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Kinetics of copper release in some calcareous soils from Sulaimani governorate, Iraqi Kurdistan Region.

ABSTRACT

This study was conducted to estimate copper release kinetic in five calcareous soils includes (Sharazor, Qaradagh, Bazian, Mawat, and Surdash) at the Sulaimani governorate, Iraqi Kurdistan – region. The highest amount of Cu release during 48 hours was least in soil Qaradagh (0.75mg kg⁻¹). While Soil Mawat exhibited the highest release (2.272mg kg⁻¹). The value of diffusion rate constant (kp) from parabolic diffusion varied from 0.123 to 0.321 with a mean of (0.221). The initial Cu release constant (αs) and Cu release rate coefficient (βs) from simple Elovich model, ranged from 0.42 to 1.164 with a mean of 0.6856, and from 2.197 to 5.952 with a mean of 3.6918. While the values of the rate constant (a and b) from the power function model with the mean were (0.249 and 0.605) respectively varied widely with the five soil. The parabolic diffusion was the best-fitted model used to describe Cu release process very well in the studied soils.

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INTRODUCTION

Copper, like other micronutrients, is essential for plants, animals, and microorganisms, but it is toxic when its concentration more than the allowable level (Baker, 1990). Copper is adsorbed on manganese and iron oxides, organic matter and the surface of clay minerals in the soil; it is also precipitated as hydroxides, sulfide and carbonate copper (McBride, 1981; Baker, 1990). Generally, the amount of total copper content in Sulaimani governorate soils is typically sufficient but its soluble form in soil solution is very low due to high pH, low organic matter, and high calcium carbonate content in calcareous soils; therefore, the bioavailability of Cu is usually low in these soils, copper deficiencies can overcome by the addition of Cu fertilisers (Rate, and Sheikh – Abdulla, 2017). DTPA combines with free Cu ions in solution, forming soluble complexes, as a result the free copper ion activity in the solution decreases. Thus, Cu ions release from the surface of soil particles in order to compensate the free Cu ions in solution (Lindsay, and Norvell, 1978). The desorption of Cu from exchangeable sites and release of Cu from organic matter, crystalline mineral, and other metastable compounds to the solution, thus this process control the mobility of soil Cu, this leads to the relative contribution of copper availability for plants (Singh et al., 1994). Reyhanitabar and Karimian, (2008) showed that simple Elovich and two constant-rates were the best models for the explanation of Cu release of calcareous soils of central Iran. Data on the kinetics of Cu release in some calcareous soil

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in Sulaimani governorate in the Kurdistan region of Iraq are limited. Thus, the aims of the current research started to:

- estimate the release kinetic of Cu from studied soils.
- determine the fitness of various models for the description of the kinetics of Cu release from studied soils.

MATERIALS AND METHODS

Soil samples were taken from the depth (0-30) cm in five calcareous soil include (Sharazor, Qaradagh, Bazian, Mawat, and Surdash) at Sulaimani governorate, Iraqi Kurdistan region. The study area lies between (longitudes 35°15' 27" N; and latitudes 45 07' 37" E) as shown in (fig.1). All the five soils classified as (Argixerolls, Rendolls, Pelloxererts, Rendolls, and Argixerolls) according to the Soil Survey Staff, (2004). The soil samples air-dried, crushed, and passed through a 2-mm sieve before soil analysis and release studies. Some basic physicochemical properties of the studied soil including particle size distribution, pH, EC, organic matter %, calcium carbonate %, and CEC determined according to the methods of soil analysis as described by (Page et al. 1982; Rayan J. et al. 2001), available concentration of Cu in studied soil as defined in DTPA method (Lindsay and Norvell, 1978).

