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Sorption of Mercury by Palygorskite Clay Mineral

A.Sh. Sallam*, M. S. Al - Sewailem and A. S. Al-Farraj
Soil Science Department, Collage of Food and Agriculture Science, King Saud University, Saudi Arabia.

PALYGORSKITE clay mineral is considered one of the dominated clay minerals in clay fraction in many soils and sediments in Arid Regions. This study aimed to determine sorption of mercury by palygorskite clay mineral. Batch sorption and leaching experiments in columns were conducted using all or some of five samples: 1) Sandy loam soil sample dominated with palygorskite, 2) the clay fraction that was separated from sample one (clay) , 3) acid washed sand, 4) and 5) the separated clay mixed with the washed sand sample at 2 level; (clay 2%) and (clay 4%), respectively. Results showed that Langmuir equation was applicable to all samples of mercury adsorption data, where the values of C/(x/m) increased with the increased in equilibrium solution concentrations. The adsorption maximum (b) and binding strength (k) values were very high for mercury in the separated clay sample compared to other samples. Adsorption maximum of the samples can be arranged a follows clay> clay 4%> clay 2% > soil. While binding strength takes the following order: clay > soil> clay 4%> clay 2%. Results also showed that Freundlich equation (to some extent) describe mercury adsorption. Results of columns experiment indicated that the highest amount of adsorbed mercury was found in the 4% clay treatment. The results suggest that palygorskite have high adsorption capacity for mercury and it act as sink for added Hg, and it can be used as a trap for holding Hg in the polluted soil.

Keywards: Palygorskite, Mercury retention, Heavy metals, Contaminated water and soils.

There are several problems dealing with heavy metals contaminated soils which effect human health and environmental quality. The mobility of heavy metals in soils is quite different and there is a risk of leaching them into ground water and/or accumulation in crops and/or human diet. The anthropogenic sources of heavy metals in soils are either primary sources, *i.e.,* heavy metals are added to the soil as an outcome of working the soil, or secondary sources where heavy metals are added to the soil as a consequence of a nearby activity, such as smelting or aerosol deposition, (Ferguson, 1990 and Nigam *et al.,* 2001). The presence of high levels of these metals in the environment may cause long-term health risks to humans and ecosystems. It is therefore mandatory that their levels in drinking water, wastewater, and water used for agricultural and recreational purposes must be reduced to within the maximum allowable concentrations recommended by national and international health authorities such as the World Health Organization (WHO, 2004).

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*E-mail of the corresponding author: asallam@ksu.edu.sa

Mercury is a naturally-occurring metal, traces of which occur in rocks of the earth's crust. Long-range transport of mercury through the atmosphere consists primarily of mercury in the elemental form (Mitra, 1986). In soils and surface waters, mercury predominantly exists in the mercuric (Hg) and mercurous (Hg^+) states, as ions with varying solubility. Zhao *et al.* (2009) suggest that chemisorption processes could be the rate-limiting steps in the process of Hg adsorption onto Polyacrylamide/attapulgite. Al-Farraj and Al-Wabel (2007a and b) found that the mining activities at Mahad AD'Dahab area in Saudi Arabia (gold mine) were positively contributed to the soil and plants pollution with heavy metals including mercury.

Clay minerals in soils and sediments were considered one of the key components controlling soil chemical reactions and heavy metal behavior in soil and waste water (Al-Jlil and Alsewailem, 2009, Falatah *et al.*, 2005 and Calabi Floody *et al.*, 2009). Nowadays palygorskite is receiving a great deal of attention due to its special layer-chain structure and low price. In Saudi Arabia, certain lagoon or lacustrine environments generated deposits of palygorskite clay (Aruma and Dammam formations) Laurent (1993), n the eastern region soils (Lee *et al.*, 1983) and Al-Kharj governorate (Sheta *et al.*, 2006). Palygorskite exhibited sorption properties towards Cd, the sorption process appearing to be relatively fast with equilibrium being attained within the first half an hour of interaction. The maximum removal capacity was 4.54 mg g^{-1} as determined by the Langmuir model (Álvarez-Ayuso and García-Sánchez, 2007). Zhang *et al.* (2009) describes the uses of a novel palygorskite; a type of magnesium aluminium silicate clay possessing a high specific surface area and pore surface activity, as a low cost and highly efficient adsorbent for sulphur dioxide (SO_2) removal.

This research was undertaken to study the ability of using palygorskite for limiting mercury pollution for soil and water. Therefore, laboratory (adsorption and desorption) and columns experiments were carried out.

