Kinetics of Phosphorus Adsorption in The Calcareous Soils of Kurdistan Region, Iraqi

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Abstract. Phosphorus is the second macronutrient which is vital for plants normal growth. In the calcareous soils, the adsorption is the main process that controls P availability to plants. This paper reviews the P adsorption phenomenon in the calcareous soils of Iraqi Kurdistan. In this study, the P adsorption capacity of the selected soils was determined by adding 10 mL of 0, 50, 100, and 200 mg P L-1 as (KH₂PO₄) to 1.0 g of soil and shaken for 12, 24 and 48 hours on horizontal shaker constantly. The suspensions at each period of incubation centrifuged and filtered then analysed for P which represented the equilibrium P concentration. The minimum adsorption 4.42±0.44 mg kg⁻¹ was recorded for soil4 with 50 mg P L⁻¹ applied solution and 12 h of incubation while the maximum adsorption 124±0.05 mg Kg⁻¹ was recorded for soil3 with 200 mg P L⁻¹ applied solution at 48 h of incubation. These results indicate that P adsorption increased with increasing applied amount of P and time of incubation. Results also showed that the Freundlich model was the best fitted according to the greater values of R² which was ranged from 0.7619 to 0.9984 compared to 0.738-0.9882 for Langmuir model and 0.7171-0.9986 for Temkin model. These results also suggest that in the studied soils P adsorbed through physical adsorption rather than chemical reaction and then decrease P availability in soil.

Key words: Phosphorus, Adsorption, Isotherms equation, calcareous soils, kinetics.

1. Introduction
Phosphorus (P) deficient soils are common and widespread in millions of hectares of cultivable land around the world. Therefore, this issue has been of great concern by many researchers [1];[2];[3]. After nitrogen, P has been considered as an essential macronutrient for plants productivity and growth and plays a vital role in the photosynthesis process. Phosphorus exists in about 370 minerals [4]. The P concentration in the soil is about 2000 times more than plants [5]. But only apatite group minerals such as fluorapatite [Ca₅(PO₄)₃F], hydroxyapatite [Ca₅(PO₄)₃(OH)] and chlorapatite [(Ca₅(PO₄)₃Cl)] contain an adequate amount of P to provide its mineralization [6]. Limited bioavailability of P in many soils is a serious nutrition restriction to the plant growth [7];[8]. In fact, adsorption and precipitation processes are mainly controlled P bioavailability in soil [9]. These processes led to insufficient about 80-90% of applied P to the soil, whereas the remaining amount as available is very low only about 10-20% as soon it is applied to the soil [10]. Phosphorus in the soil exists in many forms such as (i) fixed P, which refers to the portion that absorbed strongly or associated chemically the fraction of minerals, even sometimes be apportion of primary rock minerals. In short-term fixed P is a high insoluble form of soil P, only during weathering reactions might be converted to soluble form slowly; (ii) exchangeable or active P, is sorbed on the surface of active sites of the solid phase of soil and in equilibrium chemically with the dissolved fraction of P in soil solution. Whenever dissolved part reduced it might be available and (3) dissolved P, which is existed freely in soil solution and plants can absorb easily [10];[11];[12]. Generally, soil P can be divided into two types inorganic and organic. Availability of P to plant is depending on many various soil parameters including, amount of carbonate calcium, pH, OM (organic matter), moisture, amount and type of clay, CEC (cation exchange capacity), texture, root exudates and root density [13]. Calcareous soils have been considered as a limit factors that control the availability of nutrients as well as productivity [14]. Calcareous soils are recognized as soil that rich in free calcium carbonate (CaCO₃) and magnesium.
(MgCO₃), which are covered more than 30% of arable land worldwide. It is the dominant type of soil in many semiarid and arid regions which is affecting about more than 600 million of hectares' soils in the world (14). When P fertilizers applied to calcareous soils, it's solubility and availability to plants reduced as a consequence of serial fixation reactions. Most of them rapidly reaction with Ca²⁺ present in the soil solution to form Ca₅(PO₄)₃OH (hydroxyapatite), which called precipitated reaction. While small remain part of applied P has been adsorbed on the surface of clay minerals [15]. These continue reaction (precipitation and adsorption) of P in soil is recognized as soil P sorption [16];[17]. to control and design typical treatment processes of P insufficiency in soil, it is necessary to model P adsorption rate. This is determined to remain time needed to complete relevant reactions and to simulate the dynamic and kinetic attitude of metals on the adsorbents [18].

