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Heavy Metals Content of Commercial Inorganic Fertilizers Used in the Kingdom of Saudi Arabia

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محتوى الأسمدة المعدنية المستخدمة في المملكة العربية السعودية من العناصر الثقيلة

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خلاصة: زاد الاهتمام في الآونة الاخيرة بالعناصر الثقيلة السامة، والتي يمكن ان تدخل السلسلة الغذائية للإنسان. و تعتبر الأسمدة المعدنية أحد المصادر التي يمكن أن تسهم في اضافة تلك العناصر إلى البيئة. تم تحليل ٧٤ عينة من الأسمدة المعدنية التي يجرى تسويقها واستخدامها في المملكة العربية السعودية لتقدير محتواها من العناصر الثقيلة. تمثل هذه الاسمدة أربع مجاميع رئيسية هي: الأسمدة الفوسفاتية (٢٠ عينة)، الأسمدة السائلة (١١ عينة)، الأسمدة الذائبة في الماء متعددة العناصر (٣١ عينة) و الأسمدة الصلبة متعددة العناصر (١٢ عينة). تبين أن تركيز العناصر الثقيلة في الأسمدة يعتمد أساساً على نوع السماد والعنصر الثقيل، وأظهرت النتائج أن تركيز عنصر الكروم Cr في الأسمدة كان هو الأعلي بينما كان تركيز الكوبالت Co هو الأدنى. أوضحت النتائج أن تركيز الكادميوم في الأسمدة المختلفة كان يتراوح مابين أقل من ١ إلى ٣٦,٨ ملجم/كجم وكانت قيم الوسيط له ٣٢,٢ و ٩,٥ و ١٩,٧ و ٢,٩ وذلك للأسمدة الفوسفاتية والأسمدة السائلة والأسمدة الصلبة والأسمدة الذائبة على التوالي. كان الوسيط لقيم الرصاص والنيكل والكوبالت والكروم في الأسمدة الفوسفاتية هو ١٤,٣ و ٧٢,١ و ١١,٨ و ٢٤٩,٣ ملجم/كجم على التوالي. بينما كانت قيم الوسيط لتلك العناصر في الأسمدة السائلة ٩,٩ و ١٥,٦ و ١١,٢ و ٦٤ ملجم/كجم وفي الأسمدة الصلبة ١٥,٣ و ٤٣ و ١٢,٥ و ١٧٠,٧ ملجم/كجم. بينت النتائج إن قيمة الوسيط في الأسمدة الذائبة ٩,٨ ملجم/كجم بالنسبة للرصاص، و ٥,٦ ملجم/كجم للكوبالت و٧,٤ ملجم/كجم للنيكل. أظهرت النتائج أن تركيز العناصر الثقيلة الأربعة تحت الدراسة كان أدنى من الحد الأعلى (١٠٠ ملجم/كجم للكادميوم و ١٠٠ ملجم/كجم للكروم و ٥٠ ملجم/كجم للنيكل) المسموح به لتلك الأسمدة، وفي حدود الكميات المسجلة عالمياً. وقد وجد أن إضافة ٨٠ كجم فوسفور إلى الهكتار سنويا يمكن أن تضيف ١٢ جرام كادميوم للهكتار.