Material and Methods

Collecting and preparing the samples

Two soil samples have been collected: Sandy surface sample (Quartzipsamment) from Thimamah (Saudi Arabia). Surface sandy loam soil sample (Torrifluvents) dominated by palygorskite clay mineral from Haradh (Saudi Arabia) was selected (Al-Harby, 2005). Collected soil samples were prepared for physical, chemical and mineralogical analyses following the standard procedures. Particle size distribution was carried out according to Gee and Bauder (1996), soluble salts and pH were measured as mentioned in Rhoades (1996) and Thomas (1996), respectively. Calcium carbonate content was determined according to Loeooert and Suarez (1996). Organic matter content was determined according to Nelson and Sommers (1996). CEC and surface area were determined according to Sumner and Miller (1996) and Carter *et al.* (1986), respectively. Clay fraction (< 2 μm) was separated according to (Jackson, 1974), X-ray diffractograms were obtained using Cu-Kα radiation at 40kV and 25mA.

Identification of clay minerals was carried out according to (Dixon and Weed, 1989). Free iron and manganese oxides (Fe_d & Mn_d) were extracted using sodium citrate bicarbonate- dithionate method (CBD) according to (Mehra and Jackson, 1960). Amorphous silica and alumina (Si_{am} & Al_{am}) according to Alexiades and Jackson (1966) then Al and Si in the extracts were measured by AAS (Perkan Elmer AAnalyst 300). The main characteristics (physical, chemical and mineralogical) of the original soil, sandy soil and separated clay samples are listed in Table 1 and 2.

Sandy sample was prepared by washing it several times with HCl (0.1N) to remove carbonates, then salts and other soluble constituents were washed with distilled water. Clay was separated from soil sample according to Jackson (1974). Separated clay was mixed with washed sand at 2 and 4%.

TABLE 1. Some physical, chemical, analysis for Torrifluvents soil, Clay (separated from the soil) and Sandy soil .

Sample	Particle size distribution (%)			Texture class	EC dS m⁻¹	pH	CaCO³	O.M.	CEC Cmol Kg⁻¹	Surface area m²g⁻¹	Fe_d	Mn_d	Al_am	Si_am
	Clay	Silt	Sand				gKg⁻¹				mg Kg⁻¹			
Torrifluvents soil	9.4	6.9	83.7	LS	2.6	7.6	151.6	10.9	14.3	93	1.1	0.1	1.4	8.6
Clay (separated from the above soil)	100	-	-	C	-	-	-	-	62.7	216.6	-	-	-	-
Quartzipsamment soil	1.2	2.1	96.7	S	-	-	82.8		1.9	6.6	-	-	-	-

TABLE 2. Semi quantitative analysis of X-ray diffractograms obtained for clay fraction separated from soil samples.

Smectite	Kaolinit	Vermiculite	Chlorite	illite	Palygorskite	Interstratified minerals	Accessory minerals		
							Quartz	Feldspar	Calcite
+	+	T	T	S	+++	S	+	T	T

(++++) Dominant, (+++) High, (++)Moderate, (+) Few, (S) very few, (T) Traces

Mercury sorption experiment

Original soil, washed sand, separated clay and mixed 2 and 4% samples were used in the batch sorption experiment. Duplicate 1.0 gm of the prepared sorbent samples were placed in centrifuge tubes (50 ml) and equilibrated with 40 ml solutions of 1, 5, 10, 20, 40, 80 and 100 ug Mercury ml⁻¹ prepared from analytical grade $Hg(NO)_3$ at ambient temperature (20-22°C). The experiments were done using a background electrolyte solutions of $Ca(NO_3)_2$ 0.0114 N̲. Preliminary studies indicated that 24 hr was found to be sufficient to achieve equilibrium conditions when using flour–shaker for agitation. After shaking and centrifugation at 2500 rpm for 15 min, the supernatant solutions were measured for mercury using ICP (Inductively Coupled Plasma Emission Spectroscopy) ICP-AES (Perkin elemer, 4300 DV). The difference between the amount of mercury added initially and that in solution after equilibration was taken as the amount of sorbed mercury by the sample. Desorption of the sorbed mercury was carried out using 40 ml of 1 N̲ NH₄OAC after washings the samples with ethanol

two times to remove the remaining soluble form. After NH_4OAC addition, the suspensions were shacked for two hours, followed by centrifugation and determination of mercury in the supernatant solutions using ICP.

Columns experiment

Soil and mixture of 2 and 4% samples were used in the columns experiments. Forty PVC transparent columns with an ID of 6 cm and a length of 30 cm have been used. The bottom of column was tightly sealed with silicone adhesive. A glass tube with 5 mm diameter attached to collect the leachate. The columns were carefully hand-packed with air-dried sample (soil, clay mixed with washed sand at rate of 2 and 4%) to the desired bulk density (1.60 Mg m^{-3}) by gentle tapping.

