Uranium leaching during short term application of pit-water on a carbonate containing soil in the Mendoza province of Argentina

Juan Pablo Bonetto[1], Silvia López[1], Silvia Ratto[2], Valeria Schindler[3], Ewald Schnug[4]

[1]Agronomic Group, Ezeiza Atomic Center, National Atomic Energy Comision. Presbítero Juan Gonzalez y Aragón 15. Ezeiza, Buenos Aires Province, Argentina, E-mail: jpbonett@cae.cnea.gov.ar
[2]Edaphology, Agronomic School, Buenos Aires University. Av. San Martín 4453, 1417, Buenos Aires City, Argentina.
[3]Animal Production, Agronomic School, Buenos Aires University. Av. San Martín 4453, 1417, Buenos Aires City, Argentina.
[4]Institute of Plant Nutrition and Soil Science, Federal Agricultural Research Center, Bundesallee 50, D-38116 Braunschweig, Germany, E-mail: pb@fal.de

Abstract. Pit-water from an Uranium (U) mine in San Rafael, Argentina, was applied to soil columns in a short-term experiment to evaluate retention of U. The mine soil was coarse-textured, with $pH_{(KCl)}$ 7.7 and a carbonate content of 6%, which may favour the formation of uranyl-carbonates. Triple superphosphate (TSP) and ground plant material were added as amendments to reduce U mobility in soil. Plants were sown to study their effect on U leaching. > 99 % of the U applied was retained by the soil. Plant growth increased U mobility but also reduced leachate volumes through evapotranspiration. TSP increased plant biomass, reducing the mass of U leached, while ground plant material enhanced leaching of U.

Introduction

The San Rafael open pit Uranium Mine and Processing Facility (CMFSR) is an 800 ha site lying in the Sierra Pintada, 30 km SE of the city of San Rafael (34.6°S, 68.4°W, elevation 227 m) in the province of Mendoza, Argentina.

After an almost 10 year relapse in the mining activities, its open-air pits have become flooded through groundwater inflow and surface water run-off initially used in the facility processes. Added to the risk of overflowing, since water balance favors water accumulation in spite of being a semi-arid region, a restarting of the activities is currently in study, and the pits would need to be emptied to allow the continuation of mining processes.

The pit water cannot be disposed in the surrounding water courses due to its U content of 3500 µg l^{-1}. Among the different treatment and disposal alternatives, also application of the water to the soil is under consideration, based on the proposition that the soil will retain the contaminants, minimizing their movement to the grounwater. Uranium (VI) mobility in soils may be limited by formation of low solubility complexes, and by adsorption, preferably to Fe and Mn oxy-hydroxides, organic matter (OM) and clays (Langmuir 1978; Ribera et al. 1996).

Field irrigation with U containing waters has been studied and put to practice in Australia with varying results (Riley 1992; Noller and Zhou 1992). Willet and Bond (1992) determined that the amount of U involved in land application of effluent water from Ranger Uranium Mine (Australia), could be immobilized by low OM, poor sorption capacity, highly weathered soils, even though the equally applied major ions were expected to be readily mobile in the soil profile (Bond and Willet 1992). The CMFSR soils, however, though also coarse-textured, present a slightly alkaline pH and a higher carbonate content than those of Ranger.

In solutions of neutral to alkaline pH, presence of carbonate ions induces the formation of stable uranyl-carbonate complexes (Finch and Murakami 1999). These highly soluble complexes are mainly neutral or negatively charged, minimizing adsorption to soil particles and enhancing U mobility (Elless and Lee 1998; Finch and Murakami 1999). In contrast, the presence of phosphates, may induce precipitation of highly insoluble uranyl-phosphate complexes (Finch and Murakami 1999). The low solubility and high stability of these complexes have led to several studies on the remediation of contaminated water and soils by U immobilization with phosphates, mainly apatites (Seaman et al. 2001; Knox et al. 2003).

