CHEMICAL REMEDIATION OF CONTAMINATED SOILS WITH HEAVY MEATALS

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ABSTRACT:

This work represents a trial towards ameliorating soil polluted with some heavy metals (Ni, Co and Cd) resulted from some anthropogenic activities, i.e., human domestic and industrial wastes. To archive this target, an incubation pot experimental was conducted on two contaminated soil samples collected from El-Gabal El-Asfar farm (Qalubia Governorate) and El-Saff (Giza Governorate), which irrigated with sewage effluent and industrial wastewater, respectively. Before executing this experiment, the studied two soils were thoroughly mixed with two soil amendments, i.e., rock phosphate and calcium carbonate at a rate of 2 % for each one, then daily wetted with distilled water to a constant moisture content (field capacity). Soil samples were taken at incubation periods of 0, 25, 50 and 75 days to determine EDTA extractable Ni, Co and Cd for evaluating the effective role of the tested soil amendments in immobilizing the dominant heavy metal ions. The obtained results could be summarized in the following:

1. Increasing time of incubation period between the studied two soils and rock phosphate caused progressively decreased in the values of EDTA-extractable Ni, Co and Cd. The sorbet amounts of Ni, Co and Cd increased with prolonging time of contact between rock phosphate and soil, with the removal percentages followed the descending order: Cd > Ni > Co. The later sequence was changed at the incubation period of 75 days in El-Gabal El-Asfar soil, where the sequence of removal percentages became: Cd > Co ≥ Ni. Also, El-Gabal El-Asfar soil could be adsorbed higher amounts of Ni, Co and Cd than El-Saff soil, while the reverse was true for the sorption percentages of these metal ions in El-Saff soil, except for Cd at the incubation period of 75 days.
2. Increment of incubation time between soils and $CaCO_3$ led to markedly decreased in the amounts of EDTA-extractable Ni, Co and Cd, which seemed a highest capacity with Ni, lowest with Co and intermediate with Cd. Consequently, it could be categorized these metal ions according to their removal amounts into the following the descending order: Ni > Cd > Co regardless of type of soil. The later sequence also represented the removal capacity of $CaCO_3$ for the studied metal ions.

Key words: Chemical remediation, heavy metals, rock phosphate and $CaCO_3$ as soil amendments.

INTRODUCTION:

Contamination of soil and water by various pollutants such as pesticides, poly aromatic hydrocarbons (PAH), heavy metals, etc., have caused imbalance in the natural function of the ecosystem. Main sources of heavy metal contamination are: 1) Urban industrial aerosols and other industrial processes,

2) Liquid and solid wastes generated from animals and humans, 3) Mining activities and 4) Industrial and agricultural chemicals.

Soils can naturally reduce mobility and bioavailability of heavy metals as they are retained in soil by sorption, precipitation and complexation reactions. This natural attenuation process can be accelerated by addition of amendments to convert the soluble form to more geochemical stable solid phases.

Solidification was used to remedy the metal contaminated soils. Among them, in situ fixation of heavy metal using exterior amendments is a promising technology for cleaning up contaminated soils and wastes **(Zhou and Song, 2004)**. In situ application of soil additives modifying the physicochemical properties of the contaminating heavy metals combined with the development of biological communities and plants (**Diels *et al.*, 2002**). Fraction-based studies on the fixation of heavy metals in sediments and soils had been greatly carried out with different unitary additives, such as zeolite, cements, and limes, and used in the metal immobilization (**Cao *et al.*, 2003** and **Ciccu *et al.*, 2003**). Both in water and soil, the fixation treatment is based on the reduction of metal mobility and availability. The additives could be classified into organic, inorganic and organic-inorganic types according to the different availability in nature.