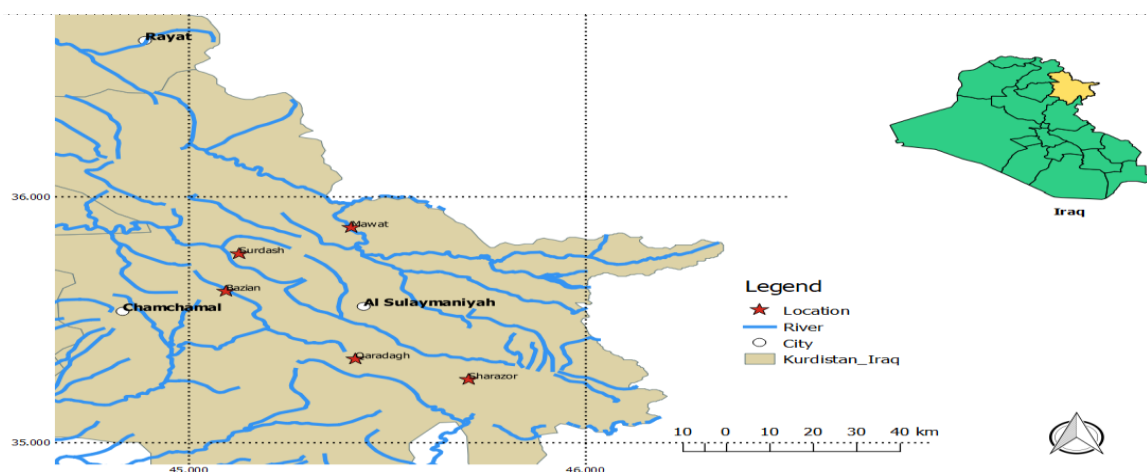


Fig. 1. The location of the studied area

Kinetic study procedure:

Release kinetics were studied using DTPA extracting solution (0.005M diethylene triamine pentaacetic acid, 0.1 M triethanolamine, and 0.01M CaCl₂) at pH=7.3; (Lindsay and Norvell 1978) as an extractant as follows: 50g oven-dried soil in duplicate were extracted with 100 ml of DTPA extracting solution with ratio (m/v of 1:2) at 25°C ±1 in a constant temperature shaker, for periods of 0.5, 1, 2, 4, 6, 8, 24,48 hours. Added five drops of toluene to prevent microbial activity. For each shaking period, the soil suspension immediately filtered through Whatman paper No.42, then the concentration of Cu determined in a solution using Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). Eight models were used to describe the copper release in the studied soil including Zero order, First order, Second Order, Third Order, Simple Elovich, Parabolic diffusion, Power function, and Hyperbolic (Martin and Sparks, 1983; Havlin et al., 1985) (Table 1). The model that gave the highest value of determination coefficient (R²) and the minimum value of root mean square error (RMSE), and Akaike information criterion (AIC) considered as the best model. The value of determination coefficient, root mean square error, and Akaike information criterion calculated according to the following equations:

$$\text{The determination coefficient (R}^2\text{)} = \frac{\sum (q_{\text{meas}} - q_{\text{apred}})^2}{\sum (q_{\text{meas}} - q_{\text{apred}})^2 + \sum (q_{\text{meas}} - q_{\text{pred}})^2}$$

$$\text{Root mean square error (RMSE)} = \left\{ \frac{\sum (q_{\text{meas}} - q_{\text{pred}})^2}{n - 2} \right\}^{1/2} \text{ where } q_{\text{meas}} \text{ and } q_{\text{pred}},$$

q_{aepred} represent the measured, predicted, and average predicted Cu released, and n is the number of measurements. Akaike information criterion(AIC) generally calculated with the software. The basic formula of $AIC = 2K - 2(\ln L)$ (Mirzaei et al. 2017).

Where K indicates the number of parameters and L indicates a probability of the data given a model (likelihood).

Table 1. Kinetic models used in the studied soils.

