



A comparative study for lead removal by pure calcite and a natural calcitic soil sample

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Abstract

The extent of removing toxic lead ions (Pb^{2+}) from aqueous solutions was investigated using pure calcite and natural calcitic soil, one of the globally available soils, as effective inorganic adsorbents. The rate of adsorption of Pb^{2+} ions on pure calcite is much faster than that on the soil sample due to the presence of other metal ions on its surface, which block the active sites and retard the adsorption process. The order of adsorption on the soil was a first-order reaction with respect to Pb^{2+} ions. The rate constant was calculated to be 0.1 min^{-1} . The half-life time of the adsorption of Pb^{2+} ions on soil was calculated to be 6.9 min at 25°C . The adsorption process on both solids fits well with the Langmuir isotherm but does not fit well with the Temkin or Freundlich isotherms. Maximum adsorption capacities were calculated from the Langmuir isotherm as 156 mg/g calcite and 74 mg/g soil at 25°C .

Keywords Heavy toxic element · Natural adsorbent · Aqueous solution · Adsorption · Kinetics

Introduction

The Palestinian territory is one of the regions with very scarce sources of water due to population growth rate and misuse of the water resources. In West Bank, water pollution has serious negative impacts on the health of the people, the economy, and the environment. Water resources are being threatened by four main sources: domestic wastewater, solid waste leachate, industrial effluents, and runoff from agricultural activities. Contamination of groundwater aquifers and springs as a result of wastewater percolation is a serious problem in several areas of West Bank. Pollution of water resources, such as springs and wells, has been identified in all districts.

One of the main sources of drinking water in the Palestinian authority region is harvested rainwater. Lead was found in all harvested water samples; its concentration ranged from 12.9 to 486.4 ppb (Malassa et al. 2014), which is much higher than the allowed World Health Organization (WHO) limit (10 ppb). At the same time, the groundwater had a much lower concentration of lead in most wells in the same region, except for some wells in dry seasons that reached a lead concentration of 109.3 ppb (Malassa et al. 2013). According to this data, we believe that lead is the most serious heavy metal contaminant in our region and soil is the main risk for it. The sources of lead metal might be attributed to uncontrolled burning (incineration) of solid wastes in illegal dumping sites (Malassa et al. 2014).

Lead ions can exchange calcium ions in calcite crystal, since the ionic radius is similar in both metal ions (1.14 \AA for Ca^{2+} compared to 1.33 \AA for Pb^{2+}) in solution (Shannon 1976). In addition, their charge-to-ionic radius ratios are also similar (1.79 for Ca^{2+} , 1.55 for Pb^{2+}), and both ions exhibit a face-centered cubic structure, resulting in the same coordination number (8) (Schwerdtfeger 2002). As a result, Pb^{2+} ions fit well in calcite crystal. The X-ray analysis done by Sturchio et al. (1997) showed that most adsorbed Pb^{2+} ions occupy Ca sites in the calcite lattice. This means that the adsorption is mainly chemisorption. Recently, Fiorito et al. (2022) suggested that both heterogeneous nucleation and

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surface co-precipitation of cerussite (PbCO_3) occur depending on the initial concentration of Pb^{2+} ions. The sorption capacity of Pb^{2+} ions on calcite increases linearly with increasing initial Pb^{2+} concentration up to a value of 1,680 (20) mg/g when the initial Pb^{2+} concentration is 80 mM (Fiorito et al. 2022).