ABSTRACT: In recent years much concern has been given to toxic heavy metals which enter the human food chain. Application of inorganic fertilizers is considered one of the potential routes of such entry. In this work 74 samples of commercial fertilizers marketed in the Kingdom of Saudi Arabia were analyzed for their heavy metal concentrations. Fertilizer samples included 20 samples of phosphatic fertilizers [monoammonium phosphate (MAP), diammonium phosphate (DAP) and triple superphosphate (TSP)], 11 samples of liquid fertilizers, 31 samples of water-soluble multiple nutrient fertilizers (WSMF) and 12 samples of solid multiple nutrient fertilizers (SMNF). Concentrations of heavy metals varied according to the type of fertilizer and the tested metal; Cr levels were the highest and Co was the lowest. Results revealed that Cd ranged from < 1 to 36.8 $mg.kg^{-1}$, with a median of 33.2 $mg.kg^{-1}$ for the phosphatic fertilizers, 9.5 $mg.kg^{-1}$ for the liquid fertilizers, 19.7 for the SMNF, and 2.9 $mg.kg^{-1}$ for the WSMF. The median values of Pb, Ni, Co and Cr in the phosphatic fertilizers were 14.3, 72.1, 11.8 and 249.3 $mg.kg^{-1}$, respectively. However, the corresponding median values of these elements, in the liquid fertilizers, were 9.9, 15.6, 11.2 and 64.0 $mg.kg^{-1}$ and 15.3, 43.0, 12.5 and 170.7 $mg.kg^{-1}$ in the SMNF. The median contents of three heavy metals in the WSMF samples were 9.8 $mg.kg^{-1}$ for Pb, 5.6 $mg.kg^{-1}$ for Co, and 7.4 $mg.kg^{-1}$ for Ni. The Cd, Co, Cr and Ni concentrations were lower than the tolerance limits for heavy metal addition (Cd 100 $mg.kg^{-1}$, Cr, 100 $mg.kg^{-1}$ and Ni, 50 $mg.kg^{-1}$), and they were in a range similar to those found internationally. It is estimated that the application of an average dose of 80 kg P ha^{-1} annually in Saudi Arabia, contributes 13 g of Cd ha^{-1} to the soil.

Keywords: inorganic fertilizer, toxic heavy metals, Saudi Arabia.

Increased concern about the contamination of soil and water resources with heavy metals has been shown in recent years. Adverse health effects consequent upon consumption of contaminated feeds have also received much attention (Singh, 1991; 1994). Heavy metals occur naturally in all soils in minute quantities, but can accumulate in agricultural soils from various sources, such as fertilizers, organic supplements,

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atmospheric deposition and urban and industrial activities. Some of these metals are not essential nutrients for plants and animals. However, sufficiently high concentrations can become toxic and constitute serious health problems whenever they enter into the human food chain (Oliver, 1997).

Schroeder and Balassa (1963) were the first to identify that fertilizers were implicated in raising some heavy metal concentrations in food crops, and since then much work has been performed to investigate the impact of impurities in fertilizers on crop uptake of potentially toxic elements. The main source of fertilizer-derived heavy metals in soils is phosphatic fertilizers, manufactured from phosphate rocks (PRs) that contain various metals as minor constituents in the ores (Allaway, 1971; Kpomblekou and Tabatabai, 1994).

Kongshaug *et al.* (1992) gave a comprehensive account of some heavy metal concentrations found in various phosphate rock (PR) deposits. It is known that these heavy metals present as impurities in PRs, are transferred to the fertilizers during processing. Williams and David (1973) found a close relationship between concentrations of Cd in TSP and their respective PR sources in Australia. Also, Wakefield (1980) reported that Triple Superphosphate (TSP) contained 60-70% of Cd found in PRs. Analysis of fertilizers commercially marketed in India, Italy, Australia, New Zealand, England and USA indicated that all P fertilizers contained significant and varying amounts of heavy metals (Williams and David, 1973; Arora *et al.*, 1975; Pezzarossa *et al.,* 1990). Charter *et al.* (1993) showed that TSP, MAP and DAP marketed in Iowa in the USA contained variable concentrations of many trace and heavy metals.

Several studies have shown that heavy metals in phosphatic fertilizers can accumulate in soil and become readily available to plants (Williams and David, 1976; Lee and Keeney, 1975). This issue has been addressed by Sauerbeck (1992) who compiled a list of the concentration ranges of some elements present in PRs and compared them with their corresponding averages in the earth´s crust. He concluded that, in terms of fertilizer use, elements which can be considered to have a potential risk of accumulation in soils are As, Cd, Cr, F, Sr, Th, U, and Zn. In a more recent study, McLaughlin *et al.* (1996) assessed the potential for contamination by phosphate fertilizers and concluded that Cd and F would accumulate at faster rates than As, Pb, or Hg.