Chemical stabilization was evaluated as one of the most cost–effective remediation techniques for heavy metal contaminated sites (**Chen *et al.*, 2000).** Sorption, ion exchange, and precipitation are principal mechanisms to convert soluble and preexisting potentially soluble solid phase forms of heavy metals to more geochemical stable solid phases, such as lime material, phosphate, Fe and Mn oxides (**Phillips, 1998** and **Chen *et al.*, 2000)**. The effects of recommended amendments for soil remediation are often attributed to increase in cation exchangeable capacity (CEC), the addition of materials with a strong preference for particular ions, and precipitation as a solid phase **(Czupyrna *et al.*, 1989*)***. **Chen *et al.* *(*2000)** used calcium carbonate, zeolite, and manganese oxide to efficiently reduce the availability of Cd and Pb in two contaminated soils, and significantly reduce the uptake of Cd and Pb by wheat shoots.

The current study deals with the effect of two different soil amendments, i.e., rock phosphate and $CaCO_3$ on immobilization of some heavy metal ions, i.e. Ni, Co and Cd in soils contaminated as a result of some anthropogenic activities.

MATERILAS AND METHODS:

An incubation pot experiment was conducted on two contaminated soil samples with some heavy metals (Ni, Co and Cd) were collected from El-Gabal El-Asfar farm (Qalubia Governorate) and Arab Abu–Said village, El-Saff district (Giza Governorate), which are irrigated with drainage water polluted with sewage effluent and industrial wastes, respectively. Two soil amendments, i.e., rock phosphate and calcium carbonate were used for immobilizing the studied heavy metals in both soils under investigation. The main characteristics of the investigated soils are presented in Table (1).

Table (1): The main physical and chemical properties of the studied soils and soil amendments.

Soil characteristics	El-Gabal El-Asfar soil	El-Saff soil
Particle size distribution %:		
Coarse sand	49.00	60.39
Fine sand	19.20	27.50
Silt	23.20	8.17
Clay	8.60	3.94
Textural class	Sandy loam	Sandy
EC (dS/m)	4.10	6.70
Soluble cations (mmol$_c$ L^{-1}):		
Ca^{2+}	17.20	35.80
Mg^{2+}	16.20	15.00
Na^{+}	9.50	18.50
K^{+}	0.98	2.50
Soluble anions (mmol$_c$ L^{-1}):		
CO_3^{2-}		
HCO_3-	6.90	3.00
Cl^-	20.20	35.00
SO_4^{2-}	16.78	33.80
Available heavy metals (mg kg^{-1}):		
Cd	0.334	0.306
Co	0.182	0.126
Ni	4.116	1.305
pH (1:2.5 soil suspension)	6.05	7.80
$CaCO_3$ %	0.39	12.45
CEC (cmol$_c$ kg^{-1})	12.65	5.72
Organic matter %	3.90	0.94

The incubation pot experimental:

The investigated soils were mixed thoroughly with rock phosphate and calcium carbonate at a rate of 2 %, and packed in plastic pots of 200 g soil capacity, the were categorized into two sets of pots prepared for an incubation experiment. The pots were daily wetted with distilled water to reach constant soil moisture content (field capacity). After being incubated at room temperature for different periods of.0, 25, 50 and 75 days. Each treatment was repeated three times, and soil samples were taken from the different treatments and incubation periods for determining the amounts of EDTA-extractable Ni, Co and Cd by using an Atomic Absorption Spectrophotometer (Perkin Elmer, Model 2380).

Laboratory analysis:

* Particle size distribution of the studied soils and bentonite was determined by the pipette method **(Piper, 1950)**.

* Organic matter content was determined according to the modified Walkely and Black's method **(Jackson (1973)**.

* Calcium carbonate was determined by using Collin's Calcimeter **(Wright, 1939)**.

* Soil pH was determined in 1:2.5 soil or amendment water suspension as described by **Page *et al.* (1982).**

* ECe and soluble anions ions were determined in soil paste extract according to **Jackson (1973)**.

* Cation exchange capacity of soil (CEC) was determined according to the method outlined by **Jackson (1973)**.