Soil contamination with heavy metals is one of the most urgent environmental issues. Heavy metals are toxic inorganic substances, such as lead (Pb), chromium (Cr), arsenic (As), zinc (Zn), cadmium (Cd), copper (Cu), mercury (Hg), and nickel (Ni) (Wuana and Okieimen 2011). Heavy metals are harmful to humans and the biosphere in general. Heavy metal accumulation in the environment has emerged due to natural and anthropogenic activities (Weissmannová and Pavlovský, 2017). These activities include urban road construction, agriculture waste, sewage disposal, gases from automobile exhausts, industrial discharges, steel plants, oil lubricants, corrosion of building materials, car batteries, and incinerating solid waste containing heavy metals (Al-Jumaily 2016; Ubwa et al. 2013). Heavy metal accumulation in the soil will cause it to lose its function in crop cultivation. The increase in heavy metal concentration in the soil is due to its interaction with water and the atmosphere. Furthermore, dust deposition is the main reason for heavy metal accumulation in soil (Adriano 2001; Li et al. 2001; Sutherland and Tolosa 2000; Ubwa et al. 2013). In a previous study done by Sandler, he showed that more than 50% of soils in Palestine are derived from dust particle size, and the chemical composition of dust has a significant impact on the ecosystem and public health (Sandler 2013; Maring et al. 2003). Calcium carbonate (CaCO_3) is one of the most abundant and reactive minerals in the natural environment and is a major component of soil. It is a dominant component of marine sediments (Morse and Mackenzie 1990). Among all CaCO_3 polymorphs, calcite is the most abundant and thermodynamically stable and the least soluble (Plummer and Busenberg 1982). The crystalline structure of calcite is hexagonal, its point group is $3\ 2/m$, and its density is $2.71\ \text{g/cm}^3$ (Anthony et al. 2003). Calcium carbonate minerals are not pure (c.f. Garrels and Christ 1965) and contain a variety of trace elements, mostly alkaline-earth elements, like magnesium (Mg), strontium (Sr), and barium (Ba), but also other elements like lithium (Li), boron (B), cadmium (Cd), uranium (U), thorium (Th), and others (e.g., Ortega et al. 2005). The enrichment of these trace elements in biogenic and inorganic CaCO_3 , relative to Ca, reflects certain environmental conditions (temperature, growth rate, pH, salinity, and the composition of seawater) at their

time of formation (e.g., Morse and Bender 1990). Carbonate minerals play a major contribution to the global carbon budget (Morse and Mackenzie 1990); they play an important role in adsorption and desorption processes in environmental systems (e.g., Langmuir 1997). Calcite is a good adsorbent for heavy metals, such as Cd^{2+} and Pb^{2+} . Many studies have been conducted in this regard (Yavuz et al. 2007; Cave et al. 2005; Chen et al. 2008). This study compares the ability of calcite and soil from the Al-Quds University area to remove lead ions from polluted water.

To the best of our knowledge, this is the first report using calcite and natural calcite for studying Pb^{2+} adsorption behavior from aqueous solutions in our region, although many researchers have worked on the adsorption of various heavy metals using calcite as the adsorbent in Latin America, Asia, and Europe.

In this paper, systematic laboratory investigations on the capacity and kinetics of Pb^{2+} adsorption from an aqueous solution on both calcite and local natural calcite from the Al-Quds University area have been reported. All of the experiments were carried out at the Al-Quds University labs in 2020.

Materials and methods

Chemicals and reagents

Calcite is (Roth, $\text{CaCO}_3 > 99\%$, Art.-Nr.P012.2) of surface area $0.5\ \text{m}^2/\text{g}$, determined by both BET and SEM, respectively (Alkhatib and Eisenhauer 2017a, b, II). All the chemicals are ACS grade of Merck, and all solutions were prepared using deionized water ($18.2\ \text{M}\Omega$). Nitric acid and hydrofluoric acids are ultrapure. The soil sample is surface soil from the field of Al-Quds University.

Preparation of soil sample

The soil sample was dried in an oven at $50\ ^\circ\text{C}$ for 48 h until its weight became constant. It was sieved through a 2 mm sieve. Complete soil analysis was carried out, including: pH, total organic carbon (TOC), chemical composition and grain size determination.

Grain size

Organic carbon was removed from 3.0 g soil sample by stirring it with 100 ml of 32% H_2O_2 aqueous solution at $60\ ^\circ\text{C}$

for 30 min, and the suspension then was sonicated for about 10 min. Aqueous solution was decanted and washing with 32% H₂O₂ was repeated three times. The sample then was washed with pure water and dried. The sample then was analyzed by AS-2011 Laser Particle Size Analyzer, with a laser beam ranging from 100 nm to 2 mm. Based on Fraunhofer diffraction theory, the particle size of the soil sample is directly proportional with the extent of light scattered (Mudroch 1997).

pH and water saturation

The pH of soil sample was measured by mixing equal masses of soil with pure water. After stirring the suspension for about 10 minutes, pH was measured using the WTW 2F40110 pH meter at room temperature.

