was assessed by colorimetric analysis, using the vanadium-molybdenum method (Cavell 1955); sodium (Na), calcium (Ca), potassium (K) – atomic emission spectrometry, AES method (SZYSZKO 1982), magnesium (Mg) – atomic absorption spectrometry, AAS method (SZYSZKO 1982).

Statistical analysis

Statistical analysis was performed using the software Statistica (StatSoft, 2010). Differences of means between treatments were tested by ANOVA and comparisons of means using LSD test, at $p=0.05$. The means and standard deviations (±SD) of five replications are reported.

Results and Discussion

In the presented research, nitrogen (N) content in the above-ground plant mass of *B. juncea* (L.) was determined by the dose of nickel as well as the addition of natural zeolite (NZ) as well as raw (RH) and modified (MH) halloysite (Table 2). In the control series (without additives), the differences in nitrogen content were additionally correlated with the increasing doses of this element. Soil contamination amounting to 320 mg $Ni \cdot kg^{-1}$ soil caused the highest increase in nitrogen in the analyzed plant. Studies by Karimi et al. (2003) also indicated that the nitrogen concentration of *Vicia faba* L. and *Brassica arvensis* L. was not reduced by nickel at these levels, in contrast to previous studies conducted by ARDUINI et al. (2006). In the presented studies, all of the immobilizing additives applied in the experiment (NZ, RH and MH) induced an increase in the nitrogen content of above-ground parts of Indian mustard plants. However, this effect was most significant in the case of the raw halloysite (RH) additive. Modified halloysite (MH) in soils with a dose of 320 mg Ni · kg^{-1} soil resulted in an over threefold increase in the nitrogen content of *B. juncea* (L.) as compared to the group to which neutralizing substances had not been applied. In an experiment conducted by WYSZKOWSKI and RADZIEMSKA (2010), the applied contamination alleviating substances (especially natural zeolite) stimulated an increase in the total nitrogen content, more evidently in *Zea mays* L. than in *Lupinus luteus* L. In another study carried out by WYSZKOWSKI and RADZIEMSKA (2013), zeolite and CaO generally caused concentrations of nitrogen compounds to increase in oat grain and straw, in contrast to the roots, where they were typically lower. The average share of nitrogen in the analyzed macronutrients increased from 30.62% (control) to 48.23% (320 mg Ni · kg^{-1} soil) (Fig. 1). Differences between the mineral sorbents and the control were not noted at the same dose of Ni ($P<0.05$).

Table 2

Effect of nickel and various mineral sorbents on N, P, K, Ca, Na and Mg concentration in Indian mustard, (g · kg^{-1} dry mass)