Sorption to inactivated plant biomass such as that of *Larrea Tidentata* has also been studied as a means for heavy metal immobilization and extraction from contaminated solutions (Gardea-Torresdey et al. 1997). This plant species has been found growing in heavy metal contaminated areas in the arid southwest region of the United States, and subsequently studied and recognized as a heavy metal accumulator (Gardea-Torresdey et al. 1996). *Larrea spp*. (mainly *L. nitida* and *L. divaricata)* thrive in most of the CMFSR and its surroundings. Its use in the form of processed tissue, as fresh OM amendment, could prove a means of increasing U sorption capacity of the soil.

Presence of vegetation can reduce water percolation due to an increase in water demand through evapotranspiration (hydraulic control), thus limiting heavy metal leaching. Plants have also the potentiality of reducing U contamination in soils by extraction through roots (phytoextraction) (Dushenkov 2003). However, plants may also increase the risk of trace metal leaching through the soil. Root growth and exploration create macropores in the soil that may enhance solute transport through preferential flow (Gabet et al 2003). Furthermore, it is recognized that plant presence may favor contaminants leaching through organic acid roots exudates that complex metals and enhance their solubility, also by enhanced microbial activity in the rhizosphere (INEEL 2000).

A column experiment was carried out to determine U leaching during a short term application of pit water to a coarse textured, low OM, high-carbonate soil. Treatments included plant presence, and addition of soil amendments such as triple superphosphate fertilizer and processed *Larrea spp*. tissue to evaluate their effect on U leaching.

Materials and methods

Experimental design and statistical analyses

The experimental design was 2x3 factorial completely randomized with 5 replicates. The factors were *plant* (two levels: plants and no plants), *amendment* (three levels: no amendment, *Larrea spp*. tissue (Lr) and triple superphosphate fertilizer (TSP)). The resultant 6 treatments and their abbreviations were: No plants, no amendment (NN); no plants, Lr (NL); no plants, TSP (NP); plants, no amendment (PN); plants, Lr (PL) and plants, TSP (PP). Statistical analysis of the data involved analysis of variance (ANOVA) using the General Lineal Model (GLM) and analysis of regression using the Regression Procedure (REG) of the Statistical Analysis System (SAS Vs. 8).

Soil and amendments

The soils in the area surrounding the CMFSR are classified as an association of Typic Paleorthids (70%) and Typic Torrifluvents (30%) (Hudson et al., 1990). The upper 20 cm layer of soil was sampled from an unaltered area of the mine, of which some properties are presented in Table 1.

The soil was air dried, passed through a 2 mm sieve, and homogenized prior to packing the columns. Commercial TSP was finely ground and applied as amend-

Table 1. Selected properties of soil, amendments and pit water used in the experiment.

	Soil	TSP	Lr	Pit-water
U (µg g^{-1})	2.4 †	84.0	0.1	3500 (µg L^{-1})
Total Phosphorus (µg g^{-1})	641	$2\ 10^5$		
Phosphorus $_{available}$ (µg g^{-1})	0.98	$2\ 10^5$		
PH $_{(KCl)}$	7.7			7.2
$C_{organic}$ (%)	0.36			
Carbonates (%)	6			
Texture	Loamy sand			

† Within baseline values for soils in San Rafael.

ment. Twigs with leaves were cut from *Larrea spp*. plants from uncontaminated areas around the CMFSR. They were oven dried, finely ground and homogenized before use as amendment. Water sampled from the pit was used for irrigation. Some properties of the pit-water and the amendments are also presented in Table 1.