To a 30.0 g of soil sample in a 250 ml conical flask, pure water was added gradually with stirring until the surface of the soil became shiny. The surface of the sample was kept shiny for one hour by adding small amounts of water as if it was needed. The flask was weighed and water needed for saturation was calculated.

Total organic carbon (TOC)

Organic carbon in the soil sample was determined via Walkley–Black chromic acid (K₂Cr₂O₇/H₂SO₄) oxidation method. 1.00 g soil was oxidized with 75.00 ml (excess amount) 1.00 M chromic acid solution while heating. The excess (remained unreacted) chromic acid was titrated with freshly prepared 0.5 M standardized FeSO₄ aqueous solution using diphenylamine as an indicator. Blank titration was carried out. The %organic C in the soil sample was calculated using the following equation:

$$\text{Organic C, \%} = ((V_{\text{blank}} - V_{\text{sample}}) \times M(\text{Fe}^{2+}) \times 0.003 \times 100 \times f^* \text{mcf}) / W.$$

where:

V_{blank} = volume of titrant in blank, mL

V_{sample} = volume of titrant in sample, mL

$M_{\text{Fe}^{2+}}$ = concentration of standardized FeSO₄ solution, molarity (M)

0.003 is carbon oxidized as in the following equation,

$$0.003 = \frac{12 \text{ g C}}{\text{mole}} \times \frac{1 \text{ mole K}_2\text{Cr}_2\text{O}_7}{6 \text{ mole FeSO}_4} \times \frac{3 \text{ mole C}}{2 \text{ mole K}_2\text{Cr}_2\text{O}_7} \times \frac{1 \text{ L}}{1000 \text{ ml}}$$

f = correction factor, 1.3

W = weight of soil, g

mcf = Moisture correction factor (ASTM D2974-00 2000).

Mineralogy

A homogenized ground soil sample was scanned by a PAN-ALYTICAL X'Pert³ Powder XRD- diffractometer equipped with a pixel detector. Scanning range: 3–70° 2 θ with a step size of 0.013° and 70.1 s per step speed.

Kinetics of adsorption

Two grams of soil sample and separately 2.0 g of calcite were taken, every 200.0 ml of 48.0 ppm of Pb²⁺ ions was added. The suspensions were stirred (2,500 rpm) for 15 min at 25 °C, and 3.0 ml samples were withdrawn at certain intervals of time after mixing. Each sample was filtered and diluted 1:1 with ultrapure nitric acid. Then, the concentration of Pb²⁺ ions was measured by ICP-MS. To validate the analysis of metal ion concentration for accuracy and precision, two concentrations of lead ions (125 and 250 ppb), which were used for the calibration curve, were also measured five times as samples during the run analysis. The standard deviation for both concentrations was ± 1.45 .

Determination of the extent of Pb²⁺ adsorption on the soil sample and calcite

Different 1.0 g samples of soil and calcite were taken. To each sample, 100.0 ml of Pb²⁺ ions of different concentrations (between 200 and 2500 ppm) were added and stirred for 2 h (2500 rpm). A 2 ml sample of each suspension was withdrawn and filtered with a Millipore filter (0.22 μm). Precisely 1.0 ml of 2% ultrapure nitric acid was added to every 1.0 ml of filtered solution. The concentration of Pb²⁺ ions was then measured using ICP-MS.