Models	Equations	Parameters
Zero order	$q_t^* = q_0^* - k_0 t$	k_0 , zero-order rate constant ($\text{mg Cu kg}^{-1} \text{ h}^{-1}$)
First Order	$\ln q_t = \ln q_0 - k_1 t$	k_1 , first-order rate constant (h^{-1})
Second order	$1/q_t = 1/q_0 + k_2 t$	k_2 , second-order rate constant [$(\text{mg Cu kg}^{-1})^{-1}$]
Third order	$1/q_t^2 = 1/q_0^2 - k_3 t$	k_3 , third-order rate constant [$(\text{mg Cu kg}^{-1})^{-2} \text{ h}^{-2}$]
Simple Elovich	$q_t = 1/\beta_s \ln(\alpha_s \beta_s) + 1/\beta_s \ln t$	α_s , initial Cu desorption rate constant ($\text{mg Cu kg}^{-1} \text{ h}^{-1}$) and β_s , Cu desorption rate constant [$(\text{mg Cu kg}^{-1})^{-1}$]
Parabolic diffusion	$q_t = q_0 - k_p t^{1/2}$	k_p , diffusion rate constant [$(\text{mg Cu kg}^{-1})^{-0.5}$]
Power function	$\ln q_t = \ln a + b \ln t$	a , initial Cu desorption rate constant [$(\text{mg Cu kg}^{-1} (\text{h}^{-1})^b)$] and b , desorption rate coefficient
Hyperbolic	$q_t / t = (q_0 / t^{1/2}) t$	$t^{1/2}$ semi decomposition time

* q_t is the amount of soil Cu released by DTPA (mg Cu kg^{-1}) after the time (h) of extraction, and q_0 , initial Cu concentration at time = 0

STATISTICAL ANALYSIS

Statistical operations performed using the statistical software addinosoft (2016). XLSTAT statistical and data analysis solution. Boston, USA.

RESULTS AND DISCUSSION

Physicochemical properties of the soil

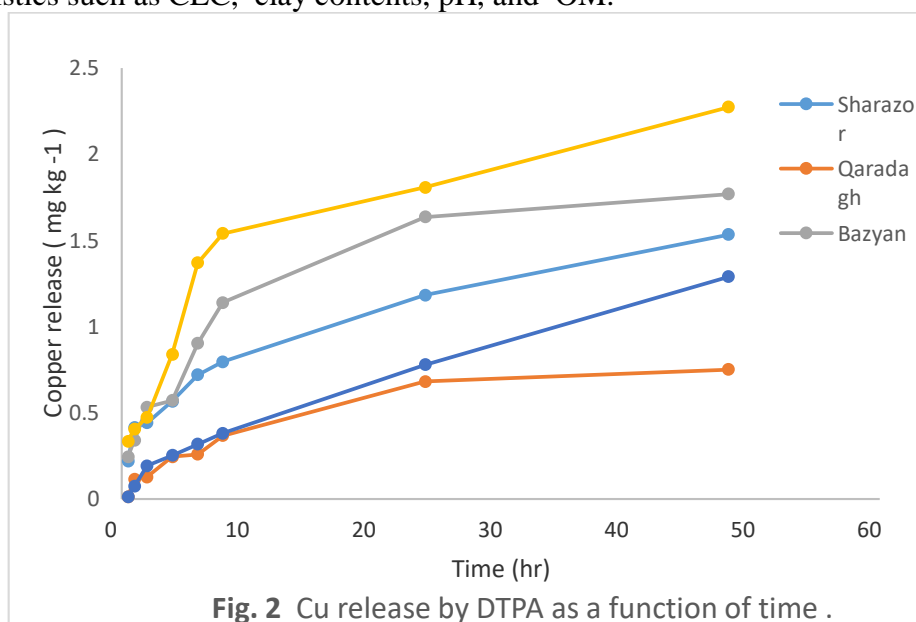
Some chemical and physical properties of the soils presented in (table 2) showed that the soils vary in their texture from silty clay to loam, with a range of organic matter, and total calcium carbonate content, from 90 to 25, and 25 to 430 g kg^{-1} respectively. Most of the soil had a neutral reaction (7.48 to 7.90). EC of the soil varied from 0.40 to 0.90 dS m^{-1} . The extractable Cu by DTPA varied from 0.12 to 0.51 mg kg^{-1} , whereas the CEC values ranged from 26.93 to 41.57 cmolc kg^{-1} . The texture classes of the studied soils ranging from (loam to Silty clay) which it means that the texture of these soils ranged from fine to moderately textured soils, and the soils were slight to moderately alkaline due to the value of the studied soils ranged between (7.48 – 7.9). Electrical conductivity (EC) of the soil samples ranged between (0.4 - 0.9 dS m^{-1}), this shows that the soil studied are non-saline and this might be due to relatively higher precipitation and variation of these locations in terms of topography. All soil considered as calcareous soil due to calcium carbonate content was between (25 – 430 gm kg^{-1}).

