

## REMEDIATION OF SOIL POLLUTED WITH INDUSTRIAL WASTES USING AN IMMOBILIZATION TECHNIQUE.

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### ABSTRACT

The current study was designed to test the potential of diammonium phosphate (DAP) in immobilizing Pb and Zn in an industrial contaminated soil. Four phosphate treatments including various ratios of added P to total heavy metals content corresponding to stoichiometric P to Pb ratio of chloropyromorphite mineral were tested. The obtained results revealed that the total contents of Pb and Zn in the topsoil were above the maximum acceptable concentrations (MAC) that reported by USEPA- 503 regulations.

In the tested soils, the data referred that the sequential extraction procedure employed in this study appeared to be useful for classifying Pb and Zn within six geochemical fractions. The fractionation studies indicated that Fe & Mn oxide fraction proved to be the most abundant for Pb (more than 41% of total Pb), whereas the residual form was the dominant for Zn (37%). The sum of the relative soluble pools implies that a great portion of these metals may be highly bioavailable. Amending the soil with DAP converted the relative soluble pools into more stable pools. Zn was distributed in the order; residual >>oxide>carbonate>organic>exchangeable > water-soluble. While the order for Pb was residual = oxide >organic >carbonate > exchangeable >water-soluble.

On an average basis, the mobility indexes of Pb and Zn decreased from 20.1 to 11.10 and from 28.2 to 15.44 in the untreated and phosphate-treated soil, respectively. The concentrations of the active portion of Pb and Zn, expressed as minus logarithm of  $ML^{-1}$  (pC) sharply decreased as a result of soluble phosphorous amending. The pC of  $Pb^{2+}$  decreased from 10.23 to 11.23 and from 7.59 to 7.75 for  $Zn^{2+}$ , suggesting that DAP has potential to immobilize Pb and Zn in industrial contaminated soil.

The results of solubility diagrams of lead-phosphate minerals suggested that chloropyromorphite mineral in equilibrium with either DAP or DCPD has been recognized as a mineral controlling Pb solubility untreated (contaminated) and treated soil solutions, respectively. Also, the results of solubility diagrams of Zn suggested that Zn-soil regulate the solubility of Zn in all the tested soil solutions.

### INTRODUCTION

The rapid development of human civilization and use of chemical substances in many human activities caused a steady increase of heavy metal pollution in soils. At present, a variety of approaches have been suggested for remediation of contaminated soils, vary in their effectiveness or cost. These technologies are classified into main four technologies; 1) excavation and land filling technologies; these technologies are highly effective and lowering risk to the environment. Otherwise, it is labor intensive and expensive; 2) Washing-based technologies involve increasing solubility's of heavy metals through special extraction, consequently, the flow of mobilized metals increased (Feick *et al.*, 1972; Ram and Verloo, 1985; Elliot

and Linn ,1987) . These technologies may be more effective in coarse – textured soils and where the drainage system can be adequately controlled; 3) Electro-chemical technologies which considered soil/ground water /sediment as electrochemical system and 4) In situ- chemical immobilization technologies which involve addition of chemicals to contaminated soil to reduce metal solubility through formation of stable metal minerals ( Madrid and Berrientos,1992 ; Ursula and Rufus ,2000). These technologies often resulting in a substantial cost saving and provide a long –term remediation solution .

Chemical immobilization studies using phosphate additions included mineral apatite and synthetic hydroxapatite materials( Chen *et al.*, 1997 ; Ma and Roa ,1997). These materials decrease metal solubility through the formation of metal – phosphate precipitates and increase long –term stability by forming less soluble and more stable metal – phosphate minerals ( Ma *et al.*, 1993). Furthermore, soluble phosphate sources could provide an abundance of soluble phosphorous and increase the efficiency of metal – phosphate formation (Berti and Cunningham ,1997 ; Cooper *et al.* ,1998 ).

This study has been performed to explore the potential of using diammonium phosphate (DAP) to control contaminant metal migration , redistribution of metals among various pools, mobility index and metal activities.

## MATERIALS AND METHODS

### Sampling site:

A soil profile having high concentrations of some heavy metals was selected alongside the main Cairo – Alexandria. Highway at km 26. This area is characterized by high traffic density and belongs to many industrial complexes.

