Supporting Information

Desorption and migration behaviour of beryllium from contaminated soils: insight for risk-based management

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Result and discussion

Table S1: Desorption of Be from different LFLS soils using various batch leaching protocols (mean ± SD)

Leaching protocol	Soil	Leaching (µg/L)	L_r (%)	K_d (L/kg)
SARS	T1	6.27 ±0.12	2.40	834
	T2	7.08 ±0.9	2.76	724
	T3	6.30 ±0.06	2.45	816
	B1	6.72 ±0.10	2.34	854
	B2	6.09 ±0.11	2.33	858
	B3	6.14 ±0.03	2.07	966
	B4	6.22 ±0.05	2.22	900
	Range	6.09-7.08	2.07-2.76	724-966
	Mean	6.40 ±.08	2.37	848
MWEP	T1	1.69 ± 0.09	0.646	3080
	T2	1.41 ± 0.12	0.549	3640
	T3	1.70 ± 0.07	0.661	3040
	B1	1.63 ± 0.02	0.567	3500
	B2	1.58 ± 0.02	0.605	3280
	B3	1.27 ± 0.06	0.426	4660
	B4	1.61 ± 0.05	0.573	3500
	Range	1.27-1.70	0.426-0.661	3040-4660
	Mean	1.55 ± 0.06	0.575	3448
	T1	3.06±0.9	1.17	1710
	T2	2.85±0.2	1.11	1802
SPLP	T3	3.17±0.7	1.23	1626
	B1	2.95±1.0	1.03	1942
	B2	2.94±1.1	1.13	1770
	B3	2.66±0.8	0.90	2220
	B4	2.90±0.8	1.03	1942
	Range	2.85-3.17	0.90-1.23	1626-2220
	Mean	2.93±2.2	1.09	1834
TCLP	T1	18.0 ± 1.25	6.81	294
	T2	17.2 ± 0.31	6.6	304
	T3	17.5 ± 0.94	6.69	298
	B1	17.7 ± 0.41	6.07	330

	B2	16.0 ± 0.05	6.02	332
	B3	16.8 ± 0.09	5.55	360
	B4	19.4 ± 0.54	6.83	292
	Range	16.0-19.4	5.55-6.83	292-360
	Mean	17.5 ± 0.51	6.37	314

Table S2: Major physicochemical properties of LFLS soils and details are described the literature[1].

Parameter	Average value ± SD
pH-water (1:5)	6.06±0.13
pH-$CaCl_2$ (1:5)	4.60±0.17
CEC (cmol/kg)	5.41±0.40
% sand	55.7±2.3
% Clay	12.7±1.2
% Silt	31.8±2.3
Soil type	Sandy loamy
%Nitrogen (N)	0.21±0.06
%Sulphur (S)	0.02±0.005
%STC	4.27±0.70
%SOC	3.70±0.44
%SOM	6.37±0.75

Table S3: Regression relationship of Be desorption with pH, EC, DOC, different heavy metal (loid)s and non-metals during SARS study

Variables	R^2	P

pH	0.668	0.05
DOC	0.857	P< 0.05
EC	0.979	P< 0.05
Mn	0.984	P< 0.05
Ba	0.920	P< 0.05
Sr	0.987	P< 0.05
Zn	0.827	P< 0.05
Cd	0.601	P< 0.05
Co	0.538	P< 0.05
Mo	0.476	P< 0.05
Se	0.470	P< 0.05
P	0.530	P< 0.05
Al	0.239	P> 0.05
Si	0.451	P< 0.05
Fe	0.111	P> 0.05
Pb	0.094	P> 0.05
Cr	0.149	P> 0.05
V	0.225	P> 0.05
S	0.048	P> 0.05

Note: R^2=Linear regression correlation; P=Significance of correlation (correlations are significant when $P < 0.05$).

Table S4: Linear regression relationship among the variables during sequential leaching of Be from sandy loamy (LFLS), calcareous, vertisol soil.