The columns were filled to a depth of 10.0 cm. The sample columns were saturated from the bottom with Di-water for one day and then left to drain the excess water for another day. Fifty ml of $Hg(NO_3)_2$ solutions in concentrations of, 10.0 and 100 mg l^{-1} were added at zero, 1, 2, 3 and 4 days. The leachates were collected from each column at each required time, and Hg concentrations were measured using the ICP-AES. At the end of the experiment, the samples in each column were sectioned at increment of 2.5 cm (4 sections). Mercury was extracted from each sections by NH_4HCO_3 -DTPA and measured by the ICP-AES.

Quality control

The used chemicals were purchased from E. Merck (AR, 99.9%). The apparatus, tubes and vessels were previously washed with 30% HNO_3, then rinsed three times with deionized water and dried in an oven. All stages of sample preparation, analysis and measuring were carried out in a clean environment.

Results and Discussion

Results showed clear differences between the samples used in their physical and chemical properties, especially in cationic exchange capacity and specific surface area (Table 1). Semi quantitative mineralogical characterization of the separated clay (Table 2) revealed that the clay was dominated with palygorskite and few kaolinite and smectite, while other clay minerals and non-clay mineral (quartz, feldspar and calcite) were present in traces quantities.

Sorption isotherms of sorbed Hg and the equilibrium Hg concentration were presented in Fig. 1, the pattern of the isotherm were quit similar with a slight difference between soil and clay. The isotherm had a relatively high slope and follows the L-shaped type. Results indicated that Hg sorption followed Langmuir adsorption isotherm (Table 3 and 4 and Fig. 2), where the values of C/(x/m) increased with the increased in equilibrium solution concentrations. The empirical constants of Langmuir eq. b and k were related to the adsorption maximum and binding strength, respectively (Table 4). The adsorption maximum (b) and binding strength (k) values were very high for mercury in the clay sample (dominant with palygorskite clay mineral) compared to other studied samples. Maximum adsorption of the studied samples can be arranged as follow clay> clay

4%> clay 2% > soil and those differences in b values might be related to the differences in the CEC and surface area. In this regard Gatima *et al.* (2006) found that adsorption of heavy metal in contaminated soils was depended upon the CEC. Binding strength (k) in the studied samples takes the following order: clay > soil > clay 4%> clay 2%. The high value of (k) for Hg indicates the ability of clay dominated with palygorskite for Hg sorption. Fig. 3 showed the ability of Freundlich adsorption equation to describe mercury adsorption on the studied samples. Moreover, it indicates the conformity of mercury adsorption on the clay dominated with palygorskite as shown by Langmuir equation.
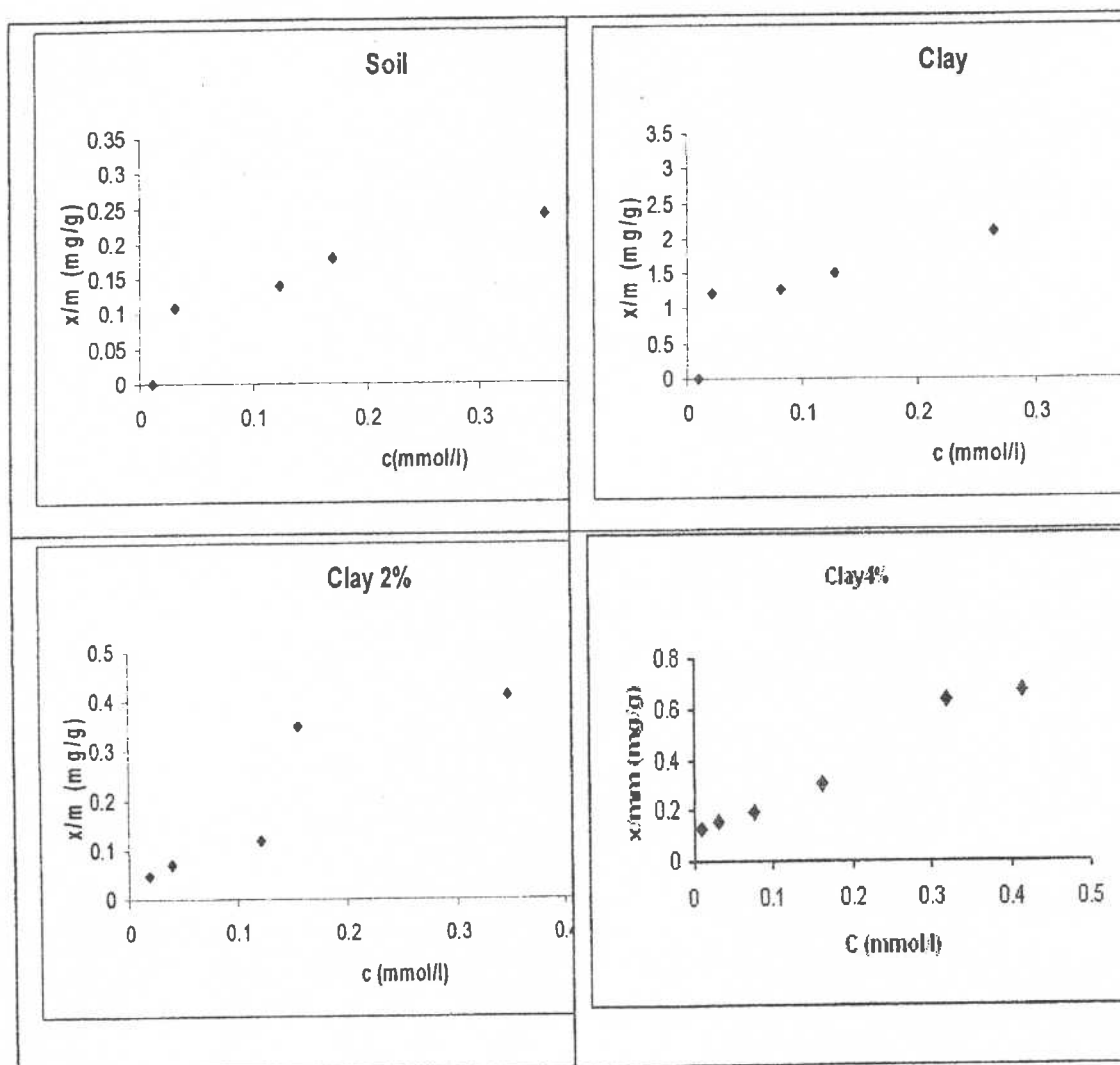