Column preparation

30 soil columns were built out of 10 cm diameter polyvinylchloride pipes, and cut to 33 cm length. Each pipe was fitted with a bored end-cap to allow for the liquids to leach. A voile cloth filter was placed at the bottom to prevent loss of fines. 318 g of acid-washed sand were added to each column as a filtering bed, covering the bottom 2 cm of the columns. Over the sand, an initial 3311 g of soil were packed, equivalent to a depth of 27 cm. The soil was added in various steps, slightly tapping the columns and humidifying. The columns were then randomly selected to receive each of the 6 treatments. A final 376 g of soil, equivalent to a 3 cm layer, were thoroughly mixed with 88.2 mg TSP, 1000 mg Lr, or shaken without amendment, and added to each column, giving concentrations of 47 mg kg soil^{-1} P and 2660 mg kg soil^{-1} Lr for the last 3 cm layers. *Agropyron elongatum* seeds were planted in separate trays with acid-washed sand. After 16 days cultivation on Hoagland solution, 30 seedlings were transplanted to the corresponding packed columns. The finished columns were placed in wooden supports specially built for the purpose. Each leachate was collected in a plastic 0.5 L bottle through a plastic funnel. The experiment was carried out in a growth chamber providing a 12 hour light period and a controlled temperature.

Irrigation and U analyses

Before initiating pit-water irrigation, distilled water was applied to all columns until field the first drops of gravitational water started to leach. The experiment commenced on first application of pit-water. Irrigation was manually applied employing controlled slow flow from a burette, ensuring that all columns received the same daily volume of water. It was applied in an amount enough to keep all columns slightly over field capacity, obtaining a minimum of leachate every day. At the end of every week, leachates were collected, their volumes measured and compared against the amount of pit-water applied. Aliquots were taken and acidified with nitric acid prior to total U measurement by means of laser fluorescence employing a Scintrex UA-3 analyzer.

Results and discussion

Leachate volumes

Irrigation volumes were increased weekly (Table 2) to compensate for increasing water use due to plant growth, and to receive a minimum amount of leachates. For the first half of the experiment, vegetated columns leached less volume than unvegetated ones, but there was no difference in leachate volumes due to amendment effect (Table 2).

The results for the second half (Table 2) revealed interactions between factors *plant* and *amendment* for leachate volumes, so comparisons of the three amending levels were performed within each of the two plant levels. No differences were found among the amendments applied to the unvegetated columns. Where plants were growing, the PP treatment leached less volume than PN and PL. This had also been observed in week three, but the difference had failed to be statistically significant. Within each of the amendment levels, *A. elongatum* considerably re-

Table 2. Weekly irrigated and leached volumes and ANOVA for leachate volume.

	Irrigated volume (ml week^{-1})					
	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
All treatments	300	400	425	550	650	830
	Leachate volume (ml week^{-1})					
Treatment[1]	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
NN	139	138	172	270	405	587
NL	123	109	167	257	386	570
NP	132	135	174	271	404	586
PN	96	67	105	116	193	315
PL	94	76	100	121	197	317
PP	93	65	81	45	59	96
	Average					
No plants	131 **a**	127 **a**	171 **a**	266	398	581
Plants	94 **b**	69 **b**	95 **b**	94	150	243
No amendment	117	102	138	193	299	451
Lr	108	92	133	189	291	443
TSP	112	100	127	158	154	341
	Probability of F[2]					
Plant	***	***	***	***	***	***
Amendment	-	-	-	**	***	***
Plant x Amend.	-	-	-	***	***	***
CV (%)	10.2	19.3	11.0	10.4	9.4	6.9

[1]NN = no plants, no amendment; NL = no plants, processed *Larrea spp*. tissue; NP = no plants, triple superphosphate; PN = plants, no amendment; PL = plants, processed *Larrea spp*. tissue and PP = plants, triple superphosphate.
[2]Probability of F (- = $p > 0.05$; * = $p < 0.05$; ** = $p < 0.01$; *** = $p < 0.001$). Means in columns followed by the same letter are not significantly different at $p \leq 0.05$ level (Fisher´s LSD test)

Table 3. Regression equations for the relationship between irrigated volume (X) and leachate volume (Y) .