	LFLS		Calcareous		Vertisol	
	R^2	P	R^2	P	R^2	P
pH	0.706	P< 0.05	0.066	P> 0.05	0.146	P> 0.05
EC (mS/cm)	0.870	P< 0.05	0.450	P> 0.05	0.085	P> 0.05
ORP (mV)	0.446	P> 0.05	0.669	P< 0.05	0.822	P< 0.05
DOC (g/kg)	0.332	P> 0.05	0.839	P< 0.05	0.175	P> 0.05
Al (mg/kg)	0.388	P> 0.05	0.015	P> 0.05	0.644	P< 0.05
Fe (mg/kg)	0.337	P> 0.05	0.139	P> 0.05	0.999	P< 0.05
Mn (mg/kg)	0.674	P< 0.05	0.725	P< 0.05	0.982	P< 0.05

Ba (mg/kg)	0.323	P> 0.05	0.564	P> 0.05	0.992	P< 0.05
P (mg/kg)	0.707	P< 0.05	0.878	P< 0.05	0.099	P> 0.05
S (mg/kg)	0.353	P> 0.05	0.967	P< 0.05	0.222	P> 0.05
K (mg/kg)	0.781	P< 0.05				
Na (mg/kg)	0.360	P> 0.05				
Ca (mg/kg)	0.802	P< 0.05				
V (mg/kg)	0.314	P> 0.05				
Cr (mg/kg)	0.315	P> 0.05				
Co (mg/kg)	0.283	P> 0.05				
Cu (mg/kg)	0.134	P> 0.05				
Zn (mg/kg)	0.005	P> 0.05				
As (mg/kg)	0.275	P> 0.05				
Sr (mg/kg)	0.578	P< 0.05				
Pb (mg/kg)	0.608	P< 0.05				

Note: R^2= Linear regression correlation; P=Significance of correlation (correlations are significant when P< 0.05).

Table S5: XRF data from different solid phases of LFLS soil using sequential leaching process.

Elements	**Fresh soil**	**F1**	**F2**	**F3**	**F4**
SiO_2 (%)	73.2	75.3	79.7	82.9	87.0
Al_2O_3 (%)	16.9	16.2	12.8	10.4	9.43
Fe_2O_3 (%)	5.96	4.90	4.10	3.59	0.406
P_2O_5 (%)	0.556	0.523	0.548	0.554	0.567
K_2O (%)	1.62	1.40	0.949	0.950	0.770
CaO (%)	0.538	0.216	0.189	0.177	0.151
TiO_2 (%)	1.22	1.20	1.17	1.16	1.23

BaO (ppm)	197	95.1	ND	ND	ND
Mn (ppm)	624	228	119	113	55.0
SO_3 (ppm)	0.213 (%)	882	0.130 (%)	29.6	ND
V_2O_5 (ppm)	267	213	216.2	163	79.5

Note: ND means “Not Detected”

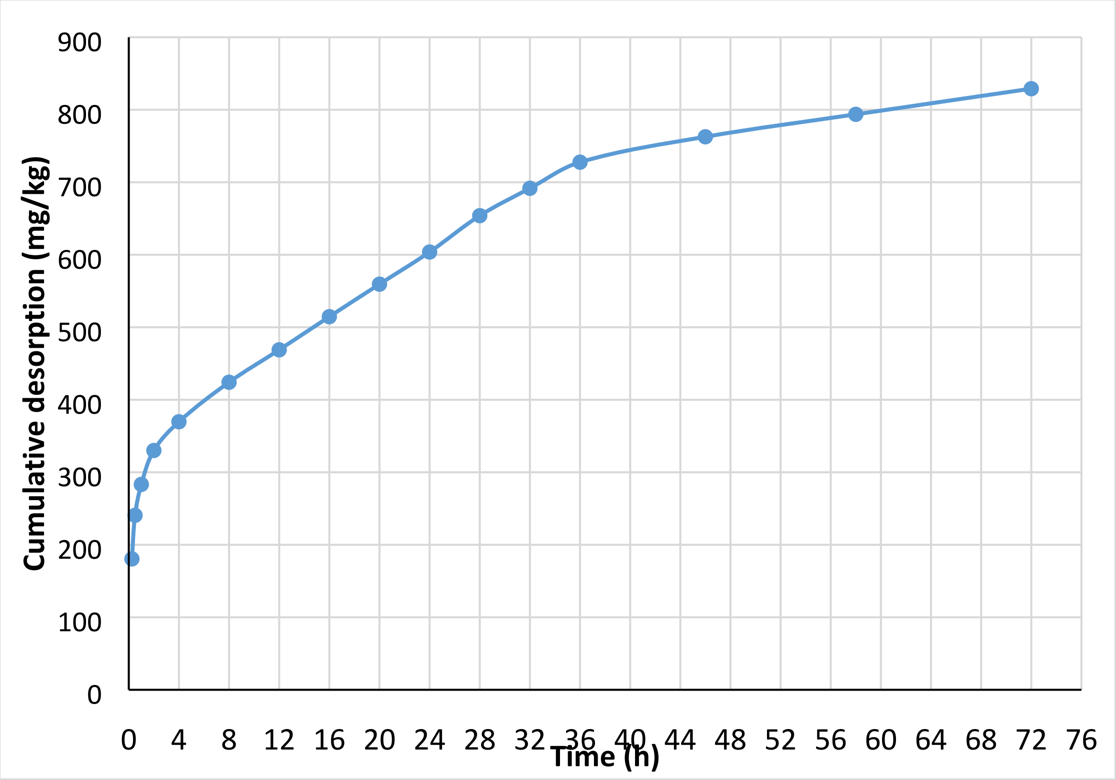

Figure S1: Cumulative desorption of Be using SARS which represents three discontinuous lines.