Treatment		Nickel concentration (mg · kg^{-1})					Mean
		0	80	160	240	320	
N	Control	11.44±0.81[a]	13.48±1.15[a]	19.40±2.32[a]	25.31±3.91[a]	29.1±5.42[a]	19.75
	NZ	10.54±0.72[a]	12.73±1.82[a]	17.92±1.99[a]	25.74±4.08[a]	30.1±6.10[a]	19.41
	RH	11.05±1.04[a]	13.24±1.52[a]	18.51±2.81[a]	27.32±4.44[a]	32.1±6.04[a]	20.44
	MH	11.32±0.78[a]	13.44±1.15[a]	18.46±2.45[a]	26.23±4.08[a]	31.0±5.79[a]	20.09
P	Control	3.66±0.21[a]	3.79±0.32[a]	4.12±0.41[a]	4.48±0.47[a]	3.66±0.54[a]	3.94
	NZ	3.82±0.19[a]	3.81±0.38[a]	4.05±0.43[a]	4.66±0.46[a]	4.51±0.61[a]	4.17
	RH	3.18±0.15[b]	3.98±0.42[a]	4.01±0.45[a]	4.73±0.44[a]	4.02±0.77[a]	3.98
	MH	3.28±0.22[b]	3.32±0.29[b]	3.89±0.33[a]	4.44±0.39[a]	4.29±0.68[a]	3.84
K	Control	12.52±0.65[a]	14.11±0.73[a]	17.56±1.46[a]	20.04±1.52[a]	21.05±2.06[a]	17.06
	NZ	13.11±0.96[a]	15.08±1.01[a]	18.11±1.14[a]	20.52±1.43[a]	16.61±1.75[b]	16.69
	RH	13.62±1.11[a]	15.31±1.32[a]	19.62±1.05[b]	21.14±1.52[a]	19.62±2.00[a]	17.86
	MH	12.94±0.99[a]	14.86±1.22[a]	17.98±0.99[a]	18.49±1.48[a]	18.90±1.93[a]	16.63
Ca	Control	3.92±0.24[a]	4.03±0.32[a]	3.86±0.40[a]	4.08±0.32[a]	3.71±0.33[a]	3.92
	NZ	3.97±0.33[a]	3.98±0.41[a]	4.00±0.44[a]	4.14±0.42[a]	4.24±0.49[b]	4.07
	RH	3.59±0.25[a]	3.38±0.32[b]	3.78±0.45[a]	4.12±0.54[a]	4.28±0.55[b]	3.83
	MH	3.73±0.21[a]	3.39±0.29[b]	3.89±0.33[a]	4.14±0.49[a]	4.24±0.41[b]	3.88
Na	Control	1.32±0.11[a]	1.46±0.16[a]	1.60±0.20[a]	1.51±0.22[a]	1.73±0.21[a]	1.52
	NZ	1.20±0.17[b]	1.44±0.27[a]	1.59±0.27[a]	1.70±0.36[a,b]	1.73±0.31[a]	1.53
	RH	1.32±0.12[a]	1.48±0.25[a]	1.51±0.32[a]	1.50±0.27[a]	1.64±0.40[a]	1.49
	MH	1.29±0.19[a]	1.40±0.31[a]	1.43±0.29[a]	1.82±0.24[b]	1.80±0.31[a]	1.55
Mg	Control	3.32±0.11[a]	3.03±0.32[a]	3.01±0.41[a]	3.15±0.57[a]	3.06±0.47[a]	3.11
	NZ	3.45±0.19[a]	3.51±0.43[a]	3.61±0.53[b]	3.60±0.46[a]	3.91±0.61[b]	3.62
	RH	3.55±0.35[a]	3.72±0.48[a]	3.72±0.55[b]	3.57±0.68[a]	4.02±0.83[b]	3.72
	MH	3.66±0.36[a]	3.62±0.29[a]	3.86±0.46[b]	3.60±0.59[a]	4.05±0.61[b]	3.76

Mean values for five samples (±standard deviation) are shown. Data within columns that do not have common indices are significantly different at $p<0.05$