Fig. 1. Relationship between adsorbed Hg by used samples and solution equilibrium concentration.

Figure 4 showed the relationship between the amounts of added mercury and that adsorbed in the used samples. Results showed that the values of correlation coefficient for the used samples can be arranged as following: soil, clay dominant by palygorskite and clay mixed with washed sand at 2 and 4% and they ranged between 0.494 and 0.653. Moreover, the amount adsorbed increased at the high application rates.

TABLE 3. Adsorption and desorption of Hg by the used samples .

Sample	(mg l⁻¹)	C (mg l⁻¹)	x/m (mg g⁻¹)	Adsorbed Hg (%)	Desorbed (mg g⁻¹)
Soil	5	2.27	0.00	54.59	0.04
	10	6.51	0.11	34.92	0.13
	20	15.60	0.14	22.02	0.36
	40	34.12	0.18	14.70	0.82
	80	71.94	0.24	10.07	1.89
	100	91.89	0.32	8.12	2.50
Clay fraction	5	1.93	0.00	61.31	0.03
	10	6.83	1.23	31.73	0.16
	20	16.23	1.27	18.88	0.47
	40	34.73	1.51	13.18	1.14
	80	71.88	2.11	10.15	2.37
	100	91.81	3.25	8.20	3.07
Sand and 2% Clay	5	3.70	0.05	26.08	0.06
	10	8.19	0.07	18.12	0.18
	20	17.03	0.12	14.86	0.43
	40	31.15	0.35	22.12	0.86
	80	69.67	0.41	12.91	1.83
	100	89.38	0.42	10.62	2.42
Sand and 4% Clay	5	1.80	0.13	64.07	0.01
	10	6.16	0.15	38.40	0.12
	20	15.18	0.19	24.13	0.38
	40	32.33	0.31	19.18	0.91
	80	64.13	0.63	19.84	1.81
	100	83.13	0.68	16.88	2.46

TABLE 4. The maximum adsorption capacity and binding energy for the adsorbed Hg in the used samples according to Langmuir equation.

Sample	b	K	R^2*
Soil	0.382643	12.12715	0.9868
Clay fraction	3.757986	12.85507	0.9483
Sand and clay 2%	0.631951	4.785002	0.6696
Sand and clay 4%	0.955657	4.887436	0.748