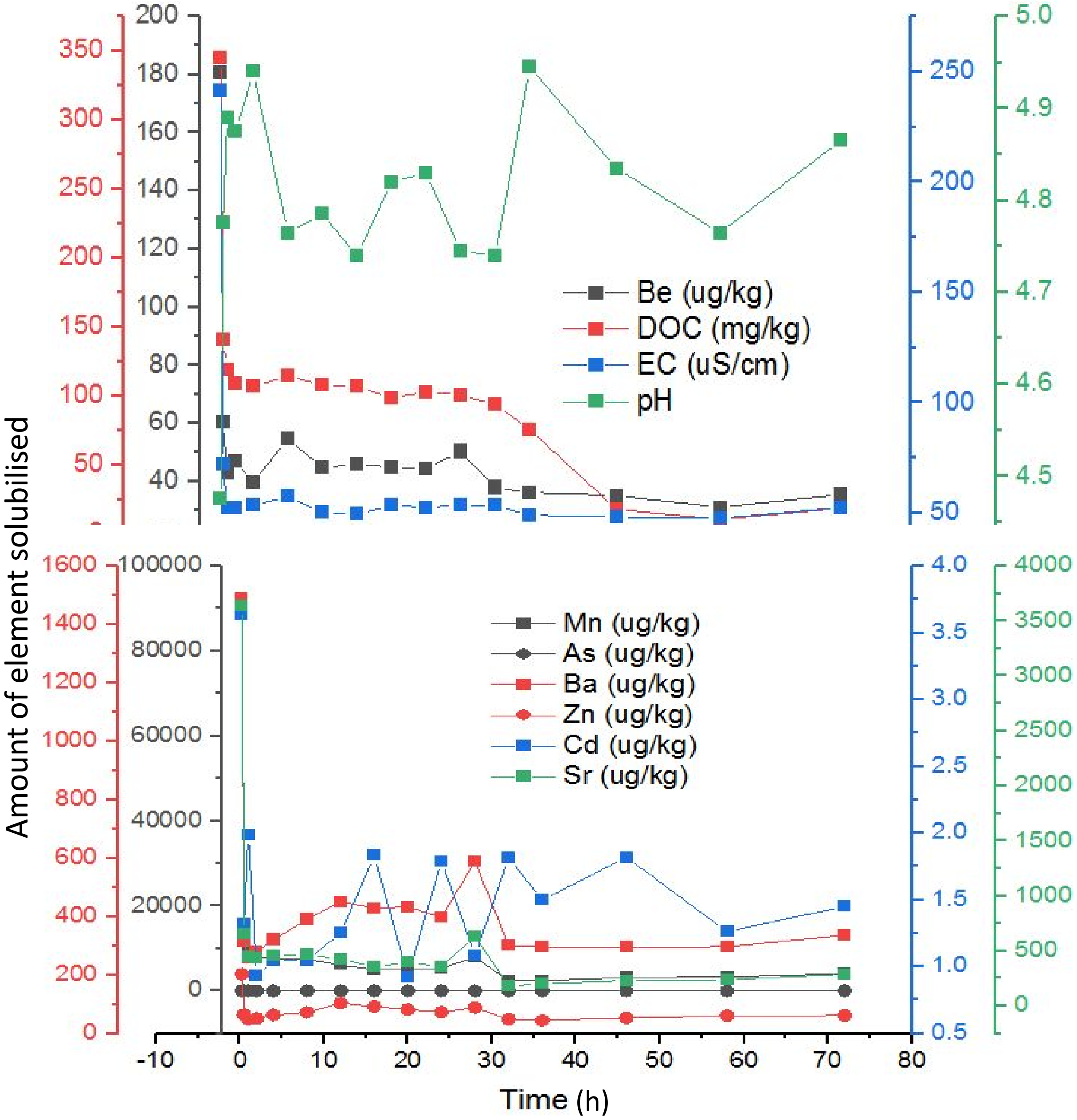

Figure S2: Kinetic dissolution of Be influenced by pH, EC (µS/cm), DOC (mg/kg), and different oxyhydroxides of metal solubility.