In the Kingdom of Saudi Arabia, inorganic fertilizers are the main chemicals used by the agricultural sector. It is believed that most growers in the Kingdom use excessive rates of inorganic fertilizers due to their relatively low cost. Average application rate of phosphatic fertilizers is about 80 kg of P $ha^{-1}yr^{-1}$. Presently, there are no regulations in the Kingdom of Saudi Arabia governing maximum permissible concentrations (MPCs) of heavy metals in fertilizers. Since there is a lack of information regarding the concentration of heavy metals in fertilizers marketed in the Kingdom, this work was initiated to assess the content of heavy metals in most of the fertilizers used in the Kingdom in order to evaluate the risks of their potential accumulation in soil.

Materials and Methods

Fertilizer materials were obtained from various companies dealing with agrochemicals. These materials represented most of the fertilizers marketed in the Kingdom of Saudi Arabia. Some of them were locally produced and the rest were imported. The fertilizer samples were grouped into four categories, namely; phosphatic, liquid, [water-soluble multiple nutrient fertilizers] WSMF, and [solid multiple nutrient fertilizers] SMNF.

The fertilizers were digested using the method of the Association of Official Agricultural Chemists (AOAC, 1984). A triplicate sample of 5.0 g was dissolved in 10-ml conc. HCl in a 100-ml beaker. The beaker was covered with a watchglass, and the contents were boiled on a hot plate for approximately 30 min. The contents were then evaporated to near dryness. After cooling, 20 ml of 0.1 M HCl was added, and the contents were gently boiled. The contents were quantitatively transferred into 100-ml volumetric flask by filtering through Whatman No. 2 filter paper. The residues were thoroughly washed with 0.1 M HCl and the volume was adjusted with the same solution to 100 ml. As for the liquid fertilizers, 5 ml of the fertilizer was dissolved in 10 ml conc. HCl and the rest of the analytical procedures were similar to those used for the other types of fertilizers.

The digest obtained was analyzed for Cd, Co, Cr, Pb and Ni using an atomic absorption spectrophotometer equipped with a graphite furnace (Perkin Elemer; Model Analyst 300). Detection limits for these elements were 0.003, 0.4, 0.1, 0.05 and 1.0 µgL, respectively.

To overcome the lack of certified reference materials (CRMs), we relied on the use of internal reference samples which were meticulously prepared according to the accepted procedures. The effectiveness of the methods used were tested by using spiked samples, (a test material with a known addition of analyte), that were later used as reference samples. These tests revealed that there was no significant bias in the method used and the values of the various metal contentsof these reference samples were considered acceptable. We also participated in local interlaboratory comparisons to gauge the accuracy of our analytical results.

The procedures used for the quality control of our analytical data were originally proposed by Kennedy *et al.* (1993) and later outlined as a standard routine procedure in the FAO Bulletin No. 74 (1998).

Results and Discussion

Metal concentrations representing 20 phosphatic fertilizers (MAP,DAP,TSP), 11 liquid fertilizers, 31 WSMF, and 12 SMNF are given in Tables 1-4. The maximum, minimum and average metal concentrations for the studied fertilizers (74 samples) are shown in Table 5. The data indicate that concentration of heavy metals varied considerably with metal and the type of fertilizers. Chromium levels were the highest (95.4 $mg.kg^{-1}$) and Co was the lowest (9.5 $mg.kg^{-1}$). The median concentration of Cr was highest in phosphatic fertilizers (249.3 $mg.kg^{-1}$), whereas its median concentration value in the WSMF was negligible, except in three fertilizer samples.

The median values of the heavy metal contents in phosphatic fertilizers were 14.3 $mg.Pb.kg^{-1}$, 33.2 $mg.Cd.kg^{-1}$, 72.1 $mg.Ni.kg^{-1}$, 11.8 $mg.Co.kg^{-1}$ and 249.3 $mg.Cr.kg^{-1}$ in the phosphatic fertilizers (Table 1). The corresponding values for the liquid fertilizers were 9.9 Pb, 9.5 Cd, 15.6 Ni, 11.2 Co and 64.0 Cr (Table 2). In the WSMF, they were 9.8, 2.9, 5.6, 7.6 and < 0.001 $mg.kg^{-1}$ (Table 3), and the SMNF they were 15.3, 19.7, 43.0, 12.5 and 170.7 $mg.kg^{-1}$ (Table 4).