Elemental analysis

Exactly 1.0 g of the soil sample was dissolved in a Mars5 digester using 60 ml ultrapure nitric acid with 30 ml ultrapure hydrofluoric acid. After the digestion was completed, the total volume of solution increased with water until reaching 100 ml in a volumetric flask. Exactly 0.10 ml of the solution was then diluted to 100 ml. The solid stock concentration of the sample to be analyzed was 10 ppm. The measured element concentrations were calculated relative to this value. The elemental ratio was measured by inductively coupled plasma mass spectrometry (ICP-MS-QP Agilent 7500cx), using indium (In) as an internal standard for quality



control and accuracy, combined with a multi-standard calibration method. The main elements were measured in ppm, while trace and rare elements were measured in a separated run in the ppb concentration range. Each sample was analyzed at least three times. Coral standard JCP-1 was used as reference material and measured after every fifth sample, which equated to 10 times during this study ($N = 10$). The JCP-1 Sr/Ca and Mg/Ca ratios were calculated to be 8.82 ± 0.03 and 4.22 ± 0.04 mmol/mol, respectively, which fall within the statistical uncertainty of the reported values (8.84 ± 0.09 mmol/mol for Sr/Ca and 4.2 ± 0.1 mmol/mol for Mg/Ca) (Hathorne et al. 2013).

Results and discussion

Soil characteristics

The soil sample is Rendzina, and its color is 7.5YR6/4. Its pH is 8.11. Water absorption is 0.62 g water per g soil. The percentage of total organic carbon was 0.5%. The grain size of the soil sample was composed of the following: 23.2% clay, 62.4% silt, and 14.4% sand. The mineralogy of the soil sample is 5% quartz, 15% clay, 80% calcite, and less than 1% dolomite and hematite, indicating the sample is mostly calcite.

Elemental analysis of the soil sample

Major elements in the soil sample were measured by ICP-MS and calculated as metal oxide percentage, as shown in Table 1. This data also confirms that the major component

Table 1 Calculated metal oxides in the soil sample

Metal oxide	Mass%
SiO ₂	10.4
Al ₂ O ₃	3.5
Fe ₂ O ₃	1.6
TiO ₂	0.2
CaO	46.0
MgO	0.6
Na ₂ O	0.1
K ₂ O	0.2
P ₂ O ₅	0.3

Table 2 Trace element concentrations in the soil sample in ppm

Ba	Co	Cr	Cu	Mn	Mo	Ni	Pb	Rb	Sb	Sr	Th	U	V	Zn	Zr
108.0	5.0	102.0	21.0	134.0	1.5	26.0	4.0	11.0	0.4	342.0	2.3	2.3	47.0	59.0	21.0

Table 3 Kinetic study of Pb²⁺ removal by 2.0 g of soil and calcite when each solid sample was stirred for 15 min with 200 ml of 48 ppm Pb²⁺ ions

Time (min)	Soil		Calcite	
	[Pb ²⁺] (ppb)	± RSD	[Pb ²⁺] (ppb)	± RSD
0	47,851	1.65	47,851	1.65
5	22,417	0.50	n.d	n.d
8	15,012	2.05	n.d	n.d
12	9581	1.84	n.d	n.d
15	7604	1.90	n.d	n.d

n.d.: not detected (below the detection limit of the instrument during three replications)

of this soil sample is calcite. The concentration of trace elements (in ppm relative to the mass of the solid samples) is reported in Table 2. As shown in Table 2, the concentration of Pb²⁺ ions in the solid sample is 4.0 µg/g soil. The concentration of Cr ions is relatively high (102 µg/g soil) due to the soil contamination by the deposited dust. The concentration of Sr²⁺ ions in the soil is also high (342 µg/g soil); this is because the major component of the soil is calcite, and Sr²⁺ ions coprecipitate with Ca²⁺ ions during the precipitation of calcite (Alkhatib and Eisenhauer 2017a, b, I).