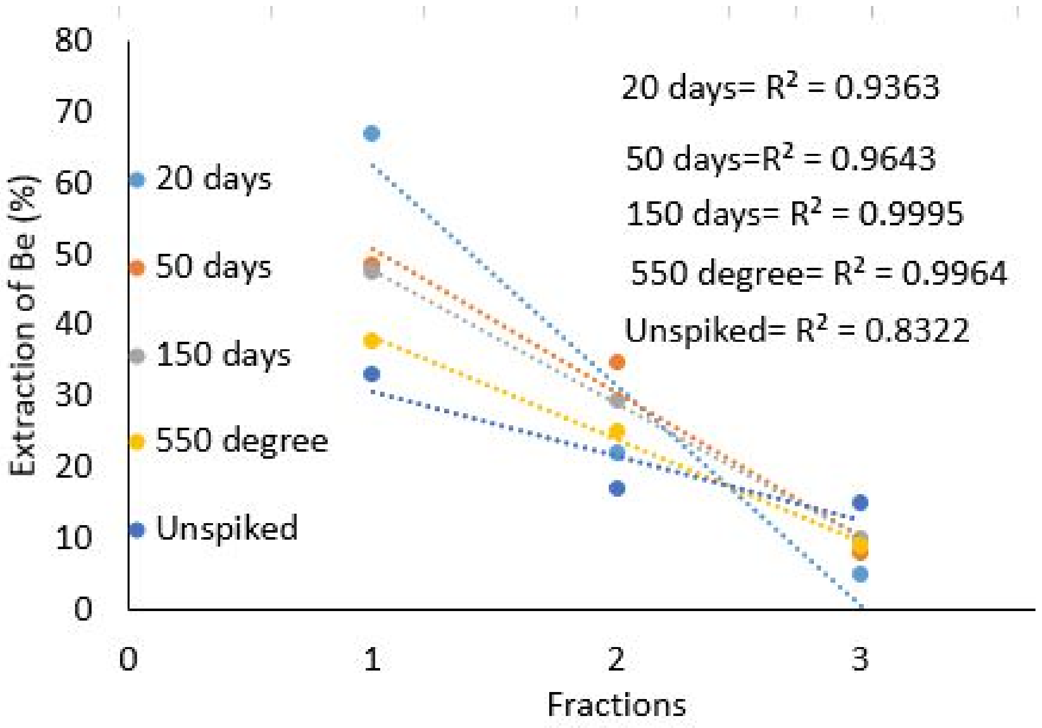

Figure S3: Linear regression relationship between desorption of Be and different fractions of BCR sequential leaching as a function of the incubation period.