* Available contents of Ni, Co and Cd were extracted with EDTA solution **(Soltanpour and Schwab, 1977)**, and determined by using an Atomic Absorption Spectrophotometer (Perkin Elmer, Model 2380).

RESULTS AND DISCUSSION:

Effect of applied soil amendments on the amounts of EDTA-extractable metal ions:

1. Rock phosphate:

Data presented in Table (2) reveal that incubating the amended soils with rock phosphate at a rate of 2 % (dry weight basis) resulted in lowering the values of EDTA-extractable Ni, Co and Cd. Also, it is obvious that increasing time of incubation period between soil and rock phosphate seemed a gradually increase in the values of EDTA-extractable Ni, Co and Cd. This means that the rock phosphate treated soils exhibited an increase in ability to adsorb Ni, Co and Cd with prolonging time of incubation period between rock phosphate and the investigated soils. Accordingly, the removal capacity of the rock phosphate (which is measured by mg of metal removed per g of rock phosphate) tended to increase with increasing incubation time, i.e., reached its highest values at the longest period of incubation (75 days).

Table (2): Effect of applied rock phosphate on EDTA-extractable metal ions and their sorption parameters for both the studied two soils.

Sorption parameter	**Incubation period**	**El-Gabal El-Asfar soil**			**El-Saff soil**		
		Ni	**Co**	**Ni**	**Co**	**Ni**	**Co**
Residual amount (mg kg^{-1})	0	4.046	0.078	0.338	1.290	0.123	0.309
	25	3.479	0.069	0.277	1.070	0.104	0.245
	50	3.196	0.063	0.258	0.955	0.094	0.220
	75	2.873	0.058	0.099	0.877	0.086	0.195
Sorbet amount (mg kg^{-1})	25	0.567	0.009	0.061	0.220	0.019	0.064
	50	0.850	0.015	0.080	0.335	0.029	0.089
	75	1.173	0.020	0.239	0.413	0.037	0.114
Sorbet amount (%)	25	14.01	11.53	18.04	17.05	15.44	20.71
	50	21.00	19.23	23.66	25.96	23.57	28.80
	75	28.99	25.64	70.71	32.01	30.08	36.89
Removal capacity (mg metal g^{-1})	25	0.028	0.000	0.003	0.011	0.001	0.003
	50	0.043	0.001	0.004	0.017	0.001	0.004
	75	0.059	0.001	0.012	0.021	0.002	0.006

At all the periods of incubation, the adsorbed amounts of the tested metal ions by the rock phosphate treated soils followed the descending order: Ni > Cd > Co. This sequence seems to be coinciding with their EDTA-extractable amounts. It is also noticed that the sequence of the studied heavy metals differed with time of incubation period between the soil and rock phosphate according to their sorption (removal) percentage. However, sorption (removal) percentages of Ni, Co and Cd reached the lowest, intermediate and highest levels on the 25, 50 and 75th days of incubation, respectively. Hence, the metal ions could be arranged according to their sorption (removal) percentage in the following descending order: Cd > Ni > Co. This sequence occurred in both the studied soils and for all the studied periods of incubation, except for the 75th days of incubation in El-Gabal El-Asfar soil with the rock phosphate, where the sequence differed slightly due to the values of sorption (removal) percentage of Cd highly exceeded those of Co and Ni. **Christopher *et al.* (2002)** reported that heavy metal ions might exchange with the released Ca^{2+} from rock phosphate and larger multivalent ions are more effectively removed. Since the investigated metal ions are arranged according to their radii in the following descending order: Cd (0.97 A^0) > Ni (0.69 A^0) > Co (0.65 A^0), and consequently the removal percentage of these ions should obey the sequence Cd > Ni > Co, which was the case in the current study.