Table (2) Some chemical and physical properties in the studied soils.

Soil No.	pH	EC (dS m^{-1})	T.CaCO ₃ (g kg^{-1})	O.M g kg^{-1}	Sand	Silt	Clay	Textural class	DTPA Cu mg kg^{-1}	CEC cmole kg^{-1}
					g kg^{-1}					
Sharazor	7.49	0.60	180	25	37.20	475.10	487.70	Silty clay	0.51	41.15
Qaradagh	7.65	0.70	25	17	383.50	370.30	246.20	loam	0.47	26.93
Bazian	7.78	0.60	100	15	55.80	430.50	513.70	Silty clay	0.20	41.57
Mawat	7.48	0.9	25	16	161.90	434.20	403.90	Silty clay	0.38	39.42
Surdash	7.90	0.40	430	9.00	91.60	490.40	418.00	Silty clay	0.12	36.39

The pattern of Cu release

The amount of Cu released as a function of time from (0.5 to 48 hours) with DTPA extracting solution in the studied soil shown in (fig.2). The pattern of copper release models generally characterized by an initial rapid reaction, then the reaction started with a constant slowdown and it arrived at equilibrium at 48 hours. Our results are convenient with the findings of Motlagh, (2012) who stated that Cu release copper patterns from 14 calcareous agricultural soil of northern Iran generally characterized by an initial rapid reaction, the release rate started much slower. Lehmann and Harter (1984) measured the chelate-promoted kinetics of Cu release from soil to evaluate the bond formation strength and referred to the two-stage desorption kinetics to high bonding sites of low and high energy. They determined that there was a transmission of Cu from low-energy sites to higher energy sites with the increase in residence time. Yu and Klarup (1994) ascribed the slow-release stage of Cu to diffusion or slow dissolution of minerals. They concluded that surface coverings, oxides may dissolve slowly in the solution, possibly leaves further of the Cu available to extraction. The highest amount of Cu release for 48 hours was the least in soil Qaradagh (0.75mg kg^{-1}). While soil Mawat exhibited the highest release (2.272mg kg^{-1}). It may be due to CEC, pH and clay content, as noted soil Qaradagh had the lowest content of clay and CEC. The order of the amount of copper released into the soil was as follows: Mawat > Bazian > Sharazoor > Surdash > Qaradagh. Patterns of Cu release are governed primarily by soil characteristics. Our results are consonant with the findings of Motlagh,(2012) who showed that CEC and clay content are considered the most active factors that control the release of copper in calcareous soil from northern Iran. Likewise, McBride, (1989) stated that the metals sorption behavior in the soil varies from one soil to another which is influenced by soil characteristics such as CEC, clay contents, pH, and OM.