In general, the concentrations of these heavy metals in the 20 phosphatic fertilizer samples were higher than those in the other types of fertilizers. Furthermore, the data showed that the concentrations of Pb, Co and Cr were relatively higher in the TSP fertilizer samples as compared to their concentrations in the other types of phosphatic fertilizers, e.g. MAP and DAP. The concentrations of these metals in the WSMF were low. This may be attributed to the production processes used in the manufacturing of WSMF that gives highly refined products.

The current data revealed that the concentrations of the heavy metals in the liquid fertilizers were remarkably variable. The SMNF exhibited the same trends as the liquid fertilizers. Apart from Co, the values of Pb, Cd, Ni, Cr were highly correlated with the P content in these fertilizers (Table 5). In contrast, analysis of 97 solid commercial fertilizers in Iowa (Charter *et al.*, 1993) indicated that the mean values, expressed in $mg.kg^{-1}$ were 11, 15, 17, 12 and 133 for Pb, Cd, Ni, Co, and Cr, respectively, in the TSP samples. They added that the corresponding values ($mg.kg^{-1}$) were 9.1,7.1, 17.0, 10 and 57 for the MAP samples and 9.8, 10, 19, 8.2 and 71 for the DAP samples. Finck (1992) compiled the average heavy metal concentrations in PR in different parts of the world. He estimated the average concentration of heavy metals in fertilizer to be 66, 165 and 189 $mg.kg^{-1}$ for Pb, Cd and Ni, respectively.

The higher phyto-availability of Cd in soil (McLaughlin *et al.*, 1996) urged many investigators to give that element more attention. In this research, the Cd concentration of phosphatic samples ranged from 22.7 to 36.8 $mg.kg^{-1}$, with a median value of 33.2 $mg.kg^{-1}$ (Table 1). However, its range of concentration was 1.9 - 27.1 $mg.kg^{-1}$ for the liquid fertilizers, 4.4 - 28.2 $mg.kg^{-1}$ for the SMNF and 0. 0 -32.7 $mg.kg^{-1}$ for the WSMF.

The obtained values for Cd were similar to those reported in other parts of the world. For instance, Cd

TABLE 1

Heavy metal concentrations ($mg.kg^{-1}$) in 20 phosphatic fertilizers.

No.	Fert.	Pb	Cd	Ni	Co	Cr
1	DAP	11.2	34.2	52.9	5.5	215.3
2		12.0	34.0	52.8	5.4	210.0
3		14.5	31.0	70.5	9.3	216.0
4		16.4	31.0	71.0	9.6	220.0
5		12.8	31.4	67.7	10.9	199.9
6		12.8	32.8	66.6	10.4	200.0
7		14.3	22.7	62.1	12.3	314.7
8		14.2	26.0	73.4	12.0	314.0
9		12.5	28.2	67.3	11.4	236.5
10		12.6	28.2	66.6	14.4	236.0
11	MAP	16.7	31.8	81.1	14.5	280.7
12		16.4	32.0	81.0	14.8	290.0
13		14.3	33.9	84.7	10.4	238.7
14		13.0	33.6	85.2	10.4	236.0
15		14.3	33.5	82.4	11.7	260.0
16		14.4	34.0	82.8	12.0	216.0
17	TSP	32.0	36.3	67.7	20.6	402.0
18		32.0	36.0	73.2	21.2	396.0
19		32.4	36.0	77.2	20.0	410.0
20		31.6	36.8	79.8	20.6	400.0
Maximum		32.4	36.8	85.2	21.2	410.0
Minimum		11.2	22.7	52.8	5.4	199.9
Mean		17.8	32.2	72.3	12.9	276.8
Median		14.3	33.2	72.1	11.8	249.3

TABLE 2

Heavy metal concentrations ($mg.kg^{-1}$) in 11 liquid fertilizers.