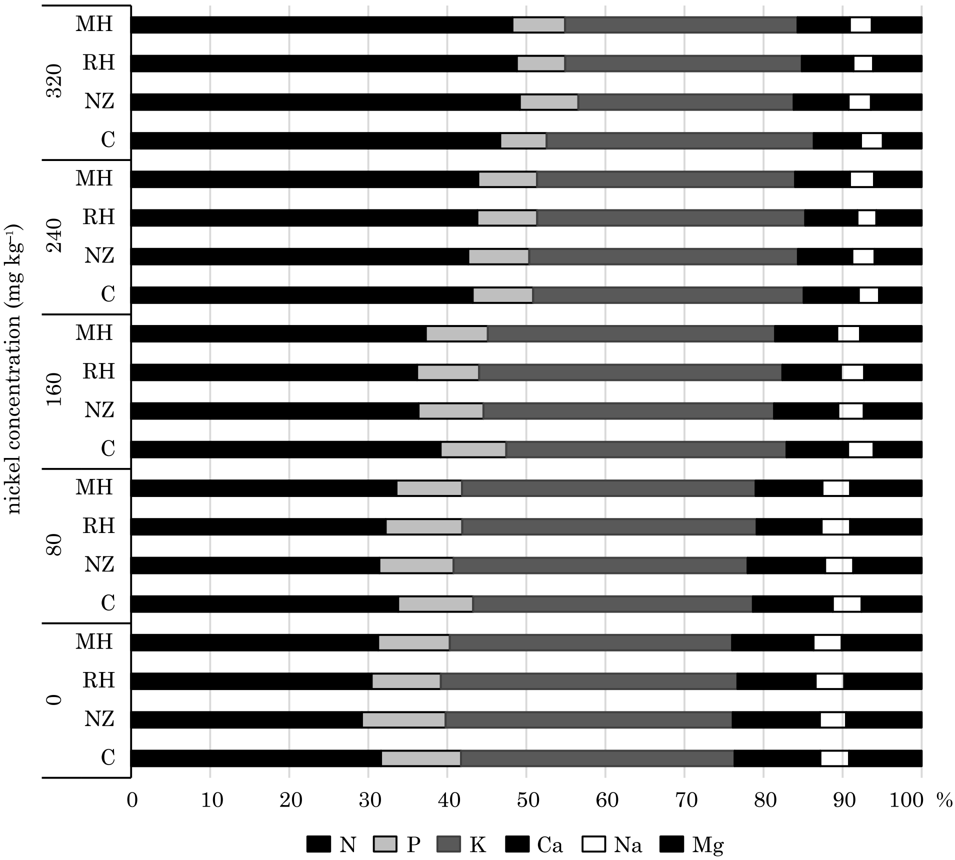

Fig. 1. The average percentage share of N, P, K, Ca, Na and Mg in above-ground parts of Indian mustard cultivated in Ni-polluted soil

Phosphorus content (P) in the above-ground parts of Indian mustard was significantly influenced by: the dose of soil contamination with nickel as well as the mineral substances in the form of natural zeolite, and raw and modified halloysite (Table 2). In the series without neutralizing additives, increasing doses of Ni only insignificantly influenced phosphorus content in the analyzed plant. Contamination of soil at 240 mg Ni · kg^{-1} soil caused the highest increase in phosphorus content (+22%) in *B. juncea* (L.). The addition of raw halloysite (+13%) and natural zeolite (9%) led to the highest increase in the average content of the analyzed element in relation to pots without neutralizing additives. In another study of RADZIEMSKA et al. (2013), modified halloysite in crops subjected to doses of 160 and 240 mg Ni · kg^{-1} soil led to a nearly twofold increase in the phosphorus content of *Zea mays* L. in relation to plants without the addition of neutralizing substances. The average percentage share of P in the total of the analyzed macronutrients decreased along with the nickel content of soil, ranging from 9.63-6.52% (Fig. 1). The increase in the concentration of phosphorus in plants contaminated with nickel was confirmed by studies of MATRASZEK et al. (2002) and KARIMI et al. (2013).

The application of nickel to soil on the whole led to increased potassium contents in plants as compared to the control series – without alleviating substances (Table 2). An almost twofold increase in K concentration was confirmed in *B. juncea* (L.) grown in pots with the highest dose of Ni (320 mg · kg^{-1}). Research by PALOCIS et al. (1998) confirm increased contents of potassium in tomatoes under the influence of nickel contamination. In the presented studies, among the substances added to the soil to alleviate nickel contamination, raw halloysite (RH) turned out to be the best, leading to a 31% increase in the average content of the described element in the test plant as compared to the control series. An analogical situation was observed in the case of natural zeolite (NZ) and modified halloysite (MH) additives, although their influence was smaller. In soils contaminated at the level of 240 and 320 mg Ni · kg^{-1} with the addition of MH, the content of the analyzed element in Indian mustard plants was approximately 10% lower than in plants grown in soil free of contamination and additives. The percentage share of potassium in the sum of N, P, K, Ca, Mg and Na was similar and ranged from 36.94–36.72% with the exception of the group with the highest dose of Ni in soil, where its share was lower, i.e. 30.08% (Fig. 1). The variable influence of the different doses of Ni in soil on K content in plants was reported by MATRASZEK et al. (2002) and KARIMI et al. (2013).