**Fig. 2** Cu release by DTPA as a function of time .

Fitting the Cu release data to mathematical kinetic models

Eight kinetic models applied on Copper releases which include zero-order, first-order, second-order, third-order, simple Elovich, parabolic diffusion, power function, and hyperbolic used to describe Cu release in the studied soils by DTPA extracting solution from 0.5 to 48 hours. The goodness of fit as determined by the mean and the range of comparison of determination coefficients (R^2), root mean square error (RMSE), and Akaike information criterion (AIC) of the models calculated and presented in (tables 3 and 4). The highest values of determination coefficient (R^2 , 0.949), the lowest values of root mean square error (RMSE, 0.117), and Akaike information criterion (AIC, -36.496) meant that the parabolic diffusion was the best-fitted model used to describe Cu release process very well (table 3, and 4). This is consistent with results obtained by Wambu et al., (2009) who reported that the parabolic diffusion equation fitted copper recoveries with $R^2 = 0.96$ that studied as a batch basis in H_2SO_4 and $CaCl_2$. Similar results were obtained by Ghasemi-Fasaei et al., (2007), who indicated that the power function, simple Elovich, and parabolic diffusion in Fars province in southern Iran, were the best-fitted models. On the other hand, Reyhanitabar and Karimian, (2008); Ghasemi-Fasaei et al. (2006) who showed that the best models for the description of Cu-desorption were two constant-rates and simple Elovich of calcareous soil from central Iran. The results of the comparison listed in (table 3, and 4) also showed that the values of R^2 , RMSE, and AIC for simple Elovich, power function, and zero-order were smaller than parabolic diffusion models could also be used to describe Cu release kinetics. On the other hand, according to R^2 , RMSE, and those models poorly described AIC values of first order, and hyperbolic kinetic models, the kinetic of Cu release in the studied soils; whereas, for the second-order, and third-order models due to lower value of R^2 , and higher values of, RMSE, and AIC the results were not so favourable. Therefore, in this study, the second-order and third-order models were not convenient for describing Cu release in the studied soil

Table3. The determination coefficient (R^2) and root mean square error (RMSE), and the Akaike information criterion (AIC) of various kinetic models used to describe the copper release in the studied soils.

Equation	Sharazor			Qaradagh			Bazian			Mawat			Surdash		
	RMSE	AIC	R^2	RMSE	AIC	R^2	RMSE	AIC	R^2	RMSE	AIC	R^2	RMSE	AIC	R^2
Zero order	0.149	-28.793	0.9	0.116	-32.756	0.839	0.291	-18.061	0.783	0.394	-13.213	0.746	0.072	-40.382	0.975
First order	0.381	-13.754	0.678	1.137	3.757	0.412	0.489	-9.733	0.604	0.530	-8.465	0.563	1.103	3.264	0.510
Second order	1.050	2.487	0.397	34.632	58.415	0.108	1.045	2.404	0.396	0.825	-1.387	0.409	28.520	55.308	0.123
Third order	6.485	31.610	0.216	3658.398	132.975	0.077	5.371	28.595	0.251	2.989	19.218	0.309	2530.362	127.076	0.081
Simple Elovich	0.115	-32.885	0.940	0.075	-39.748	0.933	0.148	-28.848	0.944	0.194	-24.540	0.938	0.174	-26.246	0.855
Parabolic diffusion	0.056	-44.542	0.986	0.061	-42.945	0.955	0.171	-26.551	0.925	0.258	-19.980	0.891	0.043	-48.464	0.991
Power function	0.110	-33.642	0.973	0.629	-5.717	0.820	0.147	-28.967	0.964	0.213	-23.008	0.929	0.504	-9.271	0.898
Hyperbolic	0.128	-31.143	0.425	0.028	-55.251	0.305	0.119	-32.399	0.469	0.162	-27.468	0.431	0.022	-59.072	0.296

The variable ranges and means of R^2 , RMSE, and AIC for the studied soils, and eight kinetic models presented in (table 4), the average of R^2 , RMSE, and AIC for all the five soil ranked as follows: Parabolic diffusion > simple Elovich > power function > zero-order > first-order > hyperbolic > second-order > third order.

Table 4. Mean and range of determination coefficient (R^2), root mean square error (RMSE), and Akaike information criterion (AIC) of different kinetic models for Cu release by DTPA in the five soils.

