Effect of Calcium Carbonate Content on Potential Toxic Heavy Metals Adsorption In Calcareous Soils

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ABSTRACT

Research on the sorption characteristics of Pb, Cd and Ni on calcareous soils of Egypt is scarce. The aim of this work is to investigate the effects of five types of calcareous soils collected from RasSedr and Marute stations, varied in their $CaCO_3$ contents and interaction between some potential toxic elements (PTE's) represented by Pb, Cd and Ni applied at two rates i.e. 1 and 3 mMon the adsorption characterization of these pollutants through the kinetic experiment conducted at DRC. The current research was initiated to investigate competitive and non-competitive sorption characteristics of Pb, Cd and Ni (on double phase forms applied to investigate soils). The obtained results revealed that Elovich, Modified Frendlich equations were the best fitted models in describing the kinetic data by having high r^2 and low SE. Increasing of $CaCO_3$ content in used soils significantly increased the adsorption of pollutants regardless the concentration used, this result was observed the increasing of the rate constants of used models. Concerning the effect of double phase on heavy metals adsoption, data showed that at different concentrations applied;represent the mixture of both (Pb& Cd)and (Pb& Ni) led to significant increase in Pb as documented in S_2. Typical, results showed that represents of Pb and Ni individually with Cd, led to increase the adsorption of Cd with Pb than with Ni.through the rate constants of used models, different mechanisms were discussed in relation to antagonistic and synergistic between PTE's studied.

Key words:Adsorption,Calcareous soils and (Elovich, Modified Frendlich Equations) and Potential toxic elements (Pb^{2+}, $Cd^{2+}$and Ni^{2+}).

Introduction

Heavy metals are among the most important soil pollutant which are concerned from both agricultural and environmental standpoints (Papazoglou*et al.,* 2007). Cadmium and nickel are among toxic heavy metals in which are important from the view of their toxicity for different organisms (Nan *et al.*, 2002 and Rooney *et al.*, 2007). Some soil properties influence bioavailability of these metals including pH, organic matter and clays contents (Wallace *et al.,* 1977; Sauerbeck, 1991; Christensen *et al*., 1996; Karaca, 2004 andWeng*et al*., 2003, 2004). Roque-Rossel*et al.* (2010) investigated Ni sorption in Limonite and concluded that some soil factors including soil porosity and pH have great effects on the mobility of this metal. Sorption mechanism is an important aspect and can be studied by using new procedures such as artificial neural network (Anagu*et al.,* 2009). Information on the sorption of metals in the presence of other metals is vital in understanding environmental risk of these metals (Chotpantarat*et al.*, 2011).

Sorption of metals is a competitive process between different ions in the solution and soil surface. The study of single metal solution has imperfect practical applications. Competitive adsorption takes place when at least two metals adsorb to the same adsorption sites (Fontes*et al.,* 2000 and Ryan *et al.,* 2011). According to the findings of Chotpantarat*et al.* (2011), sorption capacities of each metal in binary and multi-metal systems were lower than that of single metal system. Srivastava *et al.* (2009) reported that the adsorption capacity of Ni onto rice husk ash in the binary system was higher than Cd. They observed that sorption of these metals is antagonistic.

Despite of contradictory reports concerning the capability of the Langmuir and Freundlich isotherms in interpreting metals sorption in soil (Sparks, 1998), these two models are among the commonly used sorption isotherms (Peric*et al.,* 2004) and some of their parameters are satisfied in describing metals sorption phenomena (Holm *et al.,* 2003 and Usman, 2008). Srivastava *et al.* (2009) reported that the sorption of Ni and Cd onto rice husk ash followed the Freundlich isotherm.

Although voluminous researchesofheavy metals sorption characteristics of Pb, Cd and Ni in individual face are available, the sorption of double face forms on calcareous soils in Egypt is scarce. Current research aims to initiate the investigation of competitive sorption characteristics of Pb, Cd and Ni in double face forms on five soils varying generally in their characteristics and specifically in active $CaCO_3$ content.

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Materials and Methods

Soil Samples:

Five soil samples (S1-S5) were collected from surface soils (0-30 cm) of different field's locations in RasSedrand Mariut regions, of Egypt. The soils were air dried, crushed, and passed through a 2 mm sieve.Somephysical and chemical properties of the soil samples including pH, electrical conductivity (EC), calcium carbonate content ($CaCO_3$), active calcium carbonate, organic matter (OM), cation exchange capacity (CEC) and clay content were determined using standard methods mentioned by Cottenie, *et al.*(1982) and Sparks (1998)and the results were presented in Table (1).

Table 1: The main criteria of the selected soil samples.