Kinetic study for Pb²⁺ ion adsorption by the soil sample and calcite

As shown in Table 3, the adsorption of Pb²⁺ ions by calcite was too fast for the kinetics of adsorption on calcite to be explored; however, the adsorption on soil samples (mainly calcite) can be studied. This can be explained by the idea that the presence of heavy metal ions adsorbed to the soil sample retards the adsorption process more than a pure calcite sample. These already existing metal ions present in the soil sample are blocking the active centers of calcite and slowing down the adsorption of Pb²⁺, as in the study of Cave et al. (2005), where Mn²⁺ slows down the adsorption Cr³⁺. The variation of Pb²⁺ ion concentration with time is shown in Fig. 1a, and it fits the polynomial function:

$$[\text{Pb}^{2+}] = 142.15t^2 - 4877.1t + 46,165 \quad (1)$$

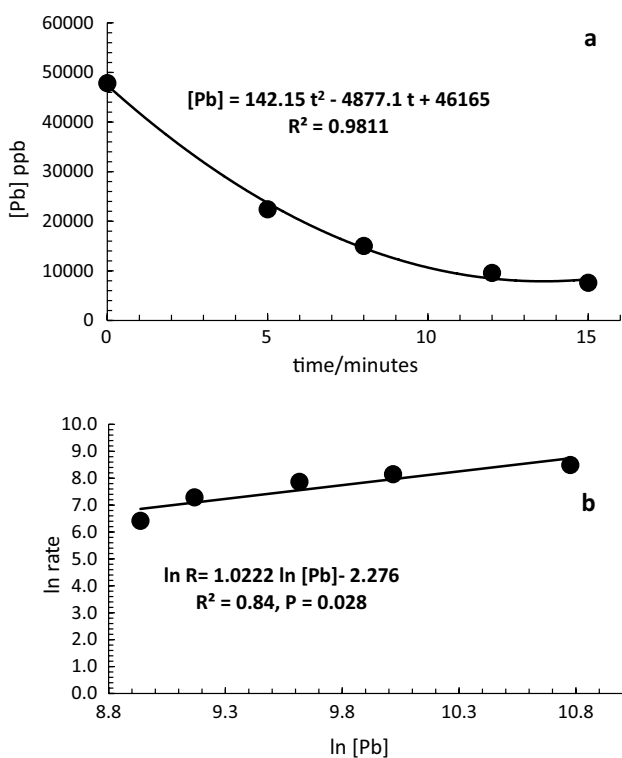


Fig. 1 The kinetics of adsorption of Pb²⁺ ions on the soil sample. **a** Changes of Pb²⁺ ion concentration as a function of time. The latter values fit a quadratic polynomial function. The instantaneous rate of reaction corresponds to the first derivative of the polynomial equation. **b** Plotting ln instantaneous rate as a function of ln [Pb²⁺]. The slope of this linear relationship is equal to the order of the adsorption reaction

The concentration of [Pb²⁺] is in ppb, and the time (*t*) is in minutes. Since the concentration of the solid material is almost constant during the whole adsorption process, the rate law can be written as:

$$\text{Rate } (R) = k' [\text{Pb}^{2+}]^X \tag{2}$$

where *k'* is the rate constant and *X* is the order of reaction with respect to the adsorbate ([Pb²⁺] ions). By taking the natural logarithm for both sides of Eq. 2, we get the following equation:

$$\ln R = X \ln [\text{Pb}^{2+}] + \ln k' \tag{3}$$

Plotting ln *R* on the y-axis versus ln [Pb²⁺] on the x-axis, as shown in Fig. 1b, will show a linear relationship; its slope is the order of reaction, which is 1 (first-order reaction). This result agrees with the principles of physical chemistry (e.g., Atkins and De Paula 2006). The rate constant was calculated from the slope to 0.1 min⁻¹. The half-life of the reaction can be estimated from the following equation:

$$t_{1/2} = \ln 2 / k' \tag{4}$$

Using Eq. 4, the half-life is estimated to be about 6.9 min. On the other hand, we can estimate it from the data points of Table 3 to be about 5 min.