b: Maximum adsorption capacity, k: Binding energy.
*In different concentration R^2 was calculated based on 12 point obtained from 6 concentrations of applied Hg^{+2} in two replicates .
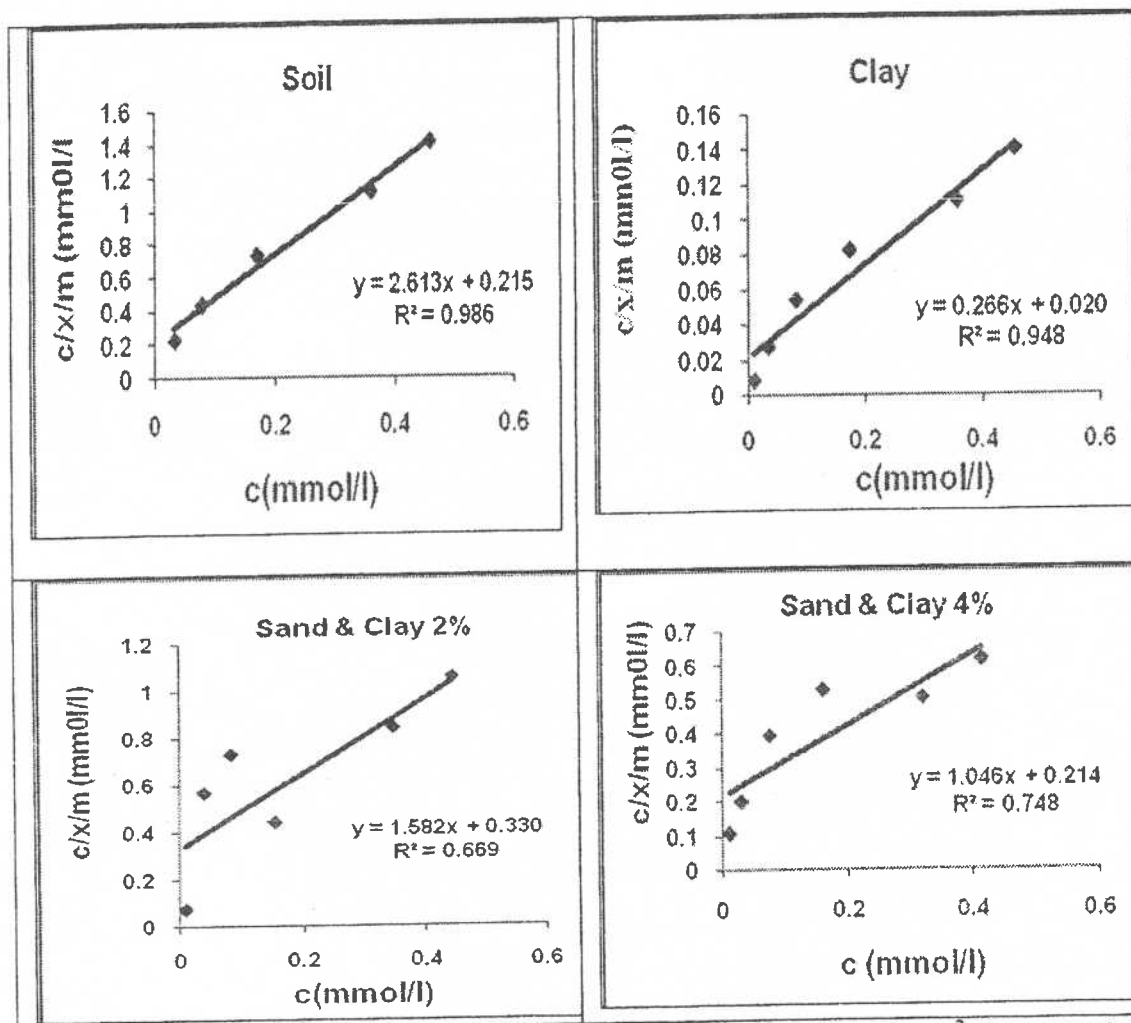
Fig. 2. Langmuir adsorption isotherm of Hg in the used samples (R^2 was calculated based on 12 point obtained from 6 concentrations of applied Hg^{+2} in two replicates).

The results (Table 3) showed the mean extracted quantities of mercury (mg g^{-1}) in two successive extraction following the Hg adsorption experiment. Generally, the amount of the extracted Hg in the first extraction was higher than the second one for all used samples. It also noted that the quantity of extracted Hg increased with increasing the adsorbed Hg. Generally, the highest amount of extracted Hg can be arrange as following: Clay > soil > 4% clay > 2% clay at most of the added Hg (20, 40, 80 and 100 mg l^{-1}).

