Assiut J. Agric. Sci., (49) No. (3) 2018 (117-127) *ISSN: 1110-0486*
Website: www.aun.edu.eg/faculty_agriculture/journals_issues_form.php *E-mail: ajas@aun.edu.eg*

Use of Sewage Sludge Treated with some Stabilizers in Reducing Nickel Availability in the Soil and Absorption by Plants

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Received on: 12/7/2018 **Accepted for publication on:** 9/8/2018

Abstract

Sewage sludge was treated with some Ni stabilizers including silica gel (Si), cement by- pass (CB) and iron oxide (FO) to reduce its availability in the soil, and absorption by onion plants (direct effect) in the first season and Jew's mallow plants (residual effect) in the second season. A loamy sand soil was amended only before the first season with sewage sludge treatments including 10 and 20% Si, CB and FO treated sludge as well as Si + CB, Si + FO and CB + FO treated ones.

Sludge treated with 20% Si was the best one in reducing Ni effects in the soil and plants, it also increased the length, shoot and bulb fresh weights, shoot and bulb dry weight of onion plants by 17.2, 148.5, 6.99, 127.8 and 159.2%, respectively, compared to the control. However, the untreated sludge application increased the soil extractable Ni from 0.30 mg/kg in the control to 2.79 mg/kg after the harvest of onion plants. Amending the soil with sludge treated with FO + CB, Si+ CB and Si+ FO significantly reduced the soil extractable Ni 100% of that amended with untreated sludge after onion harvest. The extractable Ni significantly decreased after Jew's mallow harvest from 0.30, 2.79, 1.15, 3.47 and 2.52 mg/kg to 0.00, 0.00, 0.00, 0.52 and 1.27 mg/kg with the control, untreated sludge, 10% CB, 20% CB and 10% FO treatments, respectively.

In the first season (direct effect), sludge amended with Si+ FO was the best treatment that reduced Ni content of the shoots and bulbs by 71.6 and 75.6% compared to the control. Moreover, in the second season (residual effect), sludge amended with 20% Si was the best treatment that reduced the Ni content of Jew's mallow plants by 71.8% and 66.8% compared to the control.

Keywords: *Nickel, sewage sludge, stabilization, heavy metals, residual effect*

Introduction

Sewage sludge (biosolids) represents a good source of nutrients for plant growth due to its high content of nutrients and organic matter. It is also considered as a good soil conditioner that improves the physical, chemical, and biological properties of soil (Alcantara *et al.*, 2009; Angın and Yağanoğlu, 2009). Land application of biosolids materials is a common practice in many countries that allow the reuse of biosolids produced by urban populations (Weggler *et al.*, 2004). Heavy metals that are commonly found in biosolids includelead (Pb), nickel (Ni), cadmium (Cd), chromium (Cr), cupper (Cu), and zinc (Zn). The metal concentration is governed by the nature and the intensity of the industrial activity, as well as the process type employed during the biosolids treatment (Mattigod and Page, 1983). Municipal wastes such as sewage biosolids have been used as an amendment for agricultural soils for several years. They are often considered an economically attractive alternative to disposal through land-fill and incineration (Diacono and Montemurro 2010). Using sewage sludge as a fertilizer enables cost re-ductions of nitrogen and phosphorus mineral fertilizers and may improve crop yields in sludge treated fields (Wild and Jones, 1991; Petersen *et al.,* 2003).

The only drawback in the use of sewage sludge on agricultural land is its pollutant load including heavy metals, organic compounds, pharma-ceuticals and pathogens. Potentially toxic elements are a general term for a wide range of metalssuch as cad-mium, copper, nickel, lead, zinc, mercury and chromium that originate in sewage. Heavy metal pollution represents an important environ-mental problem due to their toxic ef-fects resulting in serious ecological and health problems. The metals are concentrated in the sludge as a result of their association with settable sol-ids during the primary and secondary treatment processes (Sterritt and Les-ter, 1980). Research on the land ap-plication of sewage sludge has mainly focused on its role in the introduction of heavy metals in the food chain. The regulatory authorities normally accept remediation strategies that center on reducing the metal bioavailability only if the reduced bioavailability is equated with re-duced risk, and if the bioavailability reductions are according to be long-term (Martin and Ruby, 2004).