There are many different mathematical models developed to describe P adsorption in soils and most of them are depending on simple level constants to simulate P adsorption [19, 20, 21], such as Elovich equations [22]. Langmuir [19] and Freundlich [23]. In fact, to understand P dynamics in soil, the kinetics of adsorption is extremely important. This helps us for optimizing P fertilizer recommendation in crop production and establishment proper instruction for the elimination of wastes that rich in P [24]. There is an evident no or limited information about P adsorption kinetics in the literature regarding the soils of Erbil city and Sulaymaniyah provinces (Iraqi Kurdistan). The soils from this region are suffering from low available P, while still become one of the valuable areas for crops and vegetable production in Iraq. Therefore, this study aims to explain the kinetics of P adsorption in these two provinces calcareous soil and determine the best fit model among different adsorption isotherm models for describing the adsorption process.

2. Materials and Methods
2.1. Soil sampling
Our soil samples were collected from surface soils (0–30 cm deep) from two different characteristic ecological sites in the Erbil and Sulaymaniyah provinces, Iraqi Kurdistan. In each location, Oak forest and uncultivated locations were selected. The first site was Malakan in Erbil province (36°28'24.60''N, 44°33'44.40''E) and the second one was Badawan site in Sulaimanyah province (36°06'18.99'' N, 44°44'16.64''E). Three replicate soil samples were recovered randomly according to field trips for laboratory analysis. This region has a semi-arid climate. Between 70%-80% of the precipitation occurs between October and May, the mean annual air temperature ranges from 0 °C to 45 °C. Table 1 shows the sampling locations for four soils: Malakan oak forest soil (soil1), Malakan uncultivated soil (Soil2), Badawan oak forest soils (Soil3), Badawan uncultivated soil (Soil4) respectively.

2.2. Soil analysis
The soil samples were air dried and ground then pass through a 2 mm sieve for some chemical and physical analysis. The soil texture was found using the hydrometer method as explained by [25]. Soil pH and EC were measured from saturated paste extract by using (HANNA pH-meter, model pH21 and EC-Meter model CM-20S) respectively [26]. A modified Wakley and Black procedure were used to determine soil organic matter [27]. The cation exchange capacity (CEC) was determined by following the standard method (1M NH₄OAC) buffered at pH 7 as described by [28]. The acid neutralization procedure was applied to determine the amount of Equivalent calcium carbonate (ECaCO3) [29] and available P was found by sodium bicarbonate extraction (Olsen method) as explained by Estefan et al., [30].

2.3. Adsorption isotherm experiment
Time-dependent adsorption isotherm experiment was achieved operating three replicates from each of the four soils equilibrated in laboratory condition to examine P adoption. About 1.0 g soil samples of all soil were placed into 25 mL Plastic centrifuge tubes and then P solution, containing 0, 50, 100, and 200 mg P L⁻¹ as (KH₂PO₄) in 0.01 M CaCl₂, were added to the tubes. The samples in centrifuge tubes were shaken on a reciprocating shaker for 12, 24 and 48 hours at room temperature 20±20°C for 12, 24 and 48 hours. Periodically, following the suspensions samples were centrifuged and filtered. The filtrates were analyzed for solution P calorimetrically by spectrophotometry as defined by [31]. adsorbed P was calculated in the
normal method by subtracting the P residual in solution from the initial P and supposing the difference was the adsorbed P. The zero P solution was used as control P for the experiment and these values were subtracted from the others to accurate for the P that was released from the control soil.

2.4. Adsorption Isotherm Models

The adsorption isotherm models can be used to determine the distribution of P between solid and liquid phases through linear function [32]. So, in this study, the three widely used models, Freundlich, Langmuir and Temkin adsorption isotherm models in linear forms were employed to investigate P adsorption in the studied soils. The general forms of the three used models are presented in equations [1]; [2];[3].

2.5. Freundlich isotherm model

\[ q_{ads} = K_f (C_{sol})^n \]  \hspace{1cm} (1)

Where

- \( q_{ads} \) is the P adsorbed of soil (mg kg\(^{-1}\)), \( C_{sol} \) is the equilibrium concentration of P in soil solution (mg L\(^{-1}\)), \( K_f \) is the proportionality constant for Freundlich model (mg kg\(^{-1}\)), and \( n \) is the empirical constant related to bonding energy of soil for phosphate,

2.6. Langmuir isotherm model

\[ q_{ads} = \frac{K_L \cdot b \cdot C_{sol}}{1 + K_L \cdot C_{sol}} \]  \hspace{1cm} (2)

Where

- \( q_{ads} \) is the P adsorbed of soil (mg kg\(^{-1}\)), \( C_{sol} \) is the equilibrium P concentration in soil solution (mg L\(^{-1}\)), and \( b \) and \( K_L = \) Langmuir constants; \( b \) is the maximum adsorption capacity of the soil (mg kg\(^{-1}\)). \( K_L \) is correlated to binding energy of P to the soil. And also, the adsorption factor (RL) was achieved from equation 3.

\[ R_L = \frac{1}{1 + K_L \cdot C_{sol}} \]  \hspace{1cm} (3)

2.7. Temkin isotherm model

\[ q_{ads} = a + b \ln C_{sol} \]  \hspace{1cm} (4)

Where

- \( q_{ads} \) is the P adsorbed of soil (mg kg\(^{-1}\)), \( a \) is the amount of P adsorbed in Temkin model (mg P kg\(^{-1}\)), \( b \) is the buffer capacity of Temkin model (mL g\(^{-1}\)), and \( C_{sol} \) is the equilibrium P concentration in soil solution (mg L\(^{-1}\)).
2.8. Statistical analysis

The coefficient of determination (R^2) and standard error (SE) were dependent to select the best model to define the adsorption phenomena was determined using excel 2016 software.