It can be said that the studied samples differ in their ability for mercury retention, and in the forces of the different sites on the surfaces of these samples to adsorb mercury.
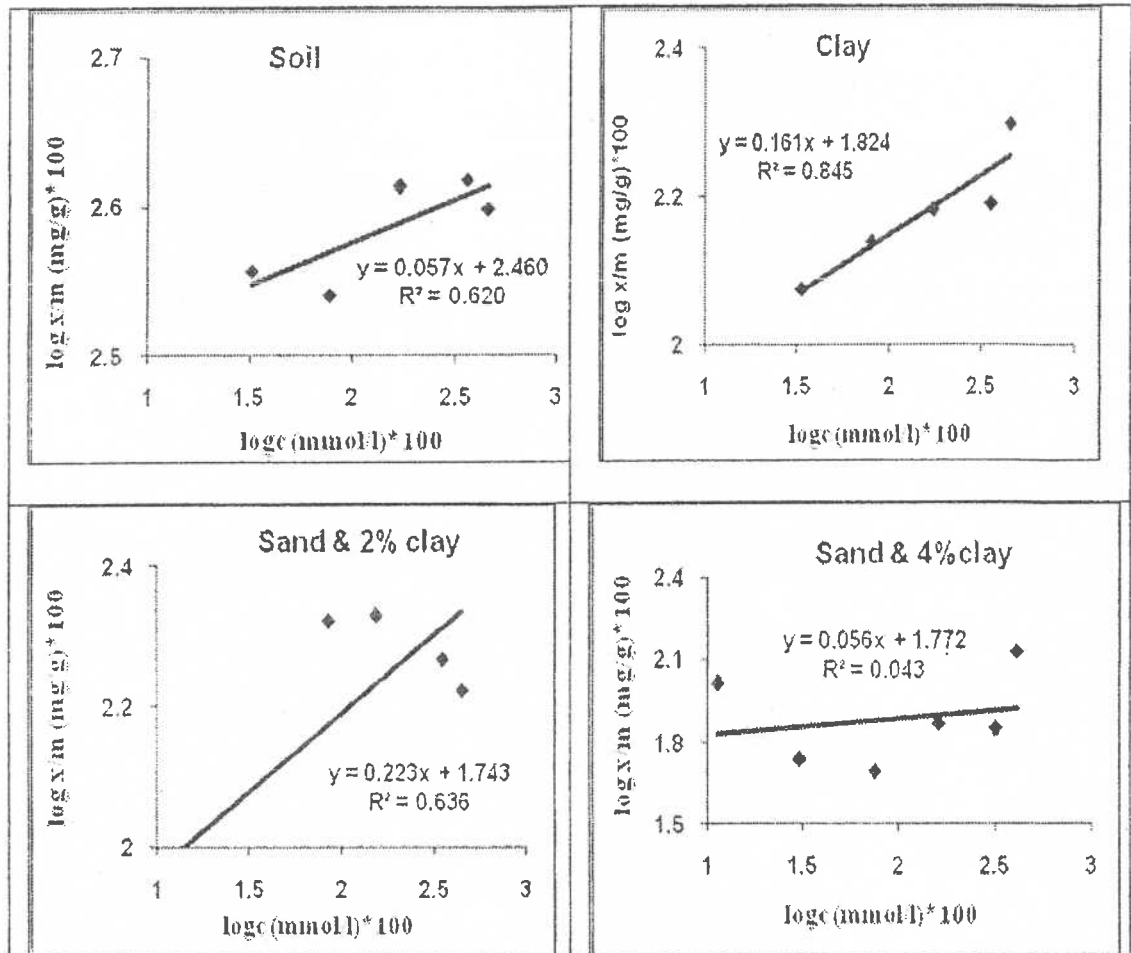
Fig. 3. Frendlich adsorption isotherm of Hg in the used samples (R^2 was calculated based on 12 point obtained from 6 concentrations of applied Hg^{+2} in two replicates).
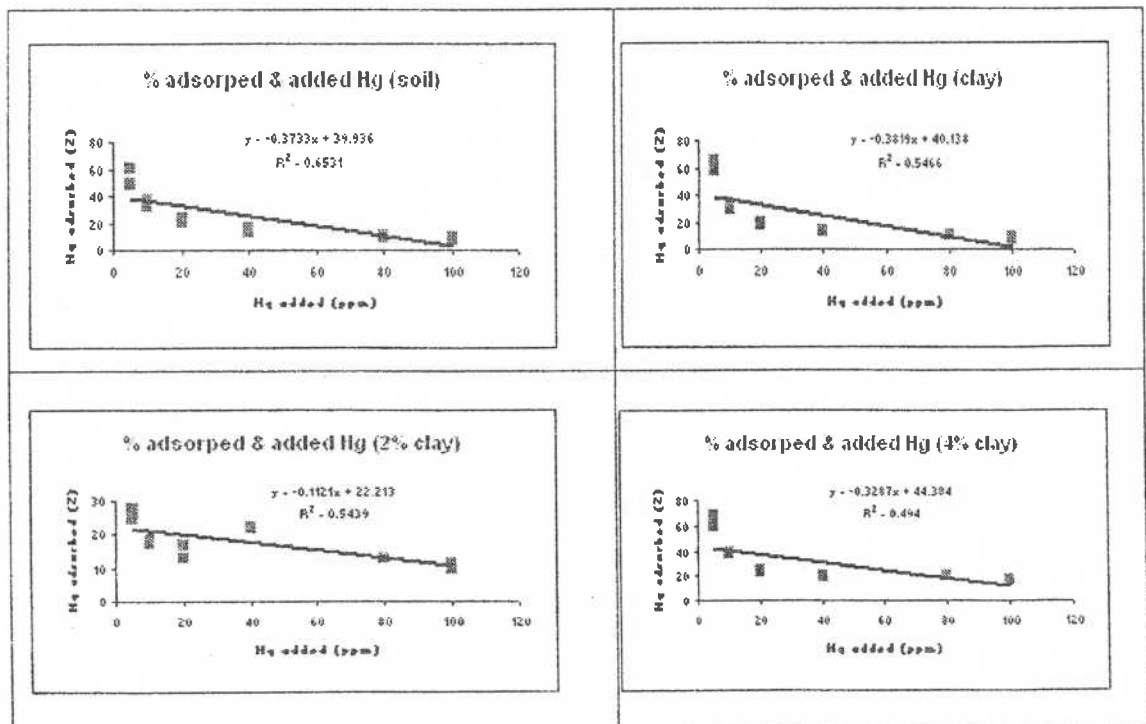