No.	Pb	Cd	Ni	Co	Cr
1	20.0	27.0	36.1	12.6	64.0
2	4.1	3.6	4.6	3.0	N.D**
3	48.5	7.3	14.5	17.7	51.7
4	10.7	7.5	14.7	17.6	50.7
5	10.0	27.1	34.3	7.5	200.0
6	9.9	25.0	31.0	8.9	203.3
7	10.0	2.1	5.6	21.1	tr*
8	9.8	1.9	5.8	21.1	tr*
9	8.1	18.0	25.6	8.6	159.0
10	8.3	18.3	25.5	8.1	142.7
11*	7.1	9.5	15.6	11.2	64.7
Maximum	48.5	27.1	36.1	21.1	203.3
Minimum	4.1	1.9	4.6	3.0	N.D
Mean	13.3	13.4	19.4	12.5	85.1
Median	9.9	9.5	15.6	11.2	64.0

tr* <0.10 $\mu g\ L^{-1}$
N.D** Non-detectable.

TABLE 3

Heavy metal concentrations (mg.kg^{-1}) in 31 water-soluble multiple nutrient fertilizers (WSMF) (mg.kg^{-1}).

No.	Pb	Cd	Ni	Co	Cr
1	8.9	12.0	3.9	7.1	tr*
2	9.9	3.6	4.3	5.8	tr.
3	8.4	2.9	4.9	8.0	tr.
4	8.7	2.7	4.3	6.7	tr.
5	15.2	3.1	6.5	8.3	tr.
6	12.0	3.4	5.9	8.2	tr.
7	11.5	3.1	6.6	6.1	tr.
8	8.6	2.1	4.2	4.1	tr.
9	13.7	2.5	6.0	9.7	tr.
10	12.5	3.5	5.6	5.8	tr.
11	14.4	2.4	6.6	8.4	tr.
12	8.2	2.6	4.1	4.8	tr.
13	6.5	2.6	4.3	6.2	tr.
14	9.3	3.0	5.6	6.3	tr.
15	6.9	1.6	2.5	4.2	tr.
16	12.9	3.0	6.4	7.6	tr.
17	10.4	2.9	5.2	7.9	tr.
18	11.1	3.6	5.3	5.7	tr.
19	9.8	2.7	5.3	6.5	tr.
20	8.3	1.8	5.6	4.5	tr.
21	9.8	3.1	6.9	8.5	tr.
22	11.8	5.7	11.9	10.8	29.3
23	17.3	5.8	9.9	15.2	tr.
24	8.3	2.5	6.2	10.5	tr.
25	0.0	0.0	0.0	0.0	tr.
26	7.6	4.1	7.3	11.9	tr.
27	11.7	32.7	45.5	8.0	236.0
28	8.7	2.7	5.3	7.9	tr.
29	13.8	14.3	31.1	7.7	164.0
30	4.9	1.3	8.0	5.8	tr.
31	7.9	1.9	5.9	10.7	tr.
Maximum	17.3	32.7	45.5	15.2	236.0
Minimum	N.D	N,D	N.D	N.D	N.D
Mean	10.0	4.5	7.8	7.4	12.5
Median	9.8	2.9	5.6	7.6	00.0

tr* <0.10 µg L^{-1} N.D** Non-detectable

concentration ranged from 6.8 to mg.kg^{-1} with a median of 8.1 in fertilizers marketed in Iowa, 1.5 - 9.7 mg.kg^{-1} in Wisconsin, (Lee and Keeney, 1975), 18 - 91 mg.kg^{-1} in Australian fertilizers (Williams and David, 1973) and >0.1 – 30 mg.kg^{-1} in Sweden (Stenstrom and Vahter, 1974).

The amount of Cd added can be estimated by considering the application rates of phosphatic fertilizers. In the Kingdom of Saudi Arabia, the average application rate of phosphatic fertilizers rarely exceeds 80 kg of P ha^{-1}.yr^{-1}. As the average Cd concentration of P fertilizers marketed in the Kingdom was found to be 32.2 mg kg^{-1}, the total expected amount of Cd annually applied would reach about 13 g of Cd ha^{-1}. In the current study the effects of these additions on soil Cd contents could be estimated, assuming all Cd remained in the 0 - 30 cm soil layer. Considering that the weight of dry soil in the surface (0-30 cm) is about 4500 tons ha^{-1}, the Cd additions in fertilizer would increase its concentrations in the 0 – 30 cm layer by 0.0026 mg.kg^{-1}. Assuming that the tolerable limit for

TABLE 4

Heavy metal concentrations (mg.kg^{-1}) in 12 solid multiple nutrient fertilizers (SMNF).