Udeigwe *et al.* (2011) reported that the immobilization mechanisms of contaminants such as heavy metals could include: 1) Preciptaton as salts, 2) the co-precipitation, 3) the surface precipitation, 4) and the adsorption to mineral surfaces stable complexes. The net negative charge of mineral surfaces increases when a specific adsorption of anions occurs leading to an increase in the retention of metal cations such as Cd^{2+}, Cu^{2+} and Zn^{2+} (Boland *et al*., 1999).

Nanoparticles of magnetite (an iron oxide mineral) have a unique su-per paramagnetism and a high surface area (Yuan *et al*., 2009) which offer more multiple surface sites for ad-sorption or interaction the iron oxide mineral as (Paljevac *et al.,* 2007) that makean innovative immobilization carrier (Sulek *et al.,* 2010; Xu *et al*., 2012). Sen *et al.* (2002) indicated that the higher adsorption of Ni^{2+} on iron oxides compared to that of $Cu^{2+}$is due to the relatively smaller size of Ni^{2+} ions. In addition, iron oxide minerals are better adsorbents compared to-kaolinnes due to the specific adsorp-tion of the metal that occurs on the iron oxides which is induced by the inner – sphere complexation.

Silica gel has a large specific surface area and fast adsorption kinet-ics (Jiang *et al*., 2007). It also pos-sesses a highly porous and rigid tex-ture (Repo *et al*., 2011; Waseem *et al*., 2011). In addition, silica gel sur-face consists of inter connected parti-cles forming a three dimensional skeleton. (Fenglian and Wang, 2011).

Cement By-pass is a product of the cement industry. In Assiut cement company, it consists of 11.88% SiO_2, 2.97% Al_2O_3, 2.60% Fe_2O_3, 47.8% CaO, 0.68% MgO, 12.13% SO_3, 2.28 Na_2O, 4.38% K_2O and 4.81% Cl (Amin, 2013). Abou-Seeda *et al.* (2005) reported that the available soil

Pb, Cd, and Ni were decreased by 10-20, 30-40 and 25-30%, respectively, with the application of cement dust. Moreover, treating the sludge with the cement kiln dust reduces the solubility and increases the immobilization of heavy metals in the treated sludge matrix (Emmerich *et al.,* 1982). Kigel *et al.* (1994) also showed that the application of cement kiln dust stabilized chromium in waste sludge.

Therefore, this study aims to evaluate the Ni availability and content of onion (direct effect) and Jew's mallow (residual effect) plants grown in a sandy soil amended with sewage sludge treated with some stabilizers (silica gel, cement by-pass and iron oxide).

Materials and Methods

1- Sewage sludge treatments

Three materials (silica gel "Si", cement by-pass "CB" and ferric oxide "FO") were used as stabilizers in order to reduce Ni availability from sewage sludge. Samples of sewage sludge were collected from El-madabigh sewage plant, Assiut, air–dried, ground using a wooden roller and sieved through a huge sieve plate. The fine tested sludge contained 51.7% organic matter, 6.0% N, 1.7% P_2O_5, 0.6%, K_2O, 11.2% CaO and 6.0 % MgO. The total heavy metals contents of the sludge were 15285, 623, 335, 3.1, 249, 0.9 and 58.7mg/kg for Fe, Zn, Cu, Cd, Pb, Cr and Ni, respectively. The fine sewage sludge was treated and thoroughly mixed with each stabilizer material at levels of 10 and 20%. Other treatments were prepared from treating and mixing the sewage sludge with two stabilizer materials, each one at a level of 10% to produce a total of 9 treatments (Table 1). For three weeks, the treated sewage sludge materials were weighed every day to add the amount of water lost during the sieving and mixed. Then, they were transferred to the lab and air – dried for another week. The nickel content of these treated sludges was extracted using 0.1M ethylene diamin tetra acetic acid (EDTA) according to Wear and Evans (1968) and then, determined (Table 1).