Treatment[1]	Regression	Probability of F[2]	R^2 (%)
NN	Y = 0.913 X - 195	***	95.6
NL	Y = 0.912 X - 211	***	95.4
NP	Y = 0.920 X - 200	***	96.2
PN	Y = 0.441 X - 83.4	**	86.0
PL	Y = 0.447 X - 84.2	**	88.2
PP	Y = 0.001 X + 72.4	-	0.0

[1]NN = no plants, no amendment; NL = no plants, processed *Larrea spp*. tissue; NP = no plants, triple superphosphate; PN = plants, no amendment; PL = plants, processed *Larrea spp*. tissue and PP = plants, triple superphosphate.
[2]Probability of F (- = p > 0.05; * = p < 0.05; ** = p < 0.01; *** = p < 0.001).

duced leachate volumes. The volume of water evapotranspired from vegetated columns resulted higher than that evaporated from unvegetated ones. Plant growth increased evapotranspiration. Dry matter yields, weighed at the end of the experiment, resulted higher both in roots (Pr < 0.0001) and in shoot (Pr < 0.0007) for PP plants than for PN and PL plants. The soil had a very low plant available Phosphorus (P) content, therefore the plants in the PP treatment benefited from the additional P supplied by the TSP to produce higher biomass, so leachate volume was lowest. Additionally, reduced plant growth may have occurred in treatments PS and PL due to U toxicity. U present in calcareous soils is expected to be not only mobile but also highly available to plants, favoring U phytotoxicity in greenhouse experiments (Shahandeh and Hossner 2002; Meyer et al. 2004; Lamas 2005). P addition has been found to alleviate toxic effects of U, probably due to complexation, reduced solubility and consequently a decrease in U availability to plants (Ebbs et al. 1998). The addition of TSP in the PP treatment may have prevented, or at least reduced, this.

Results of linear regression analyses revealed a close significant positive linear relationship between leachate volumes and irrigated volumes for all treatments except for PP (Table 3). This indicates that virtually all increase in leachate volume can be explained by increasing volumes irrigated. Treatments NN, NL and NP had very similar regression models, suggesting that there was no specific amendment effect. The models for PN and PL were also very similar to each other, and the resulting R^2 was only slightly lower than those in the no-plant treatments. This suggests that part of the irrigated water was used by plants, and that every weekly increase in irrigation volume exceeded any increasing need of the plants for water. The leachate volume in PP showed no relationship to the irrigated volumes. Being the treatment with highest plant growth, this suggests that the weekly increases in irrigation were almost completely used by the plants.

Uranium concentration in leachates

For all of the treatments U concentration in the leachates was much lower than in the applied pit water (Table 4). Since week 2, leachates from columns with plant growth had higher U concentrations than those from unvegetated columns. Week 5 showed an interaction between plant presence and amendment, but plant presence again increased U concentration in all three amendment levels. Apart from the probability of preferential flow generated by the roots (Gabet et al. 2003), rhizosphere exudates might also be at least partly responsible for the increased U concentrations. Higher metal mobility in soils due to plant presence has been reported by Banks et al. (1994) for Zn in a short-term greenhouse experiment, and by Zhu et al. (1999) for Cd and Zn in leachates from a 1 year duration column experiment, attributing this to an increase in metal solubility due to complexation with organic compounds exuded by roots and rhizosphere microorganisms.

Amendment effect was significant only for weeks 1 and 2, where Lr increased U concentrations compared to TSP and no amendment (Table 4). Lr also produced higher U concentrations in the following four weeks in unvegetated columns. Though these differences failed to be statistically significant, they clearly indicate an effect of Lr amendment not produced by TSP amendment. In vegetated col-

Table 4. U concentration in leachates and ANOVA results for U concentration in leachates.