Soil No	pH 1:2.5	EC dsm^{-1}	CaCO$_3$ %	Active CaCO$_3$%	OM %	CEC mq100g^{-1}	Surface area m^2 g^{-1}	Clay %
S$_1$	8.15	0.62	13.4	11.1	1.81	6.21	85.0	17.9
S$_2$	8.10	6.65	20.2	5.1	0.87	5.57	27.5	5.1
S$_3$	8.47	2.47	27.9	10.0	1.95	6.78	58.0	10.2
S$_4$	8.50	0.22	34.9	2.5	1.27	3.62	12.4	2.8
S$_5$	8.30	2.97	53.4	8.0	1.21	5.88	45.0	7.6

Kinetic Experiment:

Heavy-metals in chloride form of (Pb^{+2}, $Cd^{+2}$and Ni^{+2}) were used to obtain three aqua solutions of double component solutions i.e. (Pb& Cd),(Pb&Ni) and (Cd&Ni) at fixed concentrations of 1 and 3 mM prepared from these cations and kept for sorption study.

Sorption experiments were carried out using Electrical Stirred Flow Unit (ESFU) method for the 5 soil samples. The system was vigorously shaken and the solution samples were taken after different periods; 1, 2, 6, 24, 48, 96 and 168hr. at 25°Cand analyzed according toCottenie*et al.*(1982) by Atomic Absorption Spectrophotometer (AAS 200A Buck Scientific Model).

KineticsModels:

In order to investigate the mechanism of sorption and potential rate controlling heavy metals transport and chemical reaction processes, both theoretical and empirical widely used kinetic models have been applied to test the experimental data. Equations presented in Table (2) were used to fit the data of three heavy metals under investigation.

Table 2: Equations used to study the adsorption of three heavy metals.

Models	Equation	Description
Elovich	$q_t = b + (1/a) \ln t$	q_t sorption capacity at time t in min
Modified Freundlich equation	$q = bk^a$	t time in min
	$\log q = \log b + a (\log t)$	a and b constants

Thus the constants can be obtained from the slope and intercept of the linear plot of q_t versus ln t. Equations will be used to test the applicability of each equation to the kinetics of each heavy metal (II) sorption on soils.

Results and Discussion

Adsorption of the Heavy Metals on Calcareous Soils:

The concentrations ofPb, Cd and Ni adsorbed on active sites of the five calcareous soils used in this work as a result of applying these heavy metals in 1 and 3 mM in double phase of(Pb^{+2}& Cd^{+2}), (Pb^{+2}& Ni^{+2}) and(Cd^{+2}& Ni^{+2}) are illustrated in Figure (1). It is obvious from the obtained results that the adsorption of these heavy metals was enhanced by the elevated the levels of added solution from 1 to 3 mM for the three elements. Similar results were reported by Elbana and Selim (2010) and (2013).
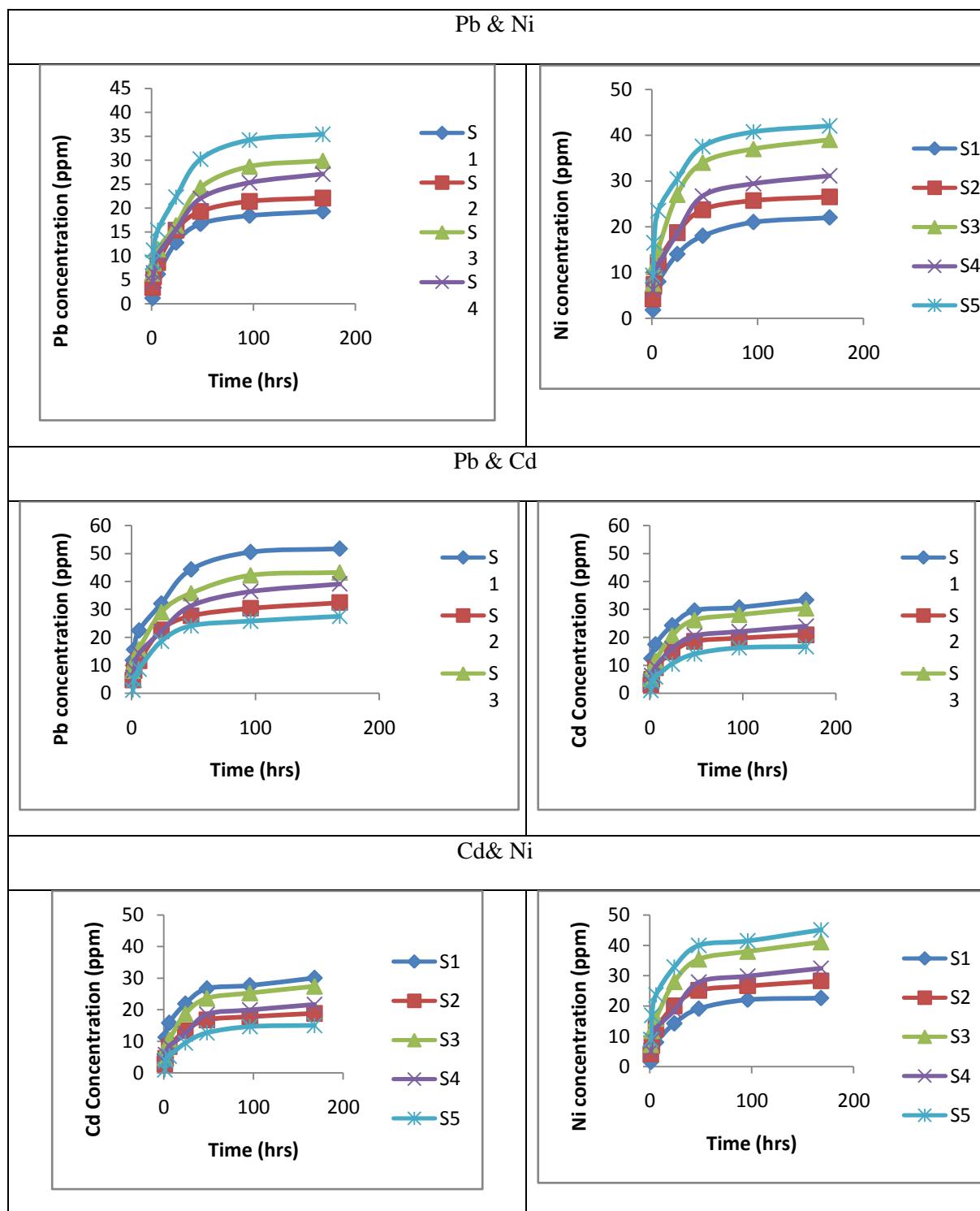
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Fig. 1: The concentrations of adsorbed heavy metals on the calcareous soils at 1 mM main concentration in different double fase forms.
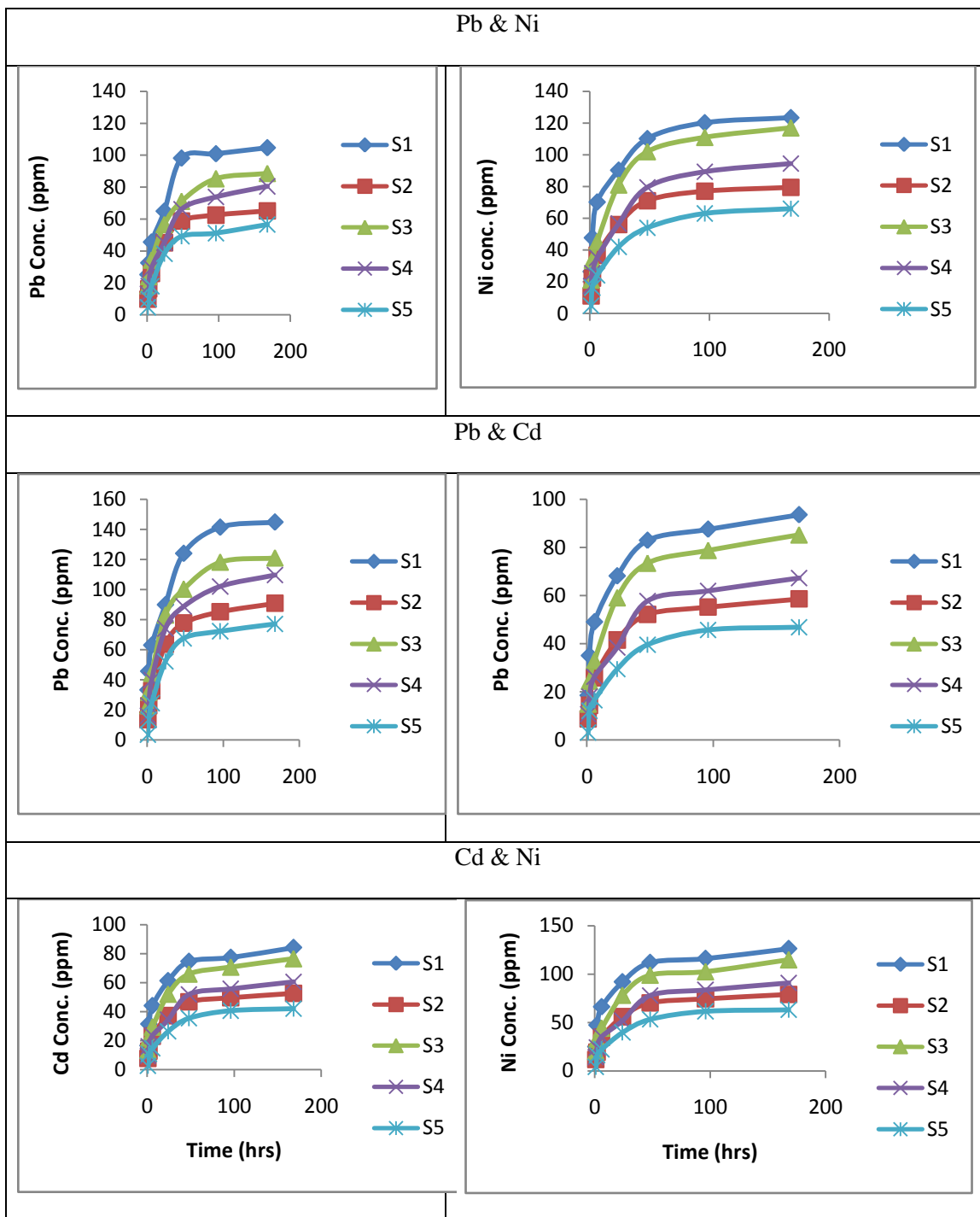
Fig. 2: The concentrations of adsorbed heavy metals on the calcareous soils at 3 mMmain concentration in different double fase forms.