Four soil samples were collected from the site at 30-cm increments down to a depth of approximately 120-cm. The soil samples were air-dried and grounded to pass a 2-mm sieve. Selected soil properties were carried out according to conventional methods outlined by Black (1965), Table 1.

### Soluble Phosphorus Treatments:

Reagent diammonium phosphate (DAP) was added as an amendment to the contaminated soil based on the total molarity of the heavy metals content in the selected soil . Four phosphate ratios to total heavy metals were selected as follows ; A, ( 0.0) ; B, ( 3: 50) ; C, ( 3:25) ; and D, (3:5) . The 3 : 5 ratio was selected as a base of comparison , since it corresponds to the stoichiometric P to Pb ratio of chloropyromorphite [  $Pb_5(PO_4)_3Cl$  ] .

Phosphate amendments were mixed thoroughly with soil samples and allowed to equilibrate prior to further analysis. The treated soils were watered until field capacity and left to dry for 2 days , they alternately moistened and dried again ; one wetting and one drying constituted a cycle ;



the treated soils were subjected to 4 wetting and drying cycles. Thereafter , the following analysis were carried:

**Partitioning of heavy metals :** The procedure which described by Salbu *et al.*, (1998) was used to fractionate Pb and Zn in the contaminated soil before and after additions of phosphate amendments to explore transformation of the heavy metals among different pools .

**Six operationally defined fractions were extracted as follows ;**

- Water soluble (F1) ;
- Exchangeable fraction ( F2 ),extracted with 1M NH<sub>4</sub>OAc, pH 7 ;
- Carbonate bounded fraction ( F3 ), extracted with (1M NH<sub>4</sub> OAc , pH 5 ) ;
- Fe&Mn oxides fraction (F4),extracted with (0.04 M NH<sub>2</sub>OH .HCl) ;
- Organic fraction (F5),extracted with ( H<sub>2</sub>O<sub>2</sub> pH ,2) and R
- Residual fraction (F6), (7M HNO<sub>3</sub> ).

The total contents of heavy metals were determined by digestion with 7 M HNO<sub>3</sub> on a hot plate for 6 h.

**Measurement of heavy metals activities:** The activities of Zn and Pb in the untreated soil sample (A), and phosphate – treated soil samples ,(B, C, and D) were subjected to speciation procedure as outlined by Hirsch and Banin (1990) . The analytical concentrations of Cd ,Co, Cu, Fe, Mn, Ni ,P , Pb ,Zn and Si were measured by using Inductively Coupled Plasma Optical Emission Spectrophotometer. NO<sub>3</sub><sup>-</sup> ,Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> were determined according Black (1965).

The previous results were computed in GEOCHEM model program (Sposito *et al*,1980) to calculate the active portions (M<sup>2+</sup> ) for Pb and Zn . The input and output data expressed as pC (-log C) were listed in Table 3.

## RESULTS AND DISCUSSION

### A-General properties of the tested soil :

Some of general properties of the tested soil are listed in Table 1 . As shown from data the studied soil was a clay loam soil with more than 35% clay content . Soil pH in all the tested samples is around 7.4 . Total calcium carbonate content ranged from 2.1 - 4.08 % . Organic matter contents ranged from 2.43% in the top layer to 1.95% in the lower layer .

**Table(1): Some soil properties and total heavy metals contents of the soil profile.**

Depth Cm	pH	Clay %	O.M %	CaCO <sub>3</sub> %	Total heavy metals content mg/kg soil			
					Pb	Zn	Ni	Cd
0-30	7.44	36.42	2.43	4.08	112.7	66.91	7.16	4.28
30-60	7.41	34.60	2.15	3.24	10.24	60.20	2.56	4.15
60-90	7.41	35.26	1.98	3.14	8.16	46.50	2.11	3.80
90-120	7.38	34.84	1.95	2.10	9.14	39.81	2.61	2.14

### **B-Total heavy metals content :**

Table 1 shows that the total content of heavy metals, in the surface layer, each in mg/kg soil is; Pb(112.7), Zn(66.91), Ni(7.16) and Cd(4.28). It is obvious that the total content of Pb and Zn in the surface layer is well above the maximum acceptable concentrations (MAC) established for these metals by US Environmental Protection Agency (1993), Method 503 (USEPA-503). The enrichment of the top soil could be due to atmospheric fallout deposition of industrial wastes and other human's activities. The data also show that the concentration of heavy metals decreased with depth. The magnitude of decreasing, however, varied from one metal to another.