Table 1. The EDTA-extractable Ni (mg/kg) in the sludge materials treated with iron oxide (FO), cement-bypass (CB) and silica gel (Si)

Treatments	EDTA- extractableNi (mg/kg)
control	15.30
FO 10%	16.50
FO 20%	13.00
CB 10%	12.30
CB 20%	12.10
Si 10%	13.80
Si 20%	11.60
FO 10% + CB 10%	12.20
FO 10% + Si 10%	14.90
CB 10% + Si 10%	10.90

2- Pot Experiments:

Pot experiments were conducted in the green house to evaluate the effect of the stabilizer treated sewage sludge on the Ni availability and content of Ni in onion (*Allium Cepa*) plants (direct effect) and Jew's mallow (*Chorcoruis Olitorus*) plants (residual effect) grown in the sandy soil. In the first season (October, 2016), onion seedlings were planted in pots filled with 5 kg of soil amended with the sludge treated with the previous different stabilizer at a level of 20 t/fed. Therefore, as it was mentioned previously, the soil was amended with 9 treatments of sewage sludge (Table 1). Each treatment was replicated 3 times.The texture of used soil was loamy sand (13.68% clay, 4.00% silt and 84.32 % sand) with pH of 7.18, EC (1:5 soil extract) of 3.22 dsm^{-1} and $CaCO_3$ content of 13.85%. The soil also contained 1.10, 0.60, 12.25, 2.15, 7.82 and 2.54 $meqL^{-1}$ of Na^{+}, K^{+}, Ca^{2+}, Mg^{2+}, CO_3^{2+} and Cl^{-}, respectively (Jakson, 1973). After 12 weeks the onion plants were harvested and the bulbs were separated from shoots, washed, dried at 70°C, ground and stored for chemical analysis. On the same pots without any new applications of the treated sewage sludge, 20 seeds of Jew's mallow were planted in July, 2017 to investigate the residual effect of the previous sludge treatments on the availability and content of Ni in these plants. After 12 weeks, the plants were harvested, washed, air dried, oven dried at 70°C, ground and stored for chemical analysis. Soil samples were collected after each growing season, thoroughly mixed, air-dried, ground to pass a 2-mm sieve and stored for chemical analysis. Nickel was extracted from the soil using EDTA method according to Wear and Evans (1968). All plant samples were digested using a mixture of H_2O_2, selenium, lithium sulfate and concentrated sulfuric acid (Parkinson and Allen, 1975). Nickel was determined, in both soil and plant digests using Flame Atomic Absorption Spectrophotometer (GBC).

This study was conducted in a simple experiment and the treatments were arranged in a completerandomized design (CRD) with three replications using SAS package version 9.0. The MSTATC 2.10 computer program prepared by Freed (1992) was used to perform the analysis of variance. The obtained data were subjected to statistical analysis of variance according to Gomez and Gomez (1984) and the means of treatments were tested using the least significant difference method (LSD) at P= 0.05.

Results and Discussion

1- Direct effects of treated sludge-materials on some vegetative properties of onion plants (first season):

Compared with the control (the soil without any treatment), the untreated sludge decreased plant length, shootand bulb fresh weight, shoot and bulbs dry weight of onion plants by 12.3, 47.8, 70.2, 33.3 and 37.0%, respectively (Table 2). This may be due to the toxic effects of heavy metals in sludge according to DeVries and Tiller (1978), significant concentrations of Cd, Cu, Mn, Ni, Pb and Zn were found in the edible parts of lettuce tops and onion bulbs that were grown on soils amended with sewage sludge.

Table 2. Direct effects of sludge treated with cement bypass (CB), ferric oxide (FO) and silica gel (Si) on some vegetative properties of onion plants

Treatment	Plant length (cm)	Fresh weight (g/pot)			Dry weight (g/pot)		
		shoots	Bulb	Total	shoots	Bulb	Total
Control	30.50	8.51	9.15	17.65	2.73	1.35	4.08
Un- Sludge	26.75	4.44	2.72	7.16	1.82	0.85	2.67
CB 10%	25.94	3.59	4.54	8.13	0.71	0.79	1.50
CB 20%	29.01	5.16	6.94	12.10	1.68	1.50	3.18
FO 10%	20.00	2.83	1.17	3.99	1.27	0.35	1.62
FO 20%	23.75	4.82	4.68	9.50	3.11	2.34	5.45
Si 10%	31.00	10.22	6.16	16.38	3.21	1.68	4.89
Si 20%	35.75	21.15	9.79	30.94	6.22	3.50	9.72
FO + CB	32.75	11.80	9.69	21.49	3.87	1.63	5.50
Si + CB	29.75	9.11	6.03	15.14	5.27	2.91	8.18
Si + FO	37.88	7.02	8.20	15.22	2.28	1.88	4.16
$LSD_{0.05}$	1.17	0.71	0.82	0.75	0.70	0.78	1.22