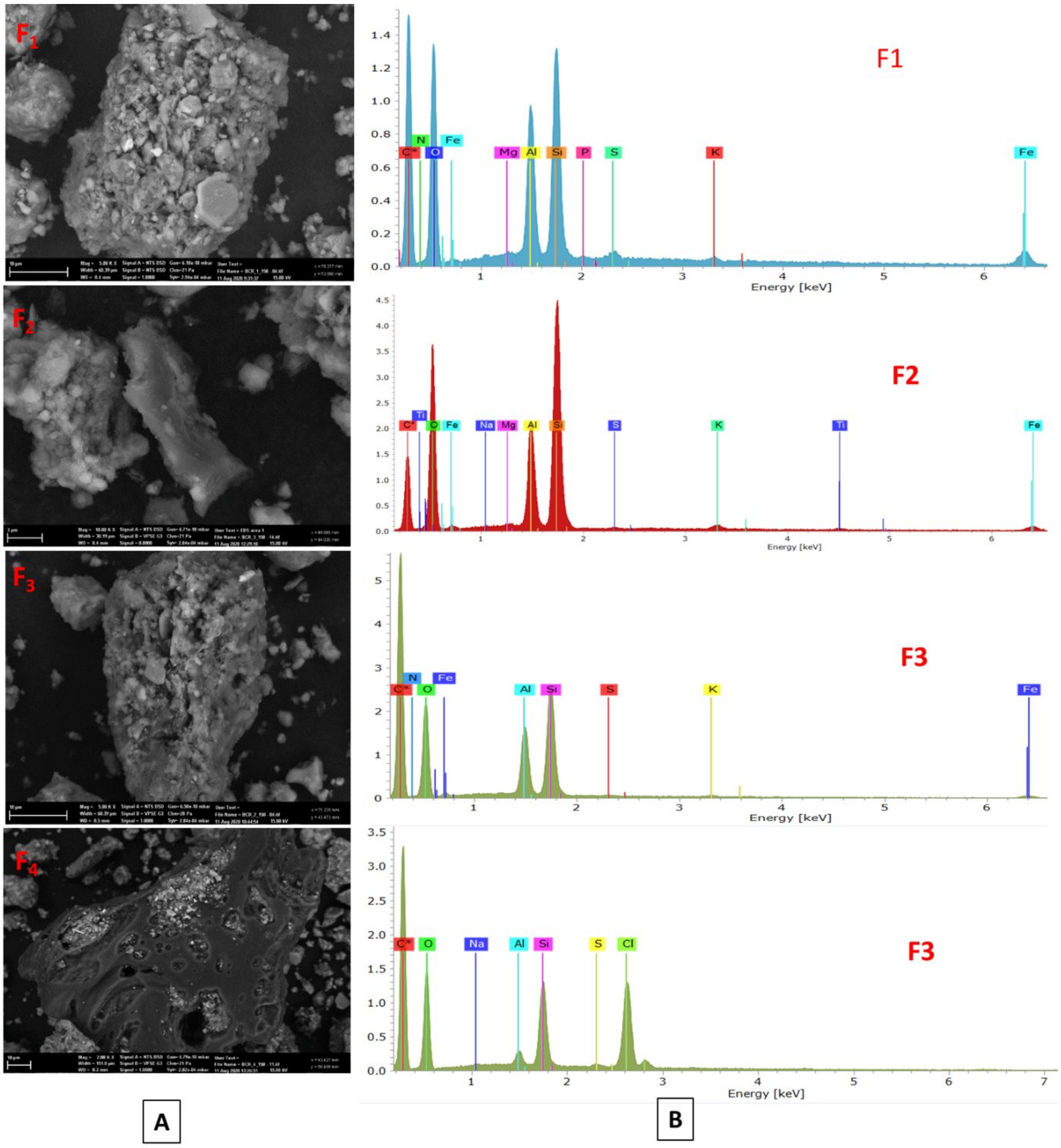

Figure S4: (A) SEM images of LFLS, and (B) SEM-EDS for major elemental composition of the four fractions (F1-F4) after Be leaching. The composition showed variation between particles but the figure is representative of the most abundant elements.

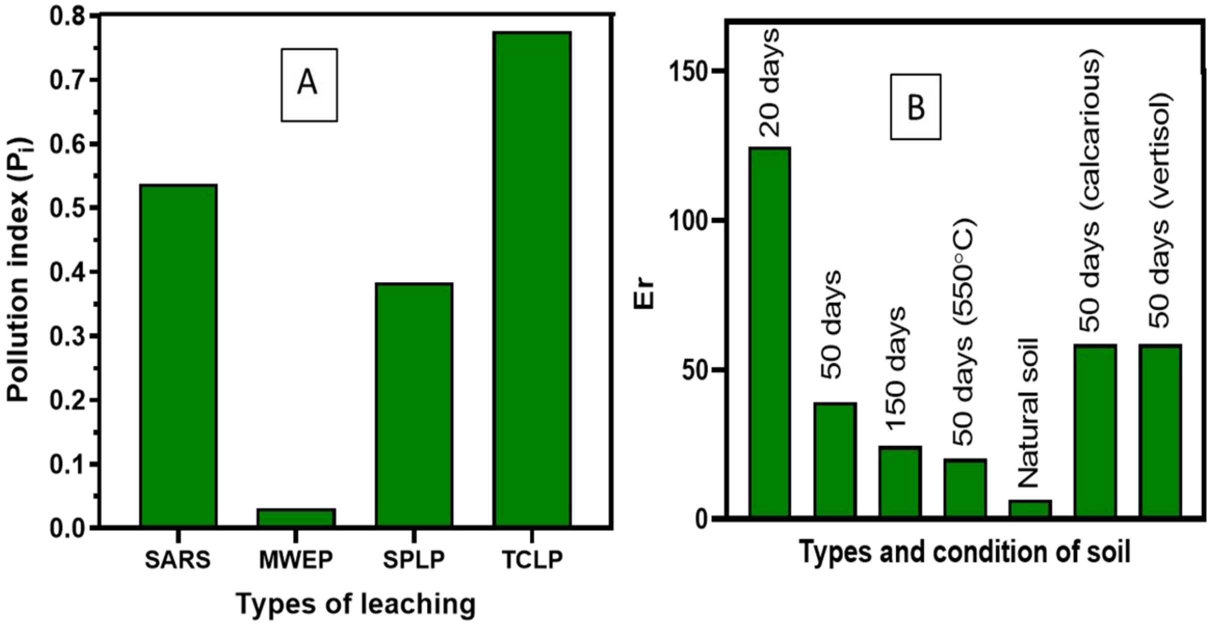

Figure S5: (A) pollution index of natural soil using batch leaching data; (B) Coefficient of potential ecological risk using sequential leaching data.