Equation	RMSE		AIC		R^2	
	Range	Mean	Range	Mean	Range	Mean
Zero order	0.072 - 0.394	0.204	- 40.382 - (-13.213)	-26.641	0.746 - 0.975	0.848
First order	0.381 - 1.137	0.728	-13.754 - (3.757)	- 4.980	0.412 - 0.678	0.553
Second order	0.825 - 34.632	13.214	-1.387 - (58.415)	23.445	0.108 - 0.409	0.286
Third order	2.989 - 3658	1240.721	19.218 - (132.975)	67.895	0.077 - 0.309	0.187
Simple Elovich	0.075 - 0.194	0.141	-39.784 - (-24.54)	- 30.461	0.855 - 0.944	0.922
Parabolic diffusion	0.043 - 0.258	0.117	-48.464 - (-19.98)	-36.496	0.891 - 0.991	0.949
Power Function	0.11 - 0.629	0.320	-33.642 - (- 5.717)	-20.121	0.82 - 0.973	0.917
Hyperbolic	0.022 - 0.162	0.092	-59.072 - (-27.468)	- 41.066	0.296 - 0.469	0.385

Kinetic Parameters

The values of the rate constants for parabolic diffusion, simple Elovich and power function models shown in (table 5). In parabolic diffusion model, k_p constant represents the diffusion rate constant is regarded as the measurement of the relative rate of Cu release in the five soils, the value of k_p ranged between 0.123 and 0.321 with a mean of (0.221). The highest value of diffusion rate constant recorded in Mawat soil and the lowest value attributed to Qaradagh soil. The lower value in the Qaradagh soil attributed to its lower clay content, and cation exchange capacity, this was consistent with statistical analysis. Our results are in agreement with results reported by Motlagh, (2012) who reported that the k_p constant of the parabolic diffusion equation has a significant relationship with CEC at $P = 0.01$. In simple Elovich model, (α_s) and (β_s) constants

clarify the initial Cu release constant and Cu release rate coefficient, respectively (Boostani et al., 2016). In this study, (α_s) varies from 0.42 to 1.164 with a mean of 0.6856, and (β_s) from 2.197 to 5.952 with a mean of 3.6918. While (a) ranged between 0.055 and 0.444 with a mean of 0.2498 and, (b) ranges from 0.398 to 0.898 with a mean of 0.6056. The magnitude of the parameters, for the simple Elovich equation (α_s and β_s),

widely different for the five soils (table 5). Our results showed that according to the (α_s) constant for the simple Elovich model, the highest magnitude of initial copper released was recorded in Mawat soil and the lowest value attributed to Qaradagh soil, this confirmed by statistical analysis. The value of (α_s) constant for the simple Elovich equation relates significantly with some soil properties such as CEC, and soil pH. Our results are in agreement with (Ghasemi-Fasaei et al. (2007) who stated that the values of initial Cu release constant (α_s) and Cu release rate coefficient (β_s) were (0.73, and 2.76) respectively for the soil of southern Iran. The value of α_s , β_s is calculated for 30 minutes and the values shown in (table 5). The values of (α_s , β_s , t) for all the five soil more than one. Our results are in agreements with Dalal, (1985) who stated that simple Elovich equation derived from the Elovich equation with the hypothesis of (α_s , β_s , t) > 1.

Havlin and Westfall (1985); Jalali and Zarabi, (2006) classified both (a) and (b) as rate constants. To better understand the meaning of the constants (a) and (b) can be obtained

by relating the constants to the rate of nutrient release instead of cumulative release (Allen et al., 1996). The smaller the value of b , the more quickly the release rate decay.

The values of the rate constant (a and b) with the mean are (0.249 and 0.605) from the power function equation widely different from the five soil (table 5). Similar results were reported by

Ghasemi-Fasaee et al., (2006) who reported that means for a and b values were 0.24 and 0.282, respectively for copper release of highly calcareous soils from southern Iran release by DTPA in the studied soils.