The application of sludge treated with silica gel + iron oxide (Si+FO) and 20% silica gel (Si) significantly increased onion length from 30.50 cm for the control and from 26.75 cm for the untreated sludge to 37.88 cm and 35.75 cm, respectively. The sludge treated with 20% silica gel was the best treatment,it increased the onion total length, shoot fresh weight, bulb fresh weight, shoot dry weight and bulb dry weight by 17.2, 148.5, 6.99, 127.8 and 159.2%, respectively, compared to the control. The respective increases were 33.6, 376.4, 259.9, 241.8 and 311.8%, compared to the untreated sludge treatment. The application of sludge treated with iron oxide and cement by-pass (FO+CB) also significantly increased the plant length, shoot and bulb fresh weights by 22.4, 165.8 and 256.3%, respectively, compared to the untreated sludge.

2- Residual effects of treated sludge materials on some vegetative properties of Jew's mallow plants (second season):

The residual effect of untreated sludge showed the same decrease trend that was indicated for onion plants (direct effect) showed with respect of the length, fresh and dry weights of Jew's mallow plants compared to the control treatment (Table 3).

Table 3. Residual effects of sludge treated with cement by-pass (CB), iron oxide (FO) and silica gel (Si) on some vegetative properties of Jew's mallow plants

Treatment	Total Length (cm)	Fresh Weight (g/pot)	Dry Weight (g/pot)
Control	10.0	2.74	0.26
Un- Sludge	9.0	1.56	0.24
CB 10%	16.3	5.20	2.10
CB 20%	22.7	10.40	2.44
FO 10%	18.0	1.44	0.36
FO 20%	24.0	12.62	5.16
Si 10%	27.0	36.60	8.60
Si 20%	32.3	13.92	2.98
FO + CB	25.3	7.64	1.64
Si + CB	31.4	9.80	2.52
Si + FO	25.5	9.74	3.16
$LSD_{0.05}$	1.4	0.49	0.06

The respective decreases were 10, 43.1 and 7.7% for the vegetative properties. The residual effect of untreated sewage sludge also exhibited the same trendrecorded for the direct untreated sewage application but with lower reduction effects on Jew's mallow vegetative properties which it may be attributed to the lower residual contents of heavy metals in the second season. Treating the sludge with any of the investigated materials resulted in significant increases in the plant length, fresh and dry weights except the 20% FO amended sludge treatment. The plant length reached 32.25 cm using the sludge treated with 20% Si resulting in an increase of 222.5% compared to the control. The level of plant uptake, bio-accumulation and tolerance of plants to heavy metals varies among different crops at different application levels of sewage sludge (Yilmaz, *et al.,* 2012).

3- Direct and residual effects of treated sludge materials on nickel content of onion (first season) and Jew's mallow (second season) plants:

The Ni content of onion shoots and bulbs (first season) and Jew's mallow plants (second season) grown on the soil amended with stabilizer treated sludges are shown in Table (4). In the first season (direct effect), the Si+FO treated sludge was the best treatment which reduced the Ni content of the shoots and bulbs by 71.6 and 75.6, respectively, compared to the control and by 87.0 and 68.7%, respectively, compared to the untreated sludge. In the second season it was also one of the best treatments in reducing Ni content of Jew's mallow plants by 60.7% compared to the control. In addition, in the first season, treating the sludge with 10 and 20% of FO significantly decreased the Ni content by 72.2 and 63.2%, respectively, for onion shoots and by 21.3 and 50.2%, respectively, for onion bulbs compared to the untreated sludge.

In the second season, the Si+FO treated sludge was the best treatment which significantly reduced the Ni content of Jew's mallow plants by 71.8 and 60.5% compared to the control and the untreated sludge, respectively. However, both 20% FO and 10% Si treated sludges reduced the Ni content of Jew's mallow plants by 56 % compared to the control. Sen *et al.* (2002) indicated that the higher adsorption of Ni^{2+} on iron oxides compared to that of $Cu^{2+}$is due to the relatively smaller size of Ni^{2+} ions. In addition, iron oxide minerals are better adsorbents compared to kaolin ones due to the specific adsorption of the metal that occurs on the iron oxides which is induced by the inner-sphere complexation. The results also showed that Jew's mallow plants could accumulate higher amounts of Ni in their tissues compared to onion plants. It seems that Jew's mallow plants have the ability to secrete root exudates that may chelate non exchangeable Ni and transform it into an exchangeable form. More researches should be done on Jew's mallow plants and their ability to absorb and accumulate Ni.