The Ni dose as well as the addition of NZ, RH and MH shaped Ca content in Indian mustard, (Table 2). In the series lacking additives (control) and increasing nickel contamination, calcium content in the tested plant was not characterized by a clear direction of values. In this series, plants grown in soil contaminated by nickel at a level of 240 mg Ni · kg^{-1} was characterized by the highest calcium content. Studies of CROOKE (1955) and PALOCIS et al. (1998) also confirm the stated dependency, observing an increase in Ca content in the biomass of oats under the influence of increasing doses of nickel. The carried out studies indicate that using substances (NZ, RH and MH) that alleviate nickel contamination had an influence on the average calcium content in the above-ground mass of Indian mustard. The listed additives had the highest influence in the case of study groups containing the highest doses of nickel. In relation to the control group, the application of RH and MH turned out to be most beneficial, leading to an 8% increase in average calcium content in the test plant. The average percentage share of calcium decreased along with the increase in Ni concentration in soil and ranged from (0 mg Ni · kg^{-1}) to 6.51% (320 mg Ni · kg^{-1} soil) (Fig. 1). A decrease in the percentage share of Ca in the leaves of plants in the *Brassicaceae* family under the influence of nickel was confirmed by PUTNIK-DELIĆ et al. in their studies (2014).

Based on the carried out studies, it turns out that using alleviating substances (natural zeolite, raw and modified halloysite), the contamination of soil with nickel had a significant influence on Na content in the above-ground mass of Indian mustard, (Table 2). In the control series (without additives), a positive correlation between the sodium content of the tested plant and increasing soil contamination with Ni occurred. The alleviating substances used in the experiment had a beneficial effect on the average sodium content in Indian mustard plants. Among the substances alleviating nickel contamination added to soil, the application of modified halloysite (MH) turned out to be most advantageous, resulting in a small increase in the average content of Na. The average percentage share of this element ranged from 2.71% in the group with a dose of 240 mg Ni $\cdot$ kg^{-1} soil, to 3.54% in group 0 (Fig. 1). Insignificant percentage changes in Na content in the sum of macroelements (N, P, K, Ca, Mg and Na) in the leaves of wild mustard (*Brassica arvensis* L.) growing on soil contaminated with various doses of nickel were obtained by KARIMI et al. (2013).

Natural and modified halloysite, and zeolite, as well as increasing doses of nickel, significantly affected the magnesium content of Indian mustard, (Table 2). Nickel can displace Mg from chrolorphyll and enzymes (FURINI 2012). The content of Mg in research by PUTNIK-DELIĆ et al.(2014) increased in several brassica species tested under conditions of excessive Ni contamination. The content of Mg in the described plant in the control series – without alleviating additives, was negatively correlated with increasing doses of nickel. The opposite dependency was noted by MATRASZEK et al. (2002), in whose studies magnesium content in the leaves of spinach increased along with the influence of increasing doses of nickel. On the other hand, studies by PALOCIS et al. (1998) prove that Mg content in oats under the influence of increasing doses of nickel also exhibited a decreasing tendency, as in the present research. Applying alleviating substances had a positive effect on average magnesium content in above-ground parts of *B. juncea* (L.). The addition of modified halloysite (MH) was found to have the most beneficial effect, leading to the highest increase in the average magnesium content of the test plants as compared to the control series. Row halloysite (RH) and natural zeolite (NZ) also had a positive effect, although to a lesser degree. The lowest average percentage share of magnesium (5.77%) in the sum of analyzed macronutrients in the above-ground parts of mustard was noted in the group treated with a 240 mg Ni $\cdot$ kg^{-1} soil dose of nickel, whereas the highest (9.66%) occurred in the control group (Fig. 1). A changeable percentage share of Mg, depending on the part of the plant, the plant species and Ni concentration of soils, was noted by KARIMI et al. (2013) and PUTNIK DELIĆ et al. (2014) in their research.

Conclusions

The content of macroelements (N, P, Na, Ca, K and Mg) in Indian mustard depended on the dose of the nickel contaminant and the application of alleviating substances incorporated into the soil. In the control series (without the addition of natural zeolite, row halloysite, modified halloysite), the differences in nitrogen, phosphorous, potassium, and sodium content were positively correlated with the increasing doses of Ni-contamination. Soil with 320 mg of Ni per 1 kg of soil led to the highest increase in nitrogen, phosphorous, potassium and mgnesium content in the above-ground parts of Indian mustard. The application of modified halloysite (MH) turned out to be most advantageous, resulting in a small increase in the average content of sodium and magnesium. Moreover, the addition of raw halloysite (RH) and natural zeolite (NZ) led to the highest increase in the average content of the phosphorus, in relation to pots without neutralizing additives.

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