Table 5. Values of the rate constants for the kinetics models that best described Cu

Soils	Parabolic diffusion	Simple Elovich			Power function		
	kp (mg Cu kg ⁻¹) ^{-0.5}	αs (mg Cu kg ⁻¹ h ⁻¹)	βs (mg Cu kg ⁻¹) ⁻¹	αs βs t*	a b (mg Cu kg ⁻¹ (h ⁻¹) ^b)	ab	
Sharazor	0.203	0.852	3.650	93.294	0.339	0.398	0.135
Qaradagh	0.123	0.240	5.952	42.854	0.055	0.808	0.044
Bazian	0.261	0.898	2.739	73.789	0.356	0.459	0.163
Mawat	0.321	1.164	2.197	76.719	0.444	0.465	0.206
Surdash	0.198	0.274	3.921	32.231	0.055	0.898	0.049
Min	0.123	0.24	2.197	32.231	0.055	0.398	0.044
Max	0.321	1.164	5.952	93.294	0.444	0.898	0.206
Mean	0.221	0.6856	3.6918	63.7774	0.249	0.605	0.119

* calculated for t = 30 min

The values of (ab) were calculated and listed for the studied soil in (table 5). Mawat soil had the highest value of (ab) constant equal to (0.206) soil and the lowest value attributed to Qaradagh soil about (0.044). This is confirmed by statistical analysis. This may be due to more content of clay and CEC in soil Mawat as compared to Qaradagh soil. Our results are consistent with the result of Ghasemi-Fasaee et al., (2007), who reported that CEC in soils with a high-level of carbonate soils likely to control the initial soil copper desorption rate.

CONCLUSIONS

The results of this study showed that the kinetics of Cu release using DTPA extracting solution in some calcareous soil from (0.5 to 48 hours) was rapid at the beginning then the reaction started with a constant slowdown and it arrived at equilibrium at 48 hours.

Parabolic diffusion was the best model based on the highest value of R², and the lowest value of RMSE, and AIC for the description of Cu release from some calcareous soil of Sulaimani governorate, Iraqi Kurdistan Region. The values of (kp), (αs, βs), and (a,b) for the parabolic diffusion, simple Elovich and power function models correlated with some soil characteristics like clay content and CEC in the studied soils.

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حركات تحرر النحاس من بعض الترب الكلسية في محافظة السليمانية بإقليم كردستان العراق

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المستخلص

أجريت هذه الدراسة لتقدير حركات تحرر النحاس من خمسة أنواع الترب الكلسية تشمل (شهرزور ، قرداغ ، بازيان ، ماوت، وسرداش) في محافظة السليمانية - إقليم كردستان العراق. كانت أكبر كمية من تحرر النحاس خلال 48 ساعة هي الأقل في تربة قرداغ (0.75 ملغم لكل كجم⁻¹)، في حين أظهرت تربة ماوت أعلى تحرر (2.272 ملغم لكل كجم⁻¹). تراوحت قيمة ثابت معدل الانتشار (kp) من الانتشار المكافئ من 0.123 إلى 0.321 بمتوسط (0.221). تراوح ثابت تحرر النحاس الأولي (α_s) ومعامل معدل تحرر النحاس (β_s) من نموذج Elovich البسيط ، من 0.42 إلى 1.164 بمتوسط 0.6856 ، ومن 2.197 إلى 5.952 بمتوسط 3.6918. في حين كانت قيم ثابت المعدل (أ و ب) من نموذج دالة القدرة مع الوسط (0.249 و 0.605) على التوالي على نطاق واسع مع التربة الخمسة. كان انتشار مكافئ أفضل نموذج مناسب تستخدم لوصف عملية تحرر النحاس بشكل جيد للغاية في التربة المدروسة.

الكلمات المفتاحية: النحاس ، تحرر النحاس ، معامل التحديد ، الحركية .