As a time factor, adsorption isotherm takes three stages, a rapid process from the starting time to about 2 hr, intermediate stage until about 100 hrand after that, almost steady state conditions until the end of reaction time (168 hr).Also the values of adsorbed elements data generally indicate that regardless the bi-forms of solutions applied in this experiment that Cd <Pb<Ni in the five soil samples, for example in S_5 they are; 315, 286 and 160 mM at the end of reaction applying 3 mM of added solution. The lower capability of Cd in competition with other elements in occupying adsorption sites on clay, calcium carbonate, and organic matter contents are the main reason of its low adsorption in the case of double phase forms used. In this respect Ghasemi-Fasaei and Jurrah (2013) refereed to competition between Cd and Zn adsorption.

Comparing the adsorption of the three metals in the five soil samples which have different content of total and active $CaCO_3$, clay percent and consequently the total surface area (Table1); data imply that at

1mMconcentration of Pb& Ni the adsorption of Ni in general was higher than Pb in all soils used. Numerically, in S_2 after 168 h in kinetic work the maximum adsorption was about 26 ppm of Ni against 22 ppm for Pb, the same result was observed in both concentration used (figure 1). In contrast, at the same concentration in the case of (Pb& Cd), the adsorbed Pb on used soils was higher than Cd in all cases of soils or concentration applied. Also, data showed that Ni concentration adsorbed on calcareous soils was higher than Cd in the case of used (Cd & Ni) as a test solution.

The obtained result may proves that the total content of $CaCO_3$ is not the limiting factor in the adsorption and retention processes but the active $CaCO_3$ fraction and the clay contents are more effective. In this work in the case of Pb and Cd, although S_5 had higher total $CaCO_3$ (53%) the active $CaCO_3$ in this soil is 8%. In S_1, however, the total $CaCO_3$ is about 13%, but the active $CaCO_3$ is about 11%. In both soils in the case of apply (Cd&Ni) as a sorption solution; S_1 adsorbed about 33 ppm, while S_5 adsorbed 17 ppm.

Adsorption Kinetics of the Heavy Metals:

To assess the retention and mobility behavior of Pb^{+2}, Cd^{+2} and Ni^{+2}) in the five calcareous soil samples,two kinetic models were applied, these models showed priority to describe the kinetic data under double phase conditionsby having high r^2 and low SE. The parameters of these estimated equations (Elovich, Modified Freundlich Equation (MFE), for two concentrations (1 and 3 mM) are recorded in Tables 3 and 4 for Pb, Cd and Ni metals respectively.

Table 3: The estimated parameters of the MFE kinetic models to assess adsorption behavior of Pb, Cd, and Ni under different double phase forms applied.