Moreover, the removal capacity values (i.e. the amount of metal removed per unit weight of the applied adsorbent of rock phosphate) increased with time of incubation period. This observation was true regardless of kind of either the studied soil or metal ion, yet these values were the highest in case of Ni, lowest in case of Co and intermediate in case of Cd. The comparison between the values of sorption percentages of Ni, Co and Cd for both the two investigated soils reveal that El-Gabal El-Asfar amended soil could adsorb higher amounts of Ni, Co and Cd than El-Saff soil at all the incubation periods. However, sorption percentages of these metal ions were relatively higher in El-Saff soil than El-Gabal El-Asfar one, except for Cd at 75 days of incubation where its sorption percentage in El-Gabal El-Asfar soil was higher than the corresponding one of El-Saff soil.

As a matter of fact, the initially available EDTA-extractable amounts of Ni, Co and Cd were higher in El-Gabal El-Asfar soil than in El-Saff soils. The higher $CaCO_3$ content of the later soil than the former one may account for such an observation. A similar finding was reported by **Lothenbach *et al.* (1997)** who found that metal ions solubility in calcareous soils is small. Also, **Robinson *et al.* (2000)** found that $CaCO_3$ significantly decreased the solubility of Ni in soil. In this connection, **Ismail (2007)** pointed out that Ni and Co were sorbet on $CaCO_3$. Moreover, **Balba (1995)** indicated that heavy metals may react with $CaCO_3$ and precipitate in the form of insoluble heavy metal carbonates.

The higher available contents of Ni, Co and Cd in El-Gabal El-Asfar soil were associated with higher sorption amounts, the reverse was true for the sorption percentages as cleared by data in Table (2). These results are in accordance with those of **Ismail (2007)** who found that the desorption values of Ni, Co and Cd as percentages of their initial concentrations, generally, decreased with increasing their initial concentrations. The effect of rock phosphate on the amounts of Ni, Co and Cd removed from El-Gabal El-Asfar soil was more pronounced, while the sorption (removal) percentages of Ni, Co and Cd were higher in El-Saff soil, probably due to its calcareous in nature.

Saxena and D' Souza (2005) reported that the exact reaction mechanism responsible for removal of metal ions by rock phosphate is unknown, yet they cleared that the sorption process, which, generally, involves species attachment from a solution to the existing solid surface by three types, namely surface adsorption, absorption or diffusion into the solid and precipitation or co-precipitation appears to be governing mechanism for retention of metal ion by rock phosphate.

It is supposed that the primary mechanism of metal ion removal by rock phosphate is governed by its dissolution followed by subsequent precipitations. Hence, the process may be represented by the following equations:

$$Ca_{10}(PO_4)_6F_2 + 12\ H^+ \xrightarrow{\text{dissolution}} 10\ Ca^{2+} + 6\ H_2PO_4^- + 2\ F^-$$

$$10\ M^{2+} + 6\ H_2PO_4^- + 2\ F^- \xrightarrow{\text{precipitation}} M_{10}(PO_4)_6F_2 + 12\ H^+$$

In addition to the above hypothesis, where P in the form of $H_2PO_4^-$ helps in precipitation metal ions, the possibility of exchange of Ca by metal ions can not be ruled out **(Perrone *et al.*, 2001)**. It may be interpreted that the dissolution of rock phosphate also provides Ca, which exchanges with aqueous metal leading to the precipitation of corresponding mineral phase. The same authors reported that there is isomorphic substitution of Ca^{2+} with heavy divalent ions, which is correlated to their ion radii and electro negativity.

2. Calcium carbonate:

Data in Table (3) show the effect of the applied $CaCO_3$ on immobilization of the studied metal ions (Ni, Co and Cd) among the different time of incubation periods for both the two investigated soils. The obtained data reveal that the extractability of Ni, Co and Cd by EDTA were emphasized by the sorption parameters of these metal ions. Also, it is obvious that increasing time of incubation period between $CaCO_3$ and each of the El-Gabal El-Asfar and El-Saff soils reduced progressively amounts of EDTA–xtractable Ni, Co and Cd. The immobilized amounts of these metal ions seemed highest with Ni, lowest with Co and intermediate with Cd. The positive effect of $CaCO_3$ on reducing the availability of heavy metals was supported by many investigators, i.e., **Moore and Loeppert (1990), Nikolov and Zlatareva (2000), Robinson *et al.* (2000) Tadroos (2004) and Ismail (2007)** who found that increasing the values of both $CaCO_3$ and pH significantly decreased the extractability of many trace elements in soils among which Ni, Co and Cd. These result are in agreement with those reported by **Balba (1995)** who pointed out that the decrease in trace metals availability upon application of $CaCO_3$ to soil is mainly related to the increase in soil pH resulting from hydrolysis of $CaCO_3$ which yields OH^- ions as follows:

$$CaCO_3 + H_2O \leftrightarrow Ca^{2+} + HCO_3^- + OH^-$$

Kabata-Pendias and Pendias (1992) reported that solubility of most metal ions will decrease with increasing soil pH. This observation can be explained by the precipitation of metal hydroxides.

Table (3): Effect of applied calcium carbonate on EDTA-extractable metal ions and their sorption parameters for both the studied two soils.

Sorption parameter	Incubation period	El-Gabal El-Asfar soil			El-Saff soil		
		Ni	Co	Ni	Co	Ni	Co
Residual amount (mg kg^{-1})	0	4.040	0.078	0.332	1.284	0.123	0.300
	25	3.909	0.058	0.234	0.860	0.087	0.189
	50	2.464	0.050	0.199	0.744	0.074	0.156
	75	2.101	0.046	0.163	0.578	0.068	0.115
Sorbet amount (mg kg^{-1})	25	0.131	0.020	0.099	0.424	0.036	0.111
	50	1.576	0.028	0.134	0.540	0.049	0.144
	75	1.939	0.032	0.170	0.706	0.055	0.185
Sorbet amount (%)	25	32.43	25.64	29.73	33.02	29.26	37.00
	50	39.01	35.89	40.24	42.05	39.83	48.00
	75	47.99	41.02	51.05	54.98	44.71	61.66
Removal capacity (mg metal g^{-1})	25	0.007	0.001	0.005	0.021	0.002	0.006
	50	0.079	0.001	0.007	0.027	0.002	0.007
	75	0.097	0.002	0.009	0.035	0.003	0.009

Sorbed amounts (mg kg^{-1}) of the concerned metal ions increased with increasing time of incubation period between soils and $CaCO_3$ over all the incubation periods, and the sorbed amounts of the studied metal ions followed the descending order: Ni > Cd > Co regardless of soil type. This sequence agrees, to a great extent, with the concentrations of the studied metal ions in both the investigated soils at zero time of incubation whose they followed the same aforementioned sequence (i.e. Ni > Cd > Co). This finding stands in well agreement with the results of **Ismail (2007)** who found that adsorbed amounts of Ni and Co on $CaCO_3$ increased progressively with increasing concentrations of the concerned metal ions in the equilibrium solution.

It is worthy to indicate that after 25 days of incubation, sorbed amounts of Ni, Co and Cd in El-Gabal El-Asfar soil were lower than the corresponding amounts sorbed in El-Saff soil although the initial concentrations of these metal ions in the former soil were obviously higher than the corresponding ones of the latter. After 50 and 75 days of incubation, sorbed amounts of both Co and Cd in $CaCO_3$ amended soil of El-Saff exceeded the corresponding amounts of metals sorbet on $CaCO_3$ amended soil of El-Gabal El-Asfar. In both these periods, sorbet Ni followed an opposite trend where it was higher in El-Gabal El-Asfar soil as compared to those of El-Saff soil. The higher original content of $CaCO_3$ in El- Saff soil might participate with the $CaCO_3$ applied as an amendment in sorption of the concerned metal ions. Therefore, the values of sorption (removal) percentages of the metal ions in El-Saff soil, generally, exceeded the corresponding sorption percentages in El-Gabal El-Asfar soil. However, in both soils the values of sorption percentages of the metal ions tended to increase by prolonging incubation period.