Table 4 Data of adsorption on calcite and the soil sample: the initial [Pb²⁺] concentration (*C_i*) in ppm and in mM, the remaining concentration of [Pb²⁺] in aqueous solution after equilibrium (*C_e*), and moles of Pb²⁺ ions adsorbed per gram of adsorbent (*S*)

Initial [Pb ²⁺]		Calcite					Soil				
<i>C_i</i> (ppm)	<i>C_i</i> (mM)	<i>C_e</i> (ppm)	<i>C_e</i> (M)	ln <i>C_e</i>	<i>S</i> × 10 ⁵	ln <i>S</i>	<i>C_e</i> (ppm)	<i>C_e</i> (M)	ln <i>C_e</i>	<i>S</i> × 10 ⁵	ln <i>S</i>
1	2	3	4	5	6	7	8	9	10	11	12
217	1.04	6.75	3.26E-05	-10.33	10.12	-9.2	4.75	2.29E-05	-10.68	10.22	-9.19
436	2.11	4.5	2.17E-05	-10.74	20.84	-8.48	15	7.24E-05	-9.53	20.33	-8.5
663	3.2	4.25	2.05E-05	-10.79	31.77	-8.05	73.5	3.55E-04	-7.94	28.43	-8.17
1068	5.15	18	8.69E-05	-9.35	50.68	-7.59	386	1.86E-03	-6.29	32.92	-8.02
1565	7.55	14	6.76E-05	-9.6	74.86	-7.2	800	3.86E-03	-5.56	36.92	-7.9
2188	10.56	333	1.64E-03	-6.42	89.24	-7.02	1383	6.68E-03	-5.01	38.85	-7.85
2464	11.89	947	4.57E-03	-5.39	73.21	-7.22	1765	8.52E-03	-4.77	33.74	-7.99

Columns 1 and 2 are the initial concentration of [Pb²⁺] ion solutions. Columns 3, 4, 5, 6, and 7 show the data for calcite, and columns 8, 9, 10, 11, and 12 show the data for soil samples. *S* = (*C_i* - *C_e*) × *V*, where *V* is the volume of solution used and it equals 0.1 L (100 ml)