Constant	(1mM)						(3 mM)					
	Cd &Ni		Pb&Cd		Pb& Ni		Pb&Ni		Pb&Cd		Cd &Ni	
	Ni_1	Cd_1	Pb_1	Cd_1	Pb_1	Ni_1	Ni_3	Pb_3	Cd_3	Pb_3	Cd_3	Ni_3
S_1												
a	0.47	0.42	0.45	0.38	0.51	0.44	0.45	0.48	0.47	0.56	0.62	0.47
b	0.44	0.26	0.36	0.31	0.29	0.47	0.92	0.81	0.75	0.81	0.71	0.88
r^2	0.93	0.93	0.94	0.93	0.96	0.95	0.94	0.97	0.93	0.94	0.93	0.93
SE	0.17	0.17	0.19	0.17	0.14	0.13	0.15	0.11	0.17	0.19	0.17	0.17
S_2												
a	0.27	0.20	0.29	0.28	0.37	0.35	0.37	0.35	0.37	0.42	0.37	0.31
b	0.72	0.54	0.76	0.59	0.62	0.73	1.18	1.07	1.04	1.21	0.99	1.17
r^2	0.97	0.97	0.98	0.97	0.98	0.97	0.96	0.85	0.97	0.98	0.97	0.97
SE	0.08	0.08	0.07	0.08	0.07	0.08	0.10	0.18	0.08	0.07	0.08	0.08
S_3												
a	0.34	0.32	0.37	0.36	0.31	0.33	0.34	0.46	0.34	0.41	0.52	0.41
b	0.93	0.75	0.93	0.80	0.82	0.93	1.39	1.27	1.24	1.38	1.20	1.38
r^2	0.98	0.98	0.99	0.98	0.99	0.99	0.98	0.99	0.98	0.99	0.98	0.98
SE	0.06	0.06	0.04	0.06	0.03	0.05	0.06	0.03	0.06	0.04	0.06	0.06
S_4												
a	0.24	0.18	0.27	0.26	0.36	0.32	0.34	0.30	0.33	0.36	0.35	0.29
b	0.79	0.61	0.80	0.66	0.69	0.82	1.28	1.15	1.10	1.25	1.06	1.23
r^2	0.98	0.98	0.99	0.98	0.99	0.99	0.99	0.99	0.98	0.99	0.98	0.98
SE	0.06	0.06	0.05	0.06	0.04	0.04	0.04	0.04	0.06	0.05	0.06	0.06
S_5												
a	0.29	0.22	0.34	0.31	0.28	0.27	0.31	0.28	0.46	0.39	0.44	0.36
b	1.08	0.90	1.10	0.95	0.96	1.08	1.54	1.42	1.39	1.56	1.35	1.52
r^2	0.96	0.96	0.99	0.96	0.99	0.96	0.96	0.99	0.96	0.99	0.96	0.96
SE	0.08	0.08	0.03	0.08	0.03	0.07	0.08	0.04	0.08	0.03	0.08	0.08

Comparing the isotherm results of the 5 soil samples, Table (3) it is found that the constants a, b and r^2 of MFE equation can be give both the rate of PTE's adsorption on the active sites of soils and the capacity factor represented by b constant. Modified Freundlich equation MFE proved to be the best in describing the kinetic data by having higher R2 and lower SE comparing to other kinetic model tested.

According to the higher coefficient of determination r^2 and the lowest standard error SE, data presented in Tables 3 and 4, the kinetics models under investigation could be arranged as follows; MFE was the best in describing the kinetic data by having high r^2 ranged between 0.93 and 0.99 with low SE ranged between 0.01 and 0.08 in all cases studied of soils or double phase solutions,followed Elovich equation. It should be mention that increasing the application rate of pollutants applied to the soil samples didn't influenced the studied constants. These obtained results are in agreement with Safarzadeh*et al.* (2009).the most important soil properties which influence Cd adsorption in calcareous soil.

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Table 4: The estimated parameters of the Elovichkinetic models to assess adsorption ofPb, Cd, and Ni under different double phase forms applied.