Methods and materials

Study area

We investigated the Little Forest Legacy Site (LFLS), which is located within the buffer zone boundary of the Australian Nuclear Science and Technology Organisation (ANSTO), on the southern periphery of Sydney, Australia. In the 1960s, according to standard practice at the time, the former Australian Atomic Energy Commission (AAEC) disposed low level radioactive waste with a significant amount Be (~1070 kg) in unlined trenches at the LFLS. The site is now under the management of ANSTO. The disposed waste materials (comprising a range of solid, semi-solids, liquids, sludges, etc.) were primarily derived from the operation of the research facility, including waste drums, chemicals, dis-used equipment, laboratory trash, radioactive waste (including various radionuclides ranging from fission products, Co-60, to actinides such as U-233 and plutonium), and, of particular relevance to the current study, significant amounts of Be [2]. A significant number of 44 gallon drums of waste were moved to the site, and these comprise a significant component of the buried wastes.

There are 79 waste trenches, were covered by a 1m local clay/shale soil cover across the trenched area of the site. The waste trenches are nominally 25 m long, 0.6 m wide, 3 m deep, and 2.7 m apart (Figure 8). Adjacent to the LFLS is a former landfill to the west, and other waste disposal sites (industrial chemicals, sewage effluent) are located to the east and northeast, which could influence background levels of Be and other contaminants. Runoff from the site could impact a local creek (known as “Turtle Creek”) on the southeast border of the site. In addition, the Barden Ridge suburb is 2.5 km to the east, and the Menai Ridge suburb is 3 km to the north of the study area.

Reagents

All reagents and chemicals used were as the analytical grade. The primarily used reagents were Hydrochloric acid (HCl), 37% (ACS grade reagent); Nitric acid (HNO_3), 70% (ACS grade reagent); Sulphuric acid (H_2SO_4), 98%; Sodium hydroxide ($NaOH$); Glacial acetic acid (CH_3COOH), 99.7 %; Humic acid (HA); Hydroxyl-ammonium chloride ($NH_2OH.HCl$); Hydrogen peroxide (H_2O_2); Ammonium acetate (CH_3COONH_4); Milli-Q water (18.2Ω).

Batch leaching of Be

Table S6: Simulated acid rain compositions in the Sydney region (Australia)*.

Ions	Concentration (µEq/L)	Concentration (µg/L)
Sodium (Na^+)	24.2	556.3
Magnesium (Mg^{2+})	6.1	74.13

Calcium (Ca^{2+})	9.0	180.4
Potassium (K^+)	2.2	86.0
Ammonium (NH_4^+)	18.6	335.5
Chloride (Cl^-)	119	4219
Nitrate (NO_3^-)	13.6	843.2
Sulphate (SO_4^{2-})	24.2	1162

Note: *The compositions of simulated acid rain solution were adopted from the following literature [3-7]. The ions were calculated and balanced from KCl, NH_4Cl, $CaCl_2$, $MgCl_2$, Na_2SO_4, HNO_3 and HCl.

Table S7: Chemical compositions and experimental conditions of different batch leaching

Leaching	Chemicals/Reagents	pH	Soil/Solution	Time (h))
SARS	See Table S6	4.5	1:20	18
SPLP (EPA 1312)	H_2SO_4/HNO_3 (60/40), DI water	5± 0.05	1: 20	18
MWEP (ASTM D 3987-85)	Reagent water	5.5±0.05	1:20	18
TCLP (EPA 1311)	0.11 M CH_3COOH; 1M NaOH; Mili-Q water.	4.93±0.02	1:20	18

Note: MWEP= Monofilled waste extraction procedure; SARS= Simulated acid rain solution; SPLP= Synthetically precipitation leaching procedure; TCLP= Toxicity characteristic leaching procedure.