Enrichment factors of the tested metals were calculated using the following formula (metal content in a given depth / metal content in last depth \*100), assuming that the metal content in the last depth is the background concentration. The enrichment factors of the tested metals indicated that only Zn and Cd showed some evidence of downward movement. In the four tested soil depths, the enrichment factor for Zn was 168, 151, 117 and 100 for 0-30, 30-60, 60-90 and 90-120 cm layers, respectively. The corresponding values for Cd were 200, 194, 178, and 100, respectively. Otherwise the enrichment factors were 1233, 112, 90 and 100 (for Pb) and 274, 98, 80 and 100 (for Ni), respectively referring to a little downward movement for Pb and Cd metals in that soil. The same conclusions were also observed by Doner (1978); Elliot and Linn (1987); Madrid and Berrientos (1992) and Elsokkary and Lag (1978).

### **C-Partitioning of Pb and Zn metals :**

Partitioning of Pb and Zn in the untreated soil and phosphate-treated soil samples expressed as percentage of their sum are given in Table 2 and illustrated in Fig 1 and 2

**1- Untreated soil :** it is clearly shown from Table 2 that the major portion of Pb is bounded with Fe & Mn oxides (ca. 42% of total Pb), this result agrees with the results of Tessier *et al.*, (1979) and El-Gendi (1994). The residual fraction was the next most important fraction, containing approximately 22%, followed in decreasing order with organic fraction, which amounted to 16% of total Pb. The significant portion of organic-Pb is not surprising, since it is well known that Pb has high affinity with organic matter (Zimdahl and Skogerboe, 1977). Fig 1 also explained that the carbonate fraction was nearly of equal magnitude like organic fraction. Meanwhile, the exchangeable fraction of Pb comprised about 6% of total Pb. Negligible amount of Pb was present in water-soluble fraction.

The fractionation of Zn in the untreated soil was shown in Fig 2, which revealed that the residual fraction was the most abundant fraction for Zn (ca. 37% of total Zn). The same observations were also reported by Kuo *et al.*, (1983) and Ma and Rao (1997). They stated that Zn is commonly occluded by silicates during weathering process. Fe & Mn fraction was the second dominant ones. It amounted to 26.5% of total Zn. Also Elsokkary and Lag (1978) also reported the importance of Zn-oxide fraction. Low portion of Zn was associated with organic matter (ca. 8% of total Zn). This may be



to the low of stability of Zn – fluvic complex (Stevenson ,1967) . The sum of exchangeable ,carbonate and water –soluble fractions of Zn comprised about 20% of total Zn in the tested soil , indicating high bioavailable of Zn in this soil .

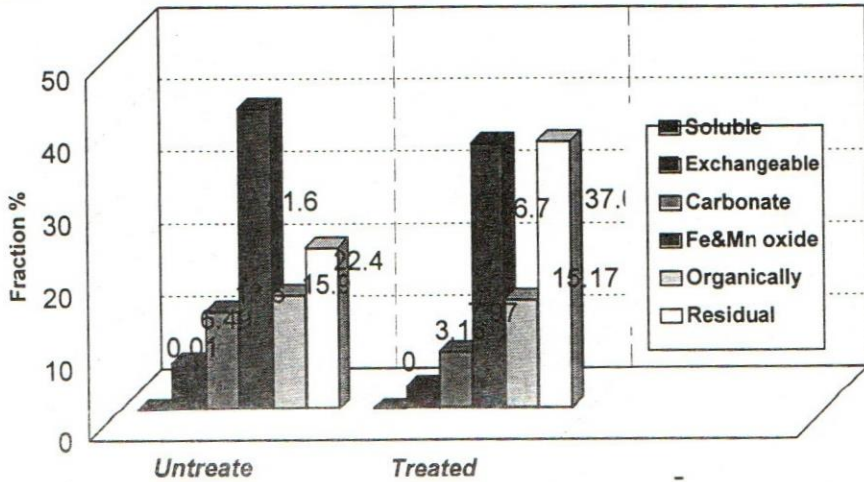


Fig.1: pb fractionation in the tested soils .