Constant	(1mM)						(3 mM)					
	Cd &Ni		Pb&Cd		Pb& Ni		Pb&Ni		Pb&Cd		Cd &Ni	
	Ni_1	Cd_1	Pb_1	Cd_1	Pb_1	Ni_1	Ni_3	Cd_3	Pb_3	Cd_3	Pb_3	Ni_3
S_1												
a	5.46	3.13	4.7	4.04	4.23	2.82	10.79	12.17	10.79	12.17	7.86	11.83
b	0.82	1.24	0.73	1.80	1.69	1.13	2.86	5.18	2.86	5.18	3.18	4.73
r^2	0.99	0.99	0.99	1.00	0.99	0.99	0.99	1.00	0.99	1.00	0.99	0.99
SE	1.43	0.86	0.95	0.81	1.16	0.77	3.09	2.43	3.09	2.43	2.11	3.25
S_2												
a	5.73	3.69	4.79	4.60	4.98	3.32	9.69	13.98	9.69	13.98	9.29	13.93
b	3.94	2.93	2.92	4.29	3.96	2.64	8.12	12.11	8.12	12.11	7.86	11.09
r^2	0.99	1.00	0.99	1.00	1.00	1.00	0.84	1.00	0.84	1.00	1.00	1.00
SE	1.47	0.79	0.96	0.98	1.07	0.71	13.60	3.00	13.60	3.00	1.99	2.99
S_3												
a	7.32	5.12	8.84	6.58	6.92	4.61	14.50	19.93	14.50	19.93	12.91	19.08
b	5.47	4.66	4.64	6.34	6.29	4.20	12.84	18.23	12.84	18.23	11.75	17.89
r^2	0.97	0.99	0.98	0.99	0.99	0.99	0.98	0.99	0.98	0.99	0.99	0.99
SE	3.65	1.30	2.24	1.89	1.76	1.17	6.68	5.46	6.68	5.46	3.28	5.07
S_4												
a	6.84	4.06	5.35	5.17	5.49	3.66	13.83	15.93	13.83	15.93	10.25	15.37
b	3.64	3.12	3.17	4.69	4.20	2.80	8.51	13.06	8.51	13.06	7.85	11.77
r_2	0.99	0.99	0.99	0.98	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.99
SE	2.09	1.44	1.48	1.98	1.95	1.30	4.72	5.43	4.72	5.43	3.64	5.47
S_5												
a	8.32	5.14	6.9	6.37	6.94	4.62	16.98	18.96	16.98	18.96	12.95	19.42
b	9.82	7.98	7.21	11.07	10.77	7.18	20.80	31.99	20.80	31.99	20.11	30.17
r^2	0.99	0.99	0.99	0.99	0.99	0.99	0.98	0.99	0.98	0.99	0.99	0.99
SE	2.82	1.21	1.84	1.55	1.63	1.09	7.74	4.87	7.74	4.87	3.04	4.56

The comparison between rate constant of MFE (a constant) in different treatments showed a variation according to the phase study. In the 1st phase (Cd & Ni), results imply that adsorption of Ni was higher than Cd in all used soils and in both concentrations applied in this work. For example, in S_5 the rate of Ni adsorption was 0.29 mg kg^{-1} min^{-1}, in contrast the rate of Cd was only 0.22 mg kg^{-1} min^{-1}. The corresponding values at 3mM concentration were 0.41 and 0.28 mg kg^{-1} min^{-1}.

In the case of (Pb& Cd), the obtained results revealed that the adsorption of Pb was higher than Cd. The numerical values of both pollutants at 3 mMl concentration applied showed that in S_1 which had highest active CaCO3, the adsorbed Pb was 0.56 mg kg^{-1} hrs^{-1}, the Cd value, however, was 0.47 mg kg^{-1} hrs^{-1}. The same trend was also observed in other soils tested. In other words, the capability of soils to sorb Pb is higher than Cd. Again, it should be mention that in the low concentration *i.e.* 1 mM, the same trend was recorded.

In the 3rd case represented by Pb and Ni, results emphasized that adsorption of Pb was higher than Ni in all soils used regardless the concentration applied of double phase solutions. Numerically in S_4, at low concentration applied (1 mM), the rate constant was 0.34mg kg^{-1} hrs^{-1} for Pb and 0.30mg kg^{-1} hrs^{-1} for Ni. By increasing the concentration to 3 mM, the respective values increased to 0.36 and 0.32 mg kg^{-1} hrs^{-1}.

Correlation coefficient have been estimated between soil properties of the five samples and the parameters of the kinetic equations (a and b) for the two concentrations of each heavy metal under consideration.The data illustrated in Figures (2-4) represents the (*a*) constant values for the three heavy metals under consideration. Regarding the other constants have the same trend as (*a*) constants; it is not necessary to illustrate the data in figures.

The (*a*) constant which represents the rate of pollutant release from calcareous soil or the intensity factor showed that increasing the application rate applied to calcareous soils led to increase the rate of pollutant release from the studied soils. Regarding the (b) contrast, data showed that in most cases the capacity factor represented by (*b*) constant gave the same trend with increasing the rate of adsorption.

The obtained data illustrated in Figures(3 and 4) indicate the following conclusion:

- The (a) and (b) constants in Elovich model is highly positive correlated with active calcium carbonated content (2.5-11.9%), surface area (12-85ml) and clay percent (2.8-12.9%). The same trend in the three concentrations of the heavy metals Pb, Cd and Ni.

- The (a) constant in Freundlich equation has also significant correlation with these soil criteria.

- The influence of organic matter content of these calcareous samples have positive correlation (range between significant and non significant) with (a and b)constants in Elovich model for Pb, Cd and Ni.