Leaching capacity of soils was calculated by the following calculation (1).

$$Leaching\ capacity\ (LC) = \frac{Leaching\ (\mu g/L) \times V(L)}{W(kg)} \ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where LC, V and W are the leaching capacity (mg/kg), volume (L) of the leaching fluid and weight (kg) of soils, respectively. Leaching ratio (%), L_r was calculated using equation (2)

$$\%\ leaching\ ration\ (L_r) = \frac{Leachable\ Be\ (\mu g/kg)}{Total\ Be\ (\mu g/kg)} \times 100 \ldots\ldots\ldots\ldots\ldots(2)$$

Different kinetic models

The kinetic data were fitted with two constant rate model (eq. 3), parabolic diffusion model (eq. 4) and pseudo second-order kinetics model (eq. 5), to evaluate the nature of desorption reaction over time [8-10].

$$ln(L_t) = b\ln(t) + \ln(a) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

$$L_t = K_p t^{0.5} + L_0 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(4)$$

$$\frac{t}{L_t} = \frac{1}{K_2 L_e 2} + \frac{t}{L_e} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

Where, L_t is the amount of Be leaching or desorption (μg Be/kg) after time t (h) period, L_e is the amount leaching (μg Be/kg) at equilibrium, and L_0 (μg Be/kg) is that present at t=0 (h); 'a' is the initial Be desorption rate constant (μg Be/kg h); 'b' is desorption coefficient (μg Be/kg); K_p is the diffusion rate constant $(\mu g\ Be/kg)^{-0.5}$, K_2 represents the pseudo second-order rate constant (kg/μg h).

Maximum desorption was calculated according to the equation (6) [11].

$$\frac{1}{\mathrm{L_t}} = \frac{1}{\mathrm{L_{t(max)}}} + \frac{\mathrm{K_t}}{\mathrm{L_{t(max)}}}\frac{1}{t} \text{...}(6)$$

Where, $L_{t(max)}$ is maximum amount of desorbed Be (µg/kg), and K_t is the time required to reach half of its maximum desorption .

Calculation of thermodynamic parameter

The Gibbs free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) were calculated according to the equation.

$$LnK_d = \frac{\Delta S}{R} + \frac{\Delta H}{RT} \text{...}(7)$$

$$\Delta G = \Delta H - T\Delta S \text{..}(8)$$

Here, K_d, R and T are the distribution coefficient, molar gas constant (8.31416 J/K.mole) and Kelvin temperature, respectively.

Table S8: Modified BCR sequential leaching procedure for desorption of Be

Step/phase	Operational phase	Reagents and operational condition
F1	Water and acid soluble and exchangeable	Taken 1 g of dry soil, added 40 ml 0.11 M CH_3COOH, shaken 16 h (23 ± 3°C), centrifuged at 4500g, 15 min, supernatant analysed by ICP-MS.
F2	Reducible	Residue from step-1, added 40 ml of 0.5 M $NH_2OH.HCl$ (pH 2, with 0.05 M HNO_3), shaken, centrifuged, analysed as step-1.
F3	Oxidisable	Residue from step-2, added 10 ml of 8.8 M H_2O_2 (pH 2), 1 h (23 ± 3°C), heated to 85 ± 2 °C (water bath) for 1 h to reduce the volume by half; then 10 ml of 8.8 M H_2O_2 (pH 2), again

		heated for 1h, uncovered the tube and heated again (85 ± 2°C) for complete oxidisation of organic matter up to evaporate all the added solution; 50 ml of 1 M NH_4OAc added and shaken for 16 h, centrifuged and analysed as above.
F4	Residual	Residue from step-3, heated (60°C) to dryness, then finally to 105°C (overnight), homogeneous by grinding for aqua regia digestion to measure Be in the residue.

Note: In step-3, we observed splitting of 50 ml PP centrifuge tube from chemical reaction during heating (85 ± 3°C) with 8.8 M H_2O_2. To overcome this danger, the tube was left for 1h at room temperature after adding H_2O_2 with occasionally manual shaking. Then it was covered loosely and heated again at 85 ± 3°C with gently manually shaking at 10 min intervals.

Briefly, 0.11 M glacial acetic acid solution was prepared, ultra-sonicated for homogenisation, added the solution to LFLS soil (40 mL:1 g) arising from different incubation periods (20, 50, 150 days), shaken (16 h) in an end-over-end shaker 60 rpm). pH, EC, oxidation-reduction potential (ORP) were recorded just prior to centrifugation (4500g for 15 minutes at 10 °C). After centrifuge, about 10 mL of the supernatant was collected using 0.45 μm syringe filter, and the rest of the supernatant was decanted carefully. About 20 mL of milli-Q was added to the residue, shaken in the end-over-end shaker for 10 minutes, centrifuges, and decanted the solution as before. This washing procedure repeated more two times, and finally, air-dried the residue.