Table 4. Effect of sludge treated with cement by-pass (CB), iron oxide (FO) and silica gel (Si) on the Ni content (me/kg) of onion plants (first season) and Jew's mallow plants (second season)

Treatment	Ni content (mg/kg)		
	Onion plants (Direct effect)		Jew's mallow plants (Residual effect)
	Shoots	Bullb	
Control	11.98	22.90	167.90
Un- Sludge	26.25	17.87	120.07
CB 10%	18.95	14.67	291.68
CB 20%	25.03	19.50	111.58
FO 10%	7.30	14.75	86.75
FO 20%	9.65	8.90	73.74
Si 10%	14.95	18.95	73.20
Si 20%	26.68	17.93	89.55
FO + CB	25.98	12.90	115.40
Si + CB	19.05	16.20	92.64
Si + FO	3.40	5.95	47.38
LSD $_{0.05}$	1.12	1.03	1.71

4- Direct and residual effects of treated sludge materials on EDTA –extractable Ni of the soil:

Table (5) showsthe amounts of EDTA- extractable Ni of the soil after harvesting both of onion (direct effect) and Jew's mallow plants (residual effect). In the first season (direct effect), the soil extractable Ni increased with applying all sludge treatments (including theuntreated sludge) except with the sludge treated with mixtures of two stabilizing materials which displayed a very high stabilizing effect on the extractable Ni. The application of untreated sludge significantly increased the extractable Ni of the soil from 0.30 to 2.79 mg/kg due to the high content of Ni in the sludge (58.7 mg/kg). Amending the soil with sludge treated with FO + CB, Si+ CB and Si+ FO significantly stabilized all amounts of the extractable Ni in the soil and sludge in the first season resulting in 100% reduction of the extractable Ni. Gomah, (2015) indicated that treating sewage sludge with silica gel mixed- with ferric oxide or zeolite, each of them at 15%, reduced 100% of the extractable Ni. Sen *et al.* (2002**)** used preliminary kinetic experiments to explain the adsorption of Ni^{2+} and Cu^{2+} metal ions on iron oxide and reported that the adsorption a two- step process including: an adsorption of the metal ion on the external surface which rapid followed by slow intraparticle diffusion in the interior parts of the particles.

Although Jew's mallow plants were grown on the soil without any new sludge treatments (residual treatments), the EDTA extractable Ni of the soils lightly increased using 20% FO and 10%Si treated sludges and markedly increased using 20% Si, FO+CB, Si+CB and Si+FO treated ones. One or more reasons can be responsible for these increases including: 1- the root exudates excreted from the Jew's mallow plants may solubilize some of the total Ni content of the sludge (Gomah and Ezzeldin, 2003), 2- the decomposition of sludge may release more Ni to the soil and/or 3- the formation of soluble organic Ni complexes that may become more extractable by EDTA. Silica gel has a high surface area (Jiang *et al*., 2007) withporosity and rigid structure (Fenglian and Wang, 2011; Repo *et al*., 2011). On the other hand, the extractable Ni of the soil markedly and significantly decreased from 0.30, 2.79, 1.15, 3.47 and 2.52 me/kg in the first season to 0.00, 0.00, 0.00, 0.52 and 1.27 me/kg in the second season for the control, untreated sludge, 10% CB, 20% CB and 10% FO sludge treatments, respectively. These results may be attributed to increasing the Ni fixation of these treatments with time resulting in decreases the extractable Ni or/ and the high Ni absorption by Jew's mallow plants.

Table 5. Effect of sludge treated with cement by-pass (CB), iron oxide (FO) and silica gel on EDTA – extractable Ni (me/kg) of the soil after the first and-second seasons.