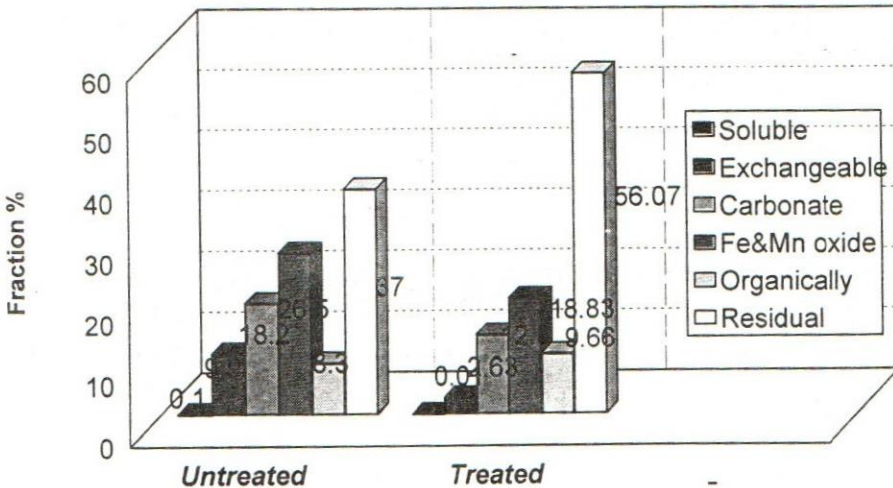


Fig.2: Zn fractionation in the tested soils .

**2- phosphate –treated soil**

Fig 1 and 2 illustrated the effect of soluble phosphorous amendments on re - distribution of the fractions tested metals . It is clear from the data presented in Table 2 that the residual and Fe & Mn bounded fractions are the most important fractions of Pb and Zn in phosphate- treated contaminated soil samples. The data show that the majority of Zn (ca. 56% of The total)

and appreciable portion of Pb (ca. 37%) are in residual form . Meanwhile, the Fe & Mn fraction amounted to 18% and 36% of total Zn and Pb , respectively .

From these results, it could be concluded that soluble phosphorous converts relative soluble pools of the tested metals (soluble + exchangeable + carbonate ) into more strongly bounded fractions(Fe & Mn oxide + organic + residual ). This means that phosphorous ,when present in sufficient amounts , reduces heavy metals solubilities. The same conclusions were also observed by Berti and Cunningham (1997) ; Cooper *et al.*,(1998) .Furthermore , in contaminated soil treated with phosphate rock , Ma and Rao (1997) suggested that lead precipitated as a fluoropyromorphite – like mineral and it was probably the solid phase of Pb that controlling its solubility in that soil

**D-Mobility factors of Pb and Zn in the tested soils:**

The effect of added phosphate on the mobility Factor (MF)of Pb and Zn calculated as proposed by Narwal *et al.*,(1999) is shown in Table 2.

**Table 2. Pb and Zn Fractionation and mobility indexes in the tested soils.**

Pb fractions %							
Treat.	Soluble	Exch..	Carb	Fe & Mn Oxide	Organic	Residual	MF**
A	0.01	6.49	13.60	41.60	15.90	22.40	20.10
B	00.00	4.50	10.49	40.51	14.30	30.20	14.99
C	00.00	3.20	8.22	34.38	15.20	39.00	11.42
D	00.00	1.70	5.20	35.22	16.00	41.88	6.90
Mean(x)	00.00	3.13	7.97	36.70	15.17	37.03	11.10
Zn fractions %							
A	0.10	9.90	18.20	26.50	8.30	37.00	28.20
B	0.02	3.19	12.60	20.00	9.40	54.79	15.81
C	0.01	2.70	14.80	18.50	9.50	54.49	17.51
D	0.01	2.00	11.00	18.00	10.09	58.90	13.01
Mean(x)	0.01	2.63	12.80	18.83	9.66	56.07	15.44

r  $x' = (B + C + D)/3$

r  $MF = (\text{water soluble} + \text{exchangeable} + \text{carbonate fractions}) / \text{Sum of the six fractions}$

In case of the untreated contaminated soil (A) , the mobility index is higher for Zn ,(28.2%) ,than that for Pb ,( 20.10%) . These findings also confirmed that although the relative soluble fractions presented in small portions; Nevertheless, they are the most bioavailable form of heavy metals .