- The low values of EC (0.22-6.65 dsm^2) have negative relation with the constants (a and b) in Elovich and Diffusion equations.

- Increase of the concentration of the added heavy metal (1 and 3 mM) has no influence on the correlation between soil parameters and the constants of kinetic equations applied for the adsorption trend of each of Pb, Cd and Ni.
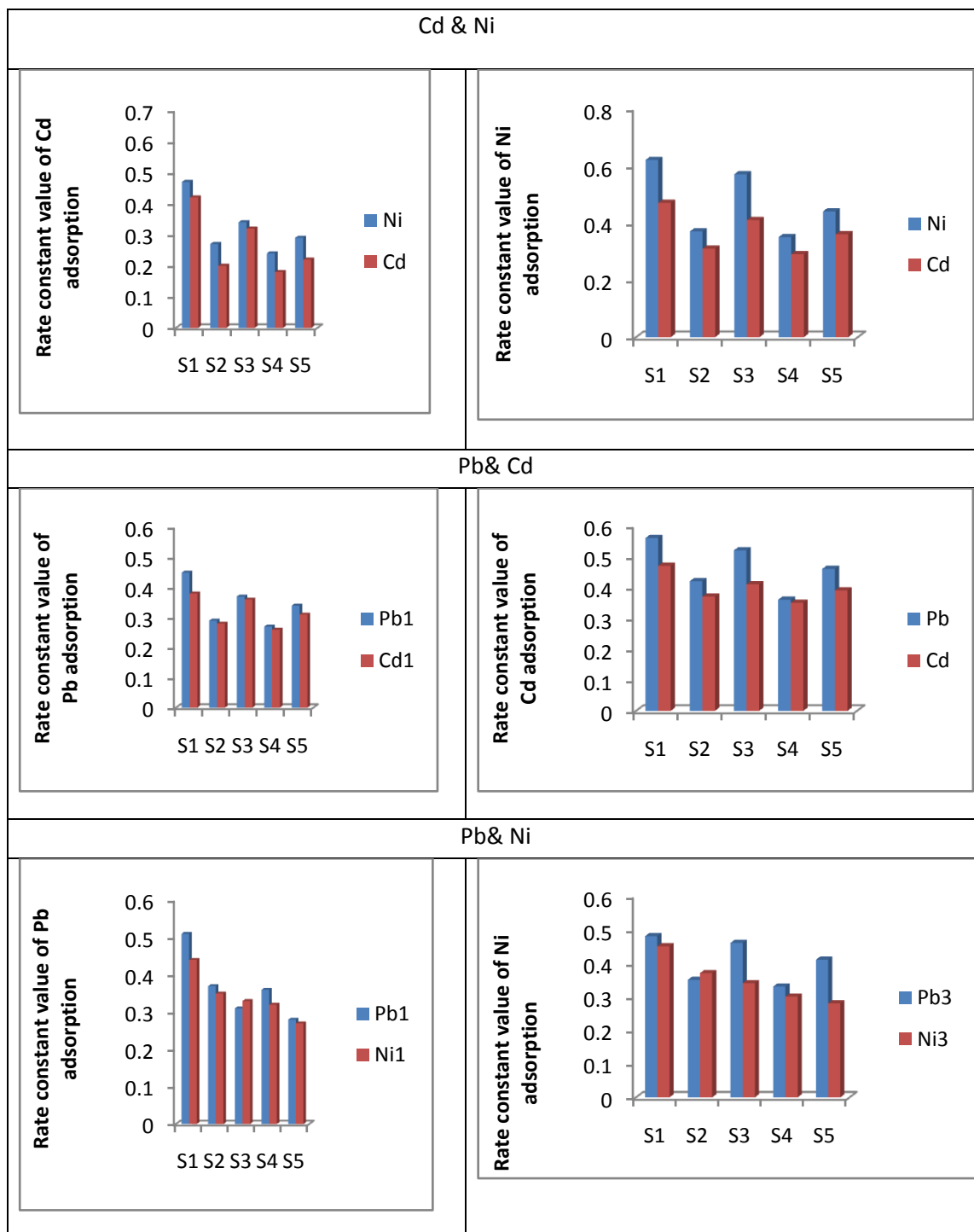