Fig. 4. The relationship between the amount added and adsorbed of mercury in different used samples.

Results of columns experiment of the studied samples are shown in Table 5 and Fig. 5 a and b. In general, data indicated that increasing the concentration of added mercury led to more sorption with the studied samples; and to a more amount of mercury extracted by NH_4HCO_3 - DTPA solution. The highest amount of extracted mercury from the whole column was in the 4% clay mixed treatment followed by the soil dominated with palygorskite then the 2% clay mixed treatment at both concentration (10 and 100 mg l^{-1}) of added Hg. Therefore, palygorskite act as a sink for added Hg and it can be used as a trap for holding Hg in the polluted water and soil. The soil hold more Hg than the 2% clay mixed treatment because the soil is dominated with palygorskite (Table 2) and hence the quantity of palygorskite to be used for controlling Hg or other heavy metals should be taken into consideration. Generally, the results suggested that palygorskite clay mineral have high potential for mercury retention.
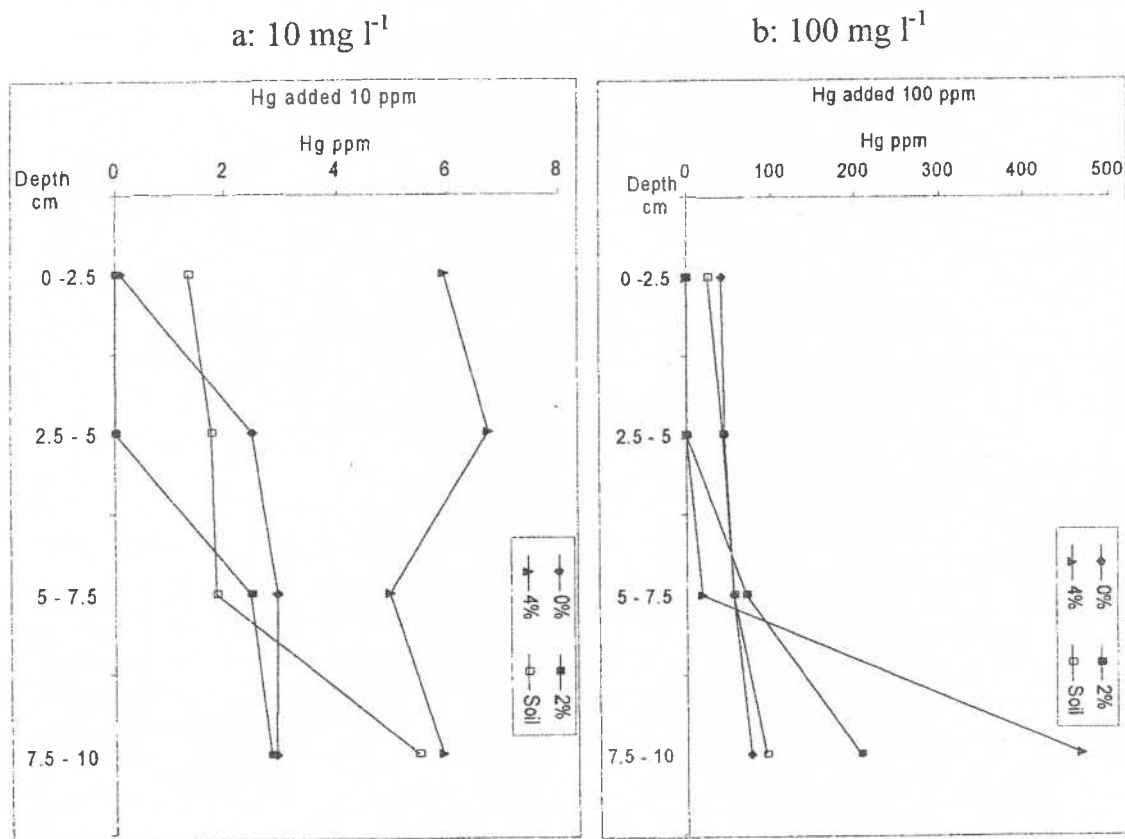
a: 10 mg l^{-1} b: 100 mg l^{-1}

Fig. 5 (a and b). The distribution of extracted mercury in the different sections of used columns at the used concentration (10 and 100 mg /l).