	U concentration in leachates (μg U L^{-1})					
Treatment[1]	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
NN	46	83	68	47	28	25
NL	84	132	88	77	43	30
NP	53	82	76	53	24	21
PN	59	116	140	140	102	101
PL	98	165	157	156	122	155
PP	53	134	146	186	188	149
Average						
No plants	61	99 **b**	77 **b**	59 **b**	32	25 **b**
Plants	70	207 **a**	148 **a**	161 **a**	137	135 **a**
No amendment	52 **b**	99 **b**	104	93	65	63
Lr	91 **a**	148 **a**	122	116	82	92
TSP	53 **b**	108 **b**	111	119	106	85
	Probability of F[2]					
Plant	-	*	***	***	***	***
Amendment	***	*	-	-	-	-
Plant x Amend.	-	-	-	-	**	-
CV (%)	21	27	37	27	33	40

[1]NN = no plants, no amendment; NL = no plants, processed *Larrea spp*. tissue; NP = no plants, triple superphosphate; PN = plants, no amendment; PL = plants, processed *Larrea spp*. tissue and PP = plants, triple superphosphate.
[2]Probability of F (- = $p > 0.05$; * = $p < 0.05$; ** = $p < 0.01$; *** = $p < 0.001$). Means in columns followed by the same letter are not significantly different at $p \leq 0.05$ level (Fisher´s LSD test).

umns, from week 3 onwards, both amendments alternately increased U concentration as compared with unamended columns, though again without statistical significance. Similarly to the discussed effect of root exudates, Lr mixing with the soil may have enhanced U mobilization due to the organic source of the amendment. As discussed by Kim (1991), trace elements, including U, are proportionally higher in groundwaters whose colloids have higher dissolved organic carbon contents, implying bondage and transport of trace elements on humic substances. In this experiment TSP also appears to be responsible for increased U concentrations, but more likely by enhancing the plant (root) effects due to the increased plant biomass, since no incidence of TSP in U concentrations of unvegetated columns was found.

Results of linear regression analyses (Table 5) showed a significant negative linear relationship between U concentration in leachates and irrigated volumes only for the unvegetated treatments. No linear relationship was found in the vegetated ones. For these analyses, values from week 1 were not included, since the distilled water applied to the columns before irrigation with pit water may have reduced U concentration of the first leachates collected.

Weekly increases of irrigated volume meant weekly increases in mass of applied U to the columns, since U concentration in the applied pit-water was constant. Therefore, for the unvegetated columns, decreasing U concentration with increasing leachate volumes indicated that the U retention capability of the soil was not exceeded during this experiment. Since vegetated columns packed the same soil, the lack of a linear relationship revealed U mobilization and leaching due to plant growth.

Table 5. Regression equations for the relationship between irrigated volume (X) and U concentration in the leachate (Y).

Treatment	Regression	Probability of F	R^2 (%)
NN	Y = - 0.132 X + 126	*	85.7
NL	Y = - 0.208 X + 193	*	83.3
NP	Y = - 0.152 X + 138	*	89.2
PN	Y = - 0.072 X + 161	-	43.4
PL	Y = - 0.037 X + 172	-	15.5
PP	Y = - 0.042 X + 136	-	9.0

[1]NN = no plants, no amendment; NL = no plants, processed *Larrea spp*. tissue; NP = no plants, triple superphosphate; PN = plants, no amendment; PL = plants, processed *Larrea spp*. tissue and PP = plants, triple superphosphate.
[2]Probability of F (- = p > 0.05; * = p < 0.05; ** = p < 0.01; *** = p < 0.001)

Mass of Uranium leached

Though only in a very small proportion of the total mass applied, pit-water U leached from the soil columns from the beginning of the experiment on (Table 6). Plant growth increased the amount of U leached in week 5, though this effect was not so important in TSP amended columns. An interaction between factors *plant* and *amendment* was produced in week 6 only, where the same effect of plant growth was observed in treatments without amendment and with Lr addition, but, again, not within TSP.