In the second step, 40 ml of 0.5 M $NH_2OH.HCl$ (adjusted to pH 2, with 0.05 M HNO_3) was added to the dried residue from step-1, and repeat the procedure as step-1. The subsequent steps were run according to Table S8. The same procedure was followed for calcareous and vertisol soil (spiked and ageing for 50 days) to compare the effective Be fractionation at different steps.

Solution and solid-phase analysis

In order to investigate the mechanisms of Be desorption, and migration, a range of analytical techniques, including TOC-L analyser, XRD, XRF, SEM, FTIR were applied. The extracted solution in each fraction after passing through 0.45µm syringe filters, dissolved organic carbon (DOC) were analysed using TOC-L analyser (Shimadzu, Japan). The DOC originated from dissolved organic matter (DOM), which is very important for Be complexation and dissolution.

Observation of mineralogical changes of the soils after extractions of Be from each fraction was conducted using XRD analysis. Briefly, a small amount of each sample was ground to a fine powder, then randomly oriented powder samples were prepared using a back-loading method followed by palletisation at high pressure. The XRD was run with a Cu Kα radiation at 45kV, 40 mA and scanned in 2θ angle range from 5° to 80° with steps of 0.026° per second. The elemental compositions of the solid phase of every fraction were also determined by x-ray fluorescence (XRF). To investigate the changes of surface morphology, the fractions of solid phases were analysed by a scanning electron microscope (SEM, Zeiss Sigma VP FESEM, Germany) fitted with a backscattered electron detector.

The dried and powdered soil samples after desorption of Be, were analysed using FTIR to explore any changes of Be-O/Be-OH bonds or any other functionality after the experiments. Thin pelleted samples were prepared by mixing with spectroscopic quality of KBr (1 mg: 100 mg KBr) as a carrier for the IR scans and a translucent sheet obtained with a mechanical presser. The spectra were counted from 500 to 4000 cm^{-1} at each of 4 cm^{-1} intervals. The background absorbance was measured using pure KBr pellets [12] .

Implications for environmental risk of Be

The Enrichment factor (EF) is usually used to express the metal contamination and toxicity caused by natural as well as anthropogenic sources [13-15] using the following equation (9). The baseline metals are usually considered Fe or Al but Al is more relevant in the case of Be due to having similar electrochemical properties [14, 16].

$$EF = \frac{(Be/Al)_{Ti}}{(Be/Al)_{Bi}}, \quad Where,\, i = 1,\, 2,3\ldots\ldots etc; \ldots\ldots\ldots\ldots\ldots\ldots\ldots(9)$$

Where $(Be/Al)_{Ti}$ and $(Be/Al)_{Bi}$ are the ratios of Be to Al in different trench and background soils, respectively. $EF < 1$, no enrichment; $1 < EF < 3$, low enrichment; $3 < EF < 5$, moderate enrichment; $EF > 5$, moderately severe to severe enrichment [14].

The toxicity/pollution assessment was carried out using all batch leaching data according to the following literature [17].

$$Polllution\ index = \frac{C_{ext}}{C_{tox}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(10)$$

$$P_i = \sqrt{\frac{{(C_{ext}/C_{tox})_{max}}^2 + {(C_{ext}/C_{tox})_{avr}}^2}{2}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots(11)$$

Where, P_i, C_{ext} (µg/L), C_{tox} (µg/L) are the pollution index, concentration of Be in the extractant, and maximum permissible concentration for toxicity. RIVM [18] reported the maximum pernisible concentration for ecological risk indication value was 0.13 µg/L, which was used in this study for C_{tox}. $Pi \leq 1.0$, $1.0 < Pi \leq 2.0$, $2.0 < Pi \leq 3.0$, $Pi > 3.0$ represent no contamination, low contamination, moderate contamination, and serious contamination, respectively [17].

Coefficient of potential ecological risk (E_r) for sequential leaching was calculated using equation 12 following previous study [19-20].

$$E_r = T_r \times \frac{C_m}{C_s} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(12)$$

Where, C_m, and C_s are the concentration of Be in the mobile (F1+F2+F3) and stable fraction (F4), respectively. Tr is the toxic risk factor of Be in the soil or sediments. The T_r value for Be is not widely reported in the literature. For this reason, we have used 3 mg/kg since RIVM [18] reported this value as for ecological risk concentration. $E_r<40$, $40\leq E_r<80$, and $80\leq E_r<160$ represents very low, moderate and high potential ecological risk [19-20].

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