No.	Pb	Cd	Ni	Co	Cr
1	12.0	20.0	43.7	5.0	161.3
2	15.8	27.2	60.0	12.3	200.0
3	15.3	28.2	61.5	12.3	213.3
4	15.3	17.9	48.8	14.9	189.3
5	16.3	18.7	49.2	17.3	201.3
6	12.3	20.5	42.2	7.3	126.2
7	11.9	19.3	37.5	6.3	156.7
8	11.5	12.9	40.0	7.5	145.3
9	14.8	21.7	60.2	12.7	200.7
10	16.1	4.4	24.2	15.2	40.0
11	16.2	22.3	37.9	16.1	180.0
12	16.7	7.9	31.0	13.3	130.0
Maximum	16.7	28.2	61.5	17.3	213.3
Minimum	11.5	4.4	24.2	5.0	40.0
Mean	14.5	18.4	44.7	11.7	162.0
Median	15.3	19.7	43.0	12.5	170.7

Cd in soil is 2 mg.kg^{-1} (Fink, 1992), and the average Cd concentration of these soils is low (according to a recent investigation by the author; 0.066 mg.kg^{-1}) it could take several hundred years of P application at the commonly used rate of fertilizers, (80 kg.P.ha^{-1}) to reach the tolerable Cd limit.

Concentrations of Cd in fertilizers found here clearly demonstrate that Cd coming from inorganic fertilizers will unlikely have an impact on Cd contents in soils. In comparison, fertilizer from Florida phosphate, which contain less than 10 mg.Cd.kg^{-1}, contributed 0.3 - 1.2 g.Cd.ha^{-1}.yr^{-1} to soil in a long-term fertility experiment (Mortvedt, 1987). On the other hand, phosphatic fertilizer manufactured from western USA deposits containing an average of 174 mg.kg^{-1} Cd contributed 100 g.Cd.ha^{-1} to the soil in a 36-year field trial in California. This amount raised the concentration in soil from 0.07 mg.kg^{-1} in the control plot to 1.0 mg.kg^{-1} Cd in the fertilized plots. In this regard, Nriagu (1980) showed that phosphatic fertilizers, with an average Cd content of 7 mg.kg^{-1}, could contribute an amount of 660 t.Cd.yr^{-1} into the environment on a global scale.

TABLE 5

Heavy metal concentrations (mg.kg^{-1}) of all types of fertilizer combined (total of 74 samples).

	Pb	Cd	Ni	Co	Cr
Maximum	48.5	36.8	85.2	21.2	410
Minimum	N.D**	N.D**	N.D**	N.D**	N.D**
Mean	13.2	15.6	32.9	10.3	119.5
Median	12.0	12.5	25.6	9.5	95.5
r	**	***	***	NS	***

r: coefficient of correlation(P and heavy metals)

NS not significant; * Significant at p< 0.05; ** Significant at p< 0.01; *** Significant at p< 0.001.

N.D** Non-detectable.

Our data revealed that Cd, Co, and Ni concentrations were generally lower than the tolerance limits given for heavy metal addition to soil. Although Cr was relatively high, except in the WSMF, it is much lower than the tolerance limit for soil (100 $mg.kg^{-1}$). The data indicate that it would take hundreds years of P additions at the normal applied rates in order to reach the tolerable limits for most of the heavy metals. Nonetheless, other possible inputs of heavy metals to agricultural soils, such as organic fertilization as sewage sludge, aerial deposition and overdose of inorganic fertilizers, must not be ignored. It should also be noted that the quality of PR ores has been declining with time and the ores now used in the production of P fertilizers contain more impurities than those used two decades ago (Liekam, 1989). All of the aforementioned aspects should be taken into consideration for a precise evaluation of heavy metal accumulation in soil, and in monitoring environmental pollution.

Conclusions

The average heavy metal contents of the fertilizers marketed in the Kingdom of Saudi Arabia are within the limits of those used worldwide. The use of these fertilizers is not expected to cause detrimental effects with regard to heavy metals pollution. However, a slight annual increase could be expected. This increase, coupled with other possible inputs of heavy metals to agricultural soils, should raise the need to monitor changes that could occur in soils and to develop pollution control strategies.

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