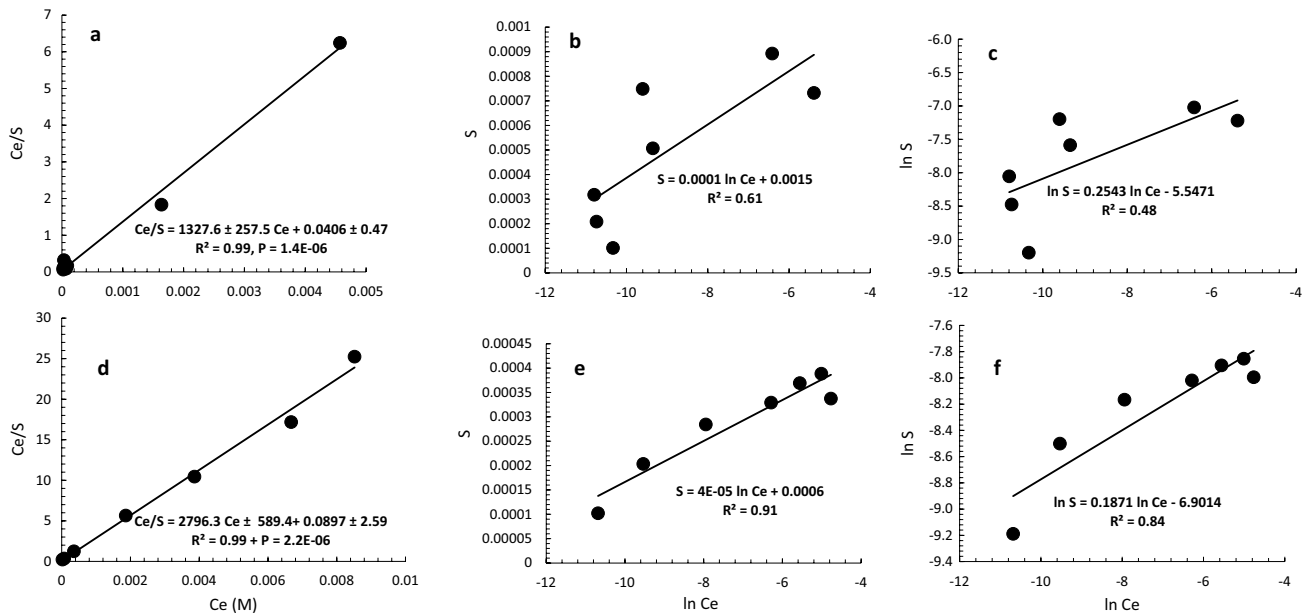


Fig. 2 Adsorption isotherms of Pb^{2+} ions on calcite (a is Langmuir, b is Temkin, and c is Freundlich) and on the soil sample (d is Langmuir, e is Temkin, and f is Freundlich). All isotherms are plotted according to Eqs. 5, 6, and 7 in the text

Pb^{2+} adsorption isotherms on calcite and the soil sample

Data of Pb^{2+} ion adsorption on calcite and the soil sample at 25 °C is shown in Table 4. It can be seen from Fig. 2a, d that adsorption of Pb^{2+} ions on calcite and soil fits well with the Langmuir isotherm (Eq. 5).

$$C_e/S = C_e/S_{\infty} + 1/(KS_{\infty}) \quad (5)$$

where C_e is the remaining concentration of Pb^{2+} ions (in M) in the aqueous solution after equilibrium, S is moles of Pb^{2+} ions adsorbed per 1.00 g adsorbent (pure calcite or solid sample), S_{∞} is the maximum moles of Pb^{2+} ions that can be adsorbed per 1.00 g adsorbent for complete monolayer coverage, and K is the thermodynamic equilibrium constant.

From Fig. 2a, the maximum moles of Pb^{2+} ions that can be adsorbed per 1.00 g calcite (S_{∞}) is 7.53×10^{-4} mol or 156 mg. The equilibrium constant (K) is $3.3 \times 10^4 M^{-1}$, which means that Pb^{2+} ion adsorption is much more favorable than desorption. From Fig. 2d, the maximum moles of Pb^{2+} ions that can be adsorbed per 1.00 g soil sample (S_{∞}) is 3.58×10^{-4} mol or 74 mg. For this, the equilibrium constant is $= 3.1 \times 10^4 M^{-1}$, which means that the surface of the soil sample of the same mass (1.0 gm) can't adsorb more than half the quantity of Pb^{2+} ions that

calcite can adsorb. This is due to the blocking of already existing metal ions on the active sites of the calcite surface. On the other hand, the thermodynamic equilibrium constant is almost equal for adsorption on calcite. It can be seen from Fig. 2b, c, e, f that neither Temkin (Eq. 6) nor Freundlich (Eq. 7) isotherms, respectively, fit the adsorption process of Pb^{2+} ions on calcite or the soil sample.

$$S = a \ln b + a \ln C_e \quad (6)$$

$$\ln S = \ln a + (1/b) \ln C_e \quad (7)$$

In Eqs. 6 and 7, a and b are constants.

Conclusion

This study aims to compare the extent of adsorption of Pb^{2+} ions between pure calcite and natural calcitic soil. Conclusions from the present study are as follows:

1. Calcite is the major mineral component in the soil sample in this study and is one of the major components in all other natural soils.
2. Pure calcite has a very high affinity to adsorb heavy metals, such as Pb^{2+} ions. Even though calcitic soil has a

significant relationship for adsorbing heavy metals, it is less than pure calcite due to the blocking of the active sites at the soil's surface and retardation of the already existing metal ions for the adsorption process.

- The maximum adsorption capacity of Pb^{2+} ions was calculated to be 156 mg/g calcite and 74 mg/g soil at 25 °C. These values may differ from other literature values due to the difference in the surface area of the adsorbents.
- The adsorption process of Pb^{2+} ions on calcite or calcitic soil is thermodynamically stable since the equilibrium constant is very high (more than $3.0 \times 10^4 M^{-1}$).
- Soil is the major sink for heavy metal contamination from dust or contaminated water.
- Environmental scientists have a great responsibility to devise easy ways to rid the soil of pollutants, especially heavy metals, to be exploited in agriculture.

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Declarations

Conflict of interest The authors declare no conflict of interest.

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