3. Results and Discussion

3.1. Soil physiochemical properties

The results of physicochemical properties of selected soils are summarized in Table (1). Soil pH ranged from 7.39–7.66 with high total CaCO_3 content between 9.80-30.7% g kg^{-1} and low organic matter content between 20.4-32.8% of selected soils reflecting the calcareous nature of the studied soils. Soil pH was positively correlated with CaCO_3 content (R^2=0.75). Soil Electrical Conductivity (EC) ranged between 0.27-0.36 dS m^{-1} indicating no saline soils. The low value of EC may be due to the quality of rainwater in the area which contains a low amount of salts. Also, it may be due to the low rainfall in these areas. The high value of CEC between 56.0-44.4 Cmole kg^{-1} was associates with soil that has the highest content in clay particles between 432.4 to 490.1 g kg^{-1}. Relatively amounts of available P as (Olsen-P) were found in all selected soils indicating high adsorption capacity of soils. And also, maybe due to low application of P fertilizer in these areas. It is clear that clayey is the dominant soil texture with one soil sample had silt clay loam texture which collected in Malakan oak forest area.

Table 1. Selected physiochemical characteristics of studied soils

| Soil type                        | Code | pH   | EC  | OM  | CaCO_3 | CEC  | Olsen-P | Clay  | Silt  | Sand  | Texture class |
|---------------------------------|------|------|-----|-----|--------|------|---------|-------|-------|-------|---------------|
| Malakan oak forest soil         | Soil1| 7.46 | 0.36| 20.4| 128.1  | 46.0 | 7.96    | 463.3 | 346.2 | 190.4 | SiCL          |
| Malakan uncultivated soil       | Soil2| 7.39 | 0.34| 28.1| 98.0   | 44.4 | 5.67    | 432.4 | 497.7 | 69.90 | C             |
| Badawan oak forest soil         | Soil3| 7.58 | 0.27| 32.8| 307.1  | 56.0 | 7.43    | 490.1 | 265.5 | 244.5 | C             |
| Badawan uncultivated soil       | Soil4| 7.66 | 0.32| 24.4| 251.1  | 50.7 | 4.80    | 437.4 | 300.1 | 262.5 | C             |

note: C= Clay, SiCL= Silty clay loam

3.2. Phosphorus adsorption

The term of adsorption is used to explain the all methods that relate to removing of P in soil solution which is mainly by surface adsorption and precipitation [33, 34]. The adsorption amount of P for all the studied soils is determined by extracting the remaining amount of P in solution from the applied amount. Comparing the highest amount of P adsorbed in these soils, it was observed that each soil has a different capacity to P adsorption. The P adsorption onto all the studied soils at different P concentrations and time of incubation is presented in (Table 2). The P adsorption increased significantly with the increase of solution P concentration and time of incubation regardless of soil type. In this study, the 200 mg L^{-1} P concentration showed the highest and 50 mg L^{-1} showed the lowest P adsorption in all the studied soils. Joardar et al., [35] reported that P adsorption is significantly increased with P concentration in calcareous soils. Asomaning, [34] reported that P adsorption increases with increase in the time of reaction. The main reason may be due to the presence of an extreme quantity of CaCO_3 content of the studied soils which is very active to P adsorption. In addition to P concentration, adsorption was also influenced by clay content and soil pH. In the study, the pH of the soil is quite high and it was assessed that in pH value more than 7, applied P can react with Ca++ to form Ca-P mineral [35]. In the case of soil texture, all the studied soils contain a high amount of clay fraction which was had a great effect on P adsorption. Khan [36] reported after comparing the three soil series that the highest adsorbed P was recorded with soil series that has the highest clay content. Also, Hanyabui [33] studied phosphorus adsorption in tropical soil
and found that in alkaline soils electrical potential of clay surface was the main factor for phosphorus adsorption. Saki [32] reported that high P adsorption by soil that contains a high amount of clay is related to high surface area and soil buffering capacity.