Values of the removal capacity increased also with time of incubation period and seemed to be in the descending order: Ni > Cd > Co. **Guo *et al.* (2006)** reported that it is a common practice to apply lime to reduce the extractable metal concentration and successfully immobilize heavy metals in soils. They added that $CaCO_3$ may reduce trace elements solubility in two ways; 1) it raises the soil pH and 2) it adsorbs trace element ions.

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المعالجة الكميائية لأراضى ملوثة بالعناصر الثقيلة

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تعتبر هذه الدراسة محاولة لمعالجة كيميائية للأراضي الملوثة ببعض العناصر الثقيلة (Ni, Co and Cd) نتيجة بعض الأنشطة البشرية والمخلفات الصناعية، ولتحقيق هذا الهدف أجريت تجربة تحضين أستخدمت فيها عينتى تربة من منطقتي الجبل الأصفر (محافظة القليوبية) والصف (محافظة الجيزة)، وهما ملوثتان بالعناصر الثقيلة نتيجة ريهما بمياه ملوثة بمخلفات الصرف الصحي والصرف الصناعى على الترتيب، حيث تم تحضينهما بعد خلطهما جيدا بالصخر الفوسفاتي أو كربونات الكالسيوم بنسبة ٢٪، وذلك في أصص سعة ٢٠٠ جرام وقد تم ترطيب تربة الأصص بصفة يومية بالماء المقطر للوصول لمحتوى رطوبى ثابت (السعة الحقلية) طوال فترة التحضين، كما أجرى التحضين على درجة الغرفة (٢٨ ± ٢ ° سليزيوس) لفترات: صفر ، ٢٥ ، ٥٠ ، ٧٥ يوماً، ثم أخذت عينات ممثلة للمعاملات على إمتداد فترات التحضين المختلفة لتحديد الجزء المستخلص من عناصر النيكل والكوبالت والكادميوم باستخدام محلول EDTA، لتقييم كفاءة محسنات التربة المضافة فى تقييد وعدم تيسر العناصر الثقيلة موضوع الدراسة، ويمكن تلخيص أهم النتائج التى تم الحصول عليها فيما يلى:-

١. زيادة زمن فترة التحضين بين صخر الفوسفات وكلا التربتين موضع الدراسة قد أدى إلى نقص كبير فى قيم عناصر النيكل والكوبالت والكادميوم المستخلصة بمحلول الـ EDTA ، وذلك بنسب مئوية لإزالة هذه العناصر أمكن ترتيبها تبعا للكمية المزالة تنازليا فيما يلى: $Cd > Ni > Co$. وقد تغير هذا التتابع بالنسبة لفترة تحضين ٧٥ يوما فى تربة الجيل الأصفر المعاملة بصخر الفوسفات حيث أصبح التتابع التنازلى كما يلى: $Cd > Co \geq Ni$. وقد تميزت تربة الجبل الأصفر المعاملة بصخر الفوسفات بمقدرتها على إدمصاص كميات أكبر من النيكل والكوبالت والكادميوم مقارنة بتربة الصف، والعكس صحيحا بالنسبة للنسب المئوية للإدمصاص فيما عدا عنصر الكادميوم عند فترة تحضين ٧٥ يوما.

٢. أدى زيادة فترة التحضين بين كلا التربتين موضع الدراسة وكربونات الكالسيوم المضافة إلى نقص واضح في قيم عناصر النيكل والكوبالت والكادميوم المستخلصة بمحلول الـEDTA ، حيث أظهرت كربونات الكالسيوم المضافة سعة أعلى ما يمكن مع النيكل وأقل ما يمكن مع الكوبالت وما بينهما مع الكادميوم. وتبعا للكميات المزالة من هذه العناصر أمكن ترتيبها تنازليا فيما يلى: $Ni > Cd > Co$ بغض النظر عن نوع التربة، وهذا الترتيب بعبر أيضا عن سعة الإزالة لمكون كربونات الكالسيوم بالنسبة للعناصر الثقيلة موضع الدراسة.