Fig. 3: The correlation between rate constant (a) of MFE for PTE's adsorption on different soil samples under different cases of double phase adsorbed solutions
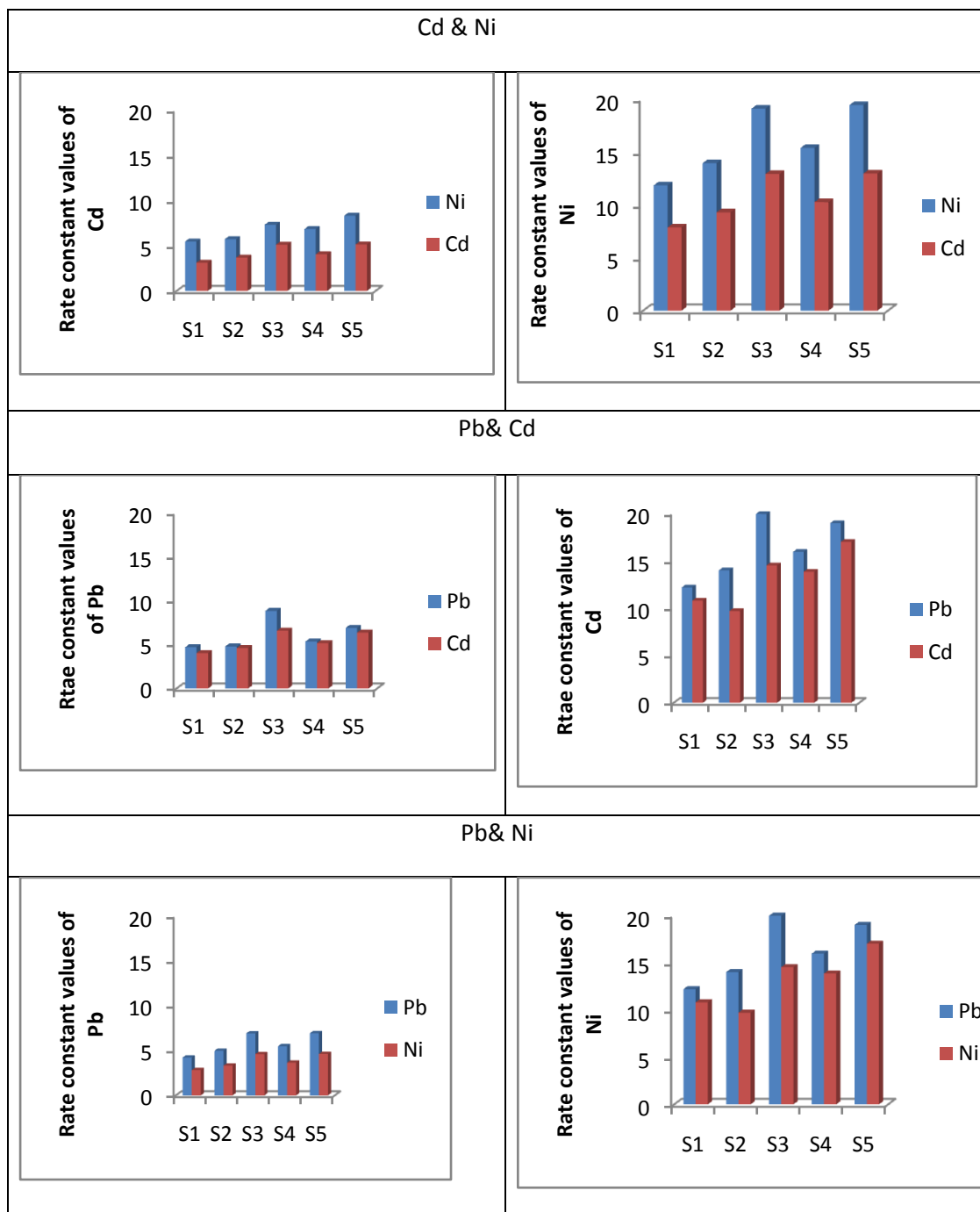
Fig. 4: The correlation between rate constant (a) of Elovich model for the studied PTE's adsorption on different soil samples under different cases of double phase adsorbed solutions

Conclusion:

The comprehension understanding of adsorption and mobility processes of potential toxic elements is very important to minimize the hazardous effects of these pollutants to environmental quality and human health. The role of calcium carbonate in the soil is very effective on the adsorption of Pb^{+2}, Cd^{+2} and Ni^{+2}). The fine participate form of $CaCO_3$ as active portion in the process of adsorption more than the total content. The variation between the double phase elements on the adsorption and retention depends mainly on the radios number and electro charges of each heavy metal. In pure solutions prepared from PTE's i.e. Pb&Ni, Pb&Cd and Cd&Ni, results indicated that competition process took place between different groups of pollutants on the active sites of calcareous soils. In general, the values of adsorbed elements proved that Ni>Cd, Pb>Cd and

Pb>Ni due to the higher capacity of Pb followed by Ni and Cd on occupying the adsorption sites on clay, calcium carbonate and organic mattercontents found in these soils.

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