It is clearly shown from the data that amending the contaminated soil with soluble phosphorous decreased mobility indexes of the tested metals and its effectiveness is much higher for Pb than that for Zn . On an average basis , the mobility index in Pb and Zn treatments is 11.1% and 15.44% , respectively. The mobility index was little affected by applied dose of soluble phosphorous , the values for Pb were 14.99%, 11.41% and 6.90% and for Zn were 15.81% ,17.51% , and 13.01% , at P : Metal ratio of 3:50 (B) ,3:25 (C) and 3:5 (D),respectively.



**E-The effect of phosphorous amending on heavy metals activity.**

The input and output data of GEOCHEM Modeling Program expressed as pC (-logC) are shown in Table 3, respectively. The obtained values of Zn<sup>2+</sup> activities .

The activity values of Zn<sup>2+</sup> and Pb<sup>2+</sup> in the untreated soil were 7.59 and 10.9 , were within the range of active Zn<sup>2+</sup> reported by Ma and Lindsay (1993) . Alternatively , the present value of Pb<sup>2+</sup> activity is much lower than that reported by El-Gendi (1994)who measured Pb activities in non-polluted , moderately polluted and highly polluted soil using the competitive chelation method given in details by El-Falaky *et al.*, (1993) and mentioned that pb<sup>2+</sup> in the three soils were ranged from 6.27 to 7.63 M. Meanwhile, in the present study , it ranged from 10.9 to 11.50 M .

**Table (3): Input and output data of the tested soils expressed as pC (-logC).**

Treat	Input data															Output data	
	pH	Cd	pb	Co	Cu	Fe	Mn	Ni	P	Zn	Si	SO <sub>4</sub>	NO <sub>3</sub>	Cl	CO <sub>3</sub>	Pb <sup>2+</sup>	Zn <sup>2+</sup>
A	7.45	7.88	6.60	5.84	6.32	5.25	6.60	5.14	7.75	5.87	2.83	2.34	2.80	1.88	3.66	10.90	7.59
B	7.43	7.01	6.65	6.07	6.57	5.31	6.58	4.61	7.87	5.97	4.02	3.50	2.81	1.82	3.64	11.10	7.69
C	7.4	7.80	6.70	5.97	6.83	5.41	6.63	4.42	7.90	6.01	3.97	3.45	2.81	1.84	3.63	11.10	7.73
D	7.38	8.00	6.76	5.96	7.00	5.52	6.68	4.26	8.31	6.13	3.97	3.45	2.82	1.82	3.63	11.50	7.85

Data also indicate that the activities of Zn<sup>2+</sup> and Pb<sup>2+</sup> were markedly decreased as a result of phosphate enrichment and the values of Pb<sup>2+</sup> activities were much affected than that for Zn . The activities value of Zn and Pb were ; (7.69 and 11.0) , (7.73 and 11.10) and (7.85 and 11.50) , respectively, at P :Metal ratio of 3:50 (B) , 3:25 (C) and 3:5 (D).

**F- Impacts of the amendments on solid phases formation of the tested metals:**

Figures 3 and 4 ,respectively show the solubilities of various lead – phosphate and Zn-Phosphate minerals which are constructed according to Lindsay (1979).

Fig 3 indicates that the predicted values of Pb<sup>2+</sup>are governed by solubility of chloropyromorphite mineral , Pb<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>Cl in equilibrium with tricalcium phosphate (TCP), in untreated soil solution ; and with dicalcium phosphate dihydrogen (DCPD) ,in treated soil solutions . In this connection , El-Gendi (1994) reported that the measured Pb<sup>2+</sup> activities were within the range maintained by Pb<sub>4</sub>O(PO<sub>4</sub>)<sub>2</sub>, Pb<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>OH, and Pb<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> at equilibrium with either hydroxy apatite (HA) or by (TCP).The same conclusions were also obtained by Nriagu (1984) who stated the importance of lead –phosphate minerals in controlling lead solubility. It is clear therefore that with increasing level of added phosphate , Pb activities decreased sharply , due to formation of less soluble Pb- phosphate compounds, i.e., immobilization of soluble Pb in the soil .