Treatment	Ni content (me/kg)	
	First Season (Direct effect)	Second Season (Residual effect)
Control	0.30	0.00
Un- Sludge	2.79	0.00
CB 10%	1.15	0.00
CB 20%	3.47	0.52
FO 10%	2.52	1.27
FO 20%	1.63	1.81
Si 10%	1.32	1.56
Si 20%	0.42	2.05
FO + CB	0.00	1.88
Si + CB	0.00	1.69
Si + FO	0.00	1.59
$LSD_{0.05}$	0.10	0.07

Very high Ni contents were recorded for the plants grown on these treatments as it is shown in Table (4). Treated sludge materials had different effects on the extractable Ni depending upon the type and the level of the stabilizing material, the application time and plant species. These results may need more analysis about the Ni forms and their contents of the soil and treatedsludge.

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Assiut J. Agric. Sci., (49) No. (3) 2018 (75-85) *ISSN: 1110-0486*
Website: *www.aun.edu.eg/faculty_agriculture/journals_issues_form.php* ***E-mail: ajas@aun.edu.eg***

أستخدام الحماة المعاملة ببعض مثبتات النيكل فى خفض تيسره فى التربة وامتصاصه بالنباتات

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الملخص

فى هذا البحث تمت معاملة حماة مياه الصرف الصحى ببعض مثبتات النيكل والتى شملت السليكا جيل ، مخلف صناعة الاسمنت وأكسيد الحديد وذلك لتقليل تيسره فى التربة وأمتصاصه بواسطة نباتات البصل (التاثير المباشر) فى الموسم الاول ولنباتات الملوخية (التأثير المتبقى) فى الموسم الثانى. تم أضافة معاملات حماة مياه الصرف الصحى والتى شملت ١٠و٢٠% سليكا جيل ، مخلف صناعة الاسمنت وأكسيد الحديد بالاضافة الى سليكا جيل + مخلف صناعة الاسمنت ، سليكا جيل + أكسيد الحديد ومخلف صناعة الاسمنت + أكسيد الحديد الى التربة الرملية الطميية قبل الموسم الاول فقط. أظهرت الحماة التى تم معاملتها بواسطة ٢٠% سليكا جيل أنها افضل معاملة فى تقليل تاثير النيكل فى التربة وعلى النباتات ، حيث أنها أدت الى زيادة الطول والوزن الطازج للمجموع الخضرى والبصلة وكذلك الوزن الجاف للمجموع الخضرى والبصلة لنباتات البصل بمقدار ١٧,٢، ١٤٨,٥، ٦,٩٩، ١٢٧,٨، ١٢٩,٢% على التوالى مقارنة بالكنترول.

أدى اضافة الحماة غير المعاملة الى زيادة تركيز النيكل الميسرفى التربة من ٠,٣٠ملجم/كجم فى الكنترول الى ٢,٧٩مجم/كجم بعد حصاد نباتات البصل. كما أن أضافة الحماة المعاملة بواسطة أكسيد الحديد + مخلف صناعة الاسمنت ، السليكا جيل + مخلف صناعة الاسمنت والسليكا جيل + أكسيد الحديد الى التربة قد أدى الى خفض معنوى لتركيز النيكل المستخلص بنسبة ١٠٠% مقارنة بالحماة غير المعاملة بعد حصاد البصل. أنخفض النيكل الميسر فى التربة بعد حصاد نباتات الملوخية من ٠,٣٠، ٢,٧٩، ١,١٥، ٣,٤٧ و٢,٥٢مجم/كجم إلى ٠، ٠، ٠,٥٢، ١,٢٧مجم/كجم وذلك لمعاملات الكنترول، الحماة غير المعاملة ، والمعاملة بواسطة ١٠% مخلف صناعة الاسمنت، ٢٠% مخلف صناعة الاسمنت و١٠% أكسيد الحديد على التوالى. كما أظهرت الحماة المعاملة بواسطة السليكا جيل + أكسيد الحديد فى الموسم الاول (التأثير المتبقى) أنها افضل المعاملات التى خفضت محتوى النيكل فى المجموع الخضرى والبصلة بنسبة ٧١,٦و٧٥,٦% مقارنة بالكنترول على التوالى. وكما أن الحماة المعاملة بواسطة ٢٠% سليكا جيل فى الموسم الثانى (التأثير المتبقى) هى أفضل المعاملات التى قللت من محتوى النيكل فى نباتات الملوخية بنسبة ٧١,٨% مقارنة بالكنترول.