For both vegetated and unvegetated columns, Lr amendment increased the mass of U leached in comparison with TSP amendment and unamended soil. Statistical significance of this effect, however, was observed only for the three last weeks. Also in week 5 and 6, leached U was even lower in TSP amended treatments than in unamended ones. The mass of U leached was obtained as the product of leachate volume and U concentration in the leachates. As discussed before, higher mobility of U produced by Lr amendment increased U concentration in leachates, thus increasing the mass of U leached from the columns, since leachate volume was not affected by this amendment.

Likewise, higher U concentration in leachates of vegetated treatments was a re-

Table 6. Weekly applied mass of U. Amount and ANOVA for mass of uranium leached weekly.

	Applied U mass (μg $week^{-1}$)					
	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
All treatments	1050	1400	1487	1925	2275	2905
	Mass of U leached (μg $week^{-1}$)					
Treatment[1]	Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
NN	6	11	12	13	11	15
NL	10	14	15	20	16	17
NP	7	11	13	14	10	12
PN	6	8	15	16	19	31
PL	9	13	17	18	23	48
PP	5	8	12	8	11	14
	Average					
No plants	8	12	13	16	12 **b**	15
Plants	7	10	15	14	18 **a**	31
No amendment	6 **b**	9	13	14 **b**	15 **b**	23
Lr	9 **a**	13	16	19 **a**	19 **a**	32
TSP	6 **b**	9	12	11 **b**	10 **c**	13
	Probability of F[2]					
Plant	-	-	-	-	**	***
Amendment	***	-	-	**	***	***
Plant x Amend.	-	-	-	-	-	***
CV (%)	22	35	40	29	28	32

[1]NN = no plants, no amendment; NL = no plants, processed *Larrea spp*. tissue; NP = no plants, triple superphosphate; PN = plants, no amendment; PL = plants, processed *Larrea spp*. tissue and PP = plants, triple superphosphate.

[2] Probability of F (- = p > 0.05; * = p < 0.05; ** = p < 0.01; *** = p < 0.001). Means in columns followed by the same letter are not significantly different at p ≤ 0.05 level (Fisher´s LSD test).

sult of enhanced U mobility caused by plant growth. Thus, the combination of plant growth and Lr amending produced the highest mass of U leached. But plant growth also reduced leachate volumes, so that only by weeks 5 and 6 were the leachate volumes of PN and PL high enough, and the U concentration in NN and NL low enough, to yield significant differences due to plant presence. The higher biomass obtained with the application of TSP in PP columns produced such lower leachate volumes that, in spite of the resultant increase in U concentration, the mass of U leached resulted lower than in PN and PL.

Conclusions

A 6 week irrigation experiment with U containing pit-water was carried out on soil columns. The coarse textured, low organic-matter soil containing 6 % carbonates, retained > 99 % of the 11042 µg U applied during the irrigation. Plant presence in some of the columns enhanced U mobility, increasing U concentration in the leachates and the mass of U leached. Plant growth also reduced the leachate volume between 30 – 65 % through evapotranspiration. Thus, upon reaching enough biomass the mass of U leached could be reduced in comparison to unamended unvegetated soil. TSP amending produced an increment in plant biomass due to the soluble P addition to a soil with low plant available P, so that vegetated, TSP amended columns discharged the lowest mass of U. However, the amount of P thus applied resulted too low to produce any effect on U leaching from unvegetated columns. Lr amending also enhanced U mobility, increasing U concentration in leachates and also the mass of U leached, since it did not affect the leachate volume.

Application of U containing pit-water to this soil allows most of the contamination to be retained by the soil, preventing it to reach groundwater. However, the U concentration in most of the leachates, specially with plant presence, were above recommended critical values for drinking water of 15 - 30 µg l^{-1} (WHO 2004), and also the mass of U charged to the soil would remain a persisting thread for the food chain or other compartments of the natural environment. Therefore, only previous treatment of the pit water to lower U concentration below critical values would make irrigation acceptable in open systems, allowing for lower concentration in leachates with minimal charge to the soil.

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