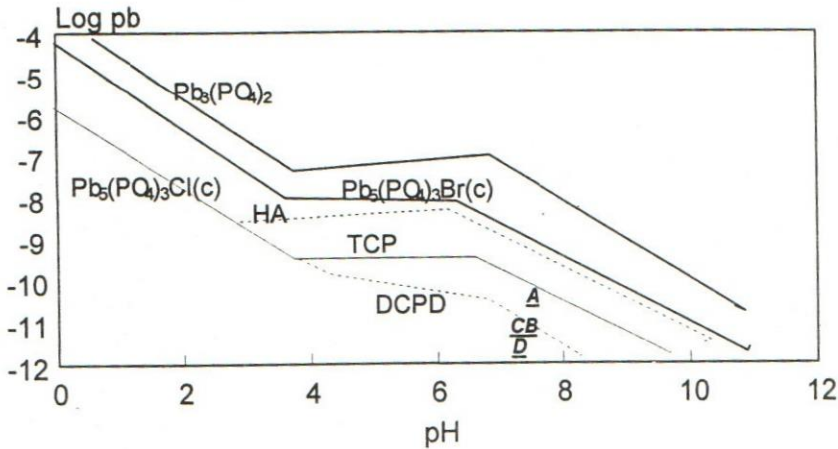


Fig.(4):Plotting Of  $Pb^{2+}$  values on the stability diagrams of lead phosphate minerals.

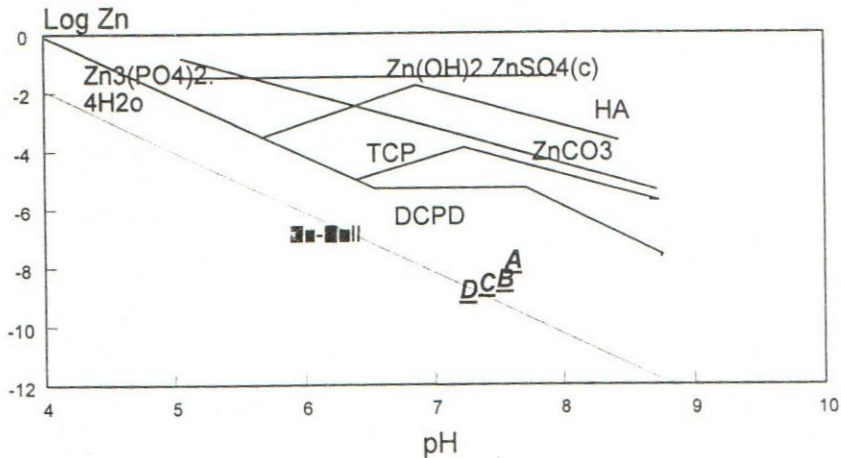


Fig.(3): Plotting  $Zn^{2+}$  values on the stability diagrams of some of Zn-minerals.

It appears from Fig. 4 that soil- Zn is the only mineral governing the solubility of  $Zn^{2+}$  activities in the tested soil solutions . The only difference is that the predicted value of  $Zn^{2+}$  in the untreated soil solutions tended to shift upward , referring to more solubility than that in the untreated soils. These findings disagree with results of Ma and Lindsay (1993) who reported that the measured  $Zn^{2+}$  activities were close to those of  $Zn_2SiO_4$  or  $ZnCO_3$ . However , the obtained results confirmed the usefulness of using di-ammonium phosphate to immobilize both Pb and Zn and decrease their toxicity in heavy metal polluted soils.



## CONCLUSIONS

Amending the soil with soluble phosphorous converted the relatively soluble pools (water soluble + exchangeable + carbonate) of the tested metals into more stable pools (Fe & Mn oxide + organically bounded + residual), resulting in decreasing mobility indexes of the metals.