TABLE 5. Total quantity of extracted Hg from the whole columns with using of 10 and 100 mg l^{-1} concentrations.

Hg concentrations added (mg l^{-1})	Soil	Sand and clay 2%	Sand and clay 4%
10	10.37	5.27	23.48
100	217.25	70.85	482.46

It can be concluded that the studied samples differ in their ability for mercury retention and in the forces of different sites on the surfaces of these samples to adsorb mercury. The palygorskite and soil dominated with palygorskite have highly adsorption capacity for mercury, which indicates the importance of using palygorskite to reduce soil and water pollution with heavy metals.

This research has demonstrated the potentiality of using fibrous clay minerals (palygorskite) for limiting soil and water pollutions by heavy metals and it can be used to reduce soil and water pollution with heavy metals. Especially these natural materials are good sorbents and inexpensive and available whether in some soils or scattered as clay deposits in many countries.

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ادمصاص الزئبق بواسطة معدن الطين الباليجورسكيت

عبد العظيم شهوان سلام ، محمد سليمان السويلم و عبد الله سليمان الفراج

قسم علوم التربة – كلية علوم الأغذية والزراعة – جامعة الملك سعود – السعودية .

يعتبر معدن طين الباليجورسكيت من المعادن التي تسود في حبيبات طين العديد من الترب والرواسب في المناطق الجافة و تهدف الدراسة إلى تقدير خواص إدمصاص الباليجورسكيت لعنصر الزئبق، حيث أجريت تجارب لإدمصاص وانطلاق عنصر الزئبق بأستخدام تجارب النظام الثابت والسريان في الأعمدة، واستخدمت لذلك كل او بعض من خمسة مواد وهي: عينة تربة رملية طميية (ترب رسوبية Torrifluvents) تم جمعها من منطقة حرض ويسود الباليجورسكيت تركيبها المعدني، الطين المفصول من تلك الترب والمشبع بالصوديوم، عينة تربة رملية مغسولة بحمض الهيدروكلوريك، عينتين من الطين المفصول مضافه إلى الرمل المغسول بنسبة ٢٪ ، و ٤٪ على التوالي. تم التعرف على بعض الخصائص الفيزيائية والكيميائية والمعدنية لتلك المواد. أوضحت النتائج انطباق معادلة لانجمير لادمصاص الزئبق في جميع العينات، حيث زادت الكمية المدمصة بزيادة تركيز محلول الاتزان في مدى التركيزات المستخدمة، وكانت السعة العظمى للادمصاص (b) وطاقة الارتباط (k) مرتفعة جدًا في عينة الطين المفصول والغير مخلوط مقارنة بالعينات الأخرى ويمكن ترتيب العينات المدروسة من حيث قيم (b) كما يلي: الطين المفصول > طين ٤٪ > طين ٢٪ > التربة. وحسب قيم (k) تأخذ العينات الترتيب التالي: طين غير مخلوط > تربة > طين ٤٪ > طين ٢٪ . تم تطبيق معادلة Freundlich لوصف إدمصاص الزئبق على العينات المدروسة وقد تشابهت النتائج (إلى حد ما) مع ما أظهرته معادلة لانجمير. أوضحت نتائج انطلاق الزئبق المدمص على أن معظم الزئبق قد تم استخلاصه في تجربة الأستخلاص الأول بخلات الأمونيوم (١ عياري). دلت نتائج تجارب الأعمدة على أن أكبر كمية من الزئبق المستخلص تم الحصول عليها بعينة الطين المخلوط بنسبة ٤٪ . بناءً على النتائج يمكن اعتبار معدن الباليجورسكيت ذو سعة ادمصاصية عالية للزئبق وذلك بما لديه من خصائص تساهم في مسكه والإبقاء عليه في ترب ومياه المناطق الملوثة. هذا البحث يشير الى امكانية استخدام معادن الطين الابرية (الباليجورسكيت) للتخفيف من تلوث التربة والمياه وخاصة ان هذه المواد طبيعية ورخيصة ومتوفر في العديد من الدول.