Soluble phosphorous compound could quickly reduce the aqueous metals concentrations as well as decreasing metal activities and consequently their bioavailability

## ACKNOWLEDGMENT

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## معالجة بعض الفلزات الثقيلة في الاراضي الملوثة صناعيا باستخدام إحدى تكنولوجيات تقليل الذوبان .

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معهد بحوث الاراضي والمياه والبيئة - مركز البحوث الزراعية - الجيزة - مصر.

حيث أن الأراضي الملوثة بالفلزات الثقيلة تشكل مشاكل خطيرة لصحة الكائنات الحية .  
فإن عملية معالجة التركيزات العالية من تلك الفلزات أصبحت في الوقت الحاضر تلاقى كثير من  
الاهتمام . صممت الدراسة الحالية لاختبار قدرة ثنائي فوسفات الأمونيوم في تقليل درجة ذوبان كل  
من الرصاص والزنك في أراضي ملوثة صناعيا .

استخدمت أربعة نسب تمثل الكمية الكلية للفلزات الثقيلة في الأراضي المدروسة إلى كمية الفوسفور  
الذائب المضاف على أساس النسبة الذرية للرصاص إلى الفوسفور في معدن  
chloropyromorphite

ولقد أوضحت النتائج أن الكمية الكلية من الرصاص والزنك كانت أعلى من الحد  
المسموح به لتلك الفلزات والمقترحة بواسطة USEPA-503 . طريقة الاستخلاص المتتابع  
المستخدمة في هذه الدراسة كانت جيدة في توضيح المفصولات الكيماوية المختلفة لكل من  
الرصاص والزنك . أوضحت الدراسة أن الرصاص في الأراضي الملوثة صناعيا يتواجد أساسا  
على صورة مرتبطة بأكاسيد الحديد والمنجنيز ( حوالي ٤١ % من الكمية الكلية للرصاص ) بينما  
الزنك يرتبط بالصورة المتبقية ( ٣٧ % ) . مجموع الصور (الذائبة + المتبادلة + المرتبطة  
بالكربونات ) تعطي دلالة واضحة على أن جزء كبير من الفلزات المدروسة تتواجد على صورة  
عالية الصلابة . أدت إضافة DAP إلى تحويل الصور القابلة للذوبان إلى الصور الأقل ذوبانا .  
فنجد أن الزنك توزع كآلاتي : المتبقية <الأكاسيد> الكربونات <المرتبط عضويا <المتبادل <الذائب  
في الماء. في حين توزيع الرصاص كان كآلاتي : الجزء المتبقية = الأكاسيد <المرتبط عضويا  
<الكربونات <المتبادل <الذائب في الماء. أوضحت النتائج كذلك أن معامل حركية كل من  
الرصاص والزنك انخفض من ( ٢٠,١ إلى ١١,١ ) ومن ( ٢٨,٢ إلى ١٥,٤٤ ) في الأراضي الملوثة  
والأراضي الملوثة المعاملة بالفوسفات على الترتيب. كذلك انخفض تركيز الجزء النشط من تلك  
المعادن نتيجة تلك الإضافات (معبرا عنه بالتركيز السالب للتركيز المولر) لكل من الرصاص و  
الزنك على الترتيب كآلاتي ( ١٠,٩ إلى ١١,٢٣ ) وكذلك من ( ٧,٥٩ إلى ٧,٧٥ ) في الأراضي  
الملوثة والأراضي الملوثة المعاملة بالفوسفات على الترتيب . وبالتالي توضح هذه النتائج أن ثنائي  
فوسفات الأمونيوم له قدرة على تحويل الرصاص والزنك إلى مركبات أقل ذوبانا في الأراضي  
الملوثة صناعيا .

كذلك أوضحت نتائج منحنيات الذوبان أن معدن Chloropyromorphite عند اتزان  
مع TCP هو المتحكم في ذوبان الرصاص في الأراضي الملوثة الغير معاملة ، أما في حالة  
الأراضي المعاملة فيكون عند اتزانه مع DCPD . في حين منحنيات ذوبان الزنك ، أوضحت أن  
Zn-soil هو المتحكم في ذوبان الزنك في محاليل الأراضي المدروسة .