### Table 2. The Amount of P Adsorbed (mg kg⁻¹) in all the studied soil and during 12h, 24h, and 48h.

| Time (h) | Applied P (mg L⁻¹) | Soil1 Adsorbed P ±SD (mg kg⁻¹) | Soil2 Adsorbed P ±SD (mg kg⁻¹) | Soil3 Adsorbed P ±SD (mg kg⁻¹) | Soil4 Adsorbed P ±SD (mg kg⁻¹) |
|---------|---------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| 12      | 50                  | 31.1±5.02                     | 28.6±0.76                     | 12.6±0.11                     | 4.42±0.44                     |
|         | 100                 | 45.2±2.51                     | 37.9±1.42                     | 14.5±2.73                     | 14.8±0.55                     |
|         | 200                 | 97.0±2.51                     | 90.8±0.71                     | 41.2±5.40                     | 42.7±3.11                     |
| 24      | 50                  | 40.6±0.05                     | 38.7±1.31                     | 22.0±1.42                     | 17.9±3.49                     |
|         | 100                 | 60.5±1.09                     | 59.0±2.62                     | 49.4±0.55                     | 23.9±2.18                     |
|         | 200                 | 108±1.91                      | 103±2.02                      | 97.8±3.33                     | 85.0±0.60                     |
| 48      | 50                  | 40.0±3.44                     | 40.3±0.60                     | 30.6±0.33                     | 21.6±1.85                     |
|         | 100                 | 72.1±1.04                     | 68.2±1.64                     | 68.1±0.44                     | 48.8±0.11                     |
|         | 200                 | 120±5.51                      | 117±0.05                      | 124±0.05                      | 89.2±0.11                     |

#### 3.3. Adsorption isotherms

The Freundlich, Langmuir and Temkin adsorption isotherm models were used to modelling P adsorption in all the studied soils using linear forms of equations. The constant P adsorption across time should produce an advanced change in the adsorption isotherm. The consideration values processed employing the isotherms models and the coefficient of determination presented in table (3). All three models were efficient to explain the experimental data. But, results showed that the Freundlich model was the best-fitted model according to the greater values of the coefficient of determination (R²) which was ranged from 0.7619 to 0.9984 compared to (0.738-0.9882) for the Langmuir model and (0.6820.9986) for Temkin model as shown in Table (3), which achieved from their equations developed from Figure (1, 2, 3). As expected, the Freundlich adsorption capacity, KF of soil between (4.47-15.2 L kg⁻¹) is larger compared to 1.54-2.35 L kg⁻¹ for soil 2, 6.73-10.2, L kg⁻¹ for soil3, and 0.69-1.06 L kg⁻¹ for soil4. This result is in agreement with Khan et al., (36) who found that Freundlich model was the best fitted for P adsorption in the studied soils. Furthermore, Mbene et al., (37) studied P fixation and found that Freundlich model was described P adsorption very well in the calcareous soil. This hypothesis can more be approved established on the present of points in Figure (1) for Freundlich and (Figures 2, 3) for Langmuir and Temkin plots. Figures 2 and 3 show that the linearized data fitting to Langmuir and Temkin model created curves than straight lines and this were conflicting to Figure 1 where the linearized data according to Freundlich, all the studied soils presented lines. Furthermore, a higher variability of parameters related to binding energy for both the Freundlich and Langmuir models proposing different mechanisms of P adsorption. High KF value more than 0.4 suggests a large role of precipitation in the adsorption process [38].
Table 3. The value of parameter constants of selected adsorption isotherm models in the studied soils.

| Soil  | Time (h) | Freundlich Isotherm | Langmuir Isotherm | Temkin Isotherm |
|-------|----------|---------------------|-------------------|-----------------|
|       |          | a                   | Kf (mg kg⁻¹) | n               | R² | SE | KL (mg L⁻¹) | RL | R² | SE | AT (mg L⁻¹) | bt | R² | SE |
| 1     | 12       | 1.58                | 4.47           | 0.8879          | 0.120          | 102 | 0.02 | 0.18.0.47     | 0.8623 | 0.006 | 0.11 | 69.3 | 0.796 | 22.18 |
|       | 24       | 2.42                | 15.2           | 0.9388          | 0.075          | 99.0 | 0.07 | 0.07-0.22     | 0.8836 | 0.004 | 0.37 | 89.0 | 0.865 | 17.92 |
|       | 48       | 1.92                | 12.4           | 0.9982          | 0.015          | 147 | 0.04 | 0.11-0.34     | 0.9923 | 0.001 | 0.28 | 65.5 | 0.987 | 6.436 |
| 2     | 12       | 1.54                | 1.54           | 0.7993          | 0.166          | 87.0 | 0.02 | 0.19-0.48     | 0.759  | 0.008 | 0.09 | 72.3 | 0.717 | 25.24 |
|       | 24       | 2.35                | 2.35           | 0.9741          | 0.047          | 98.0 | 0.06 | 0.08-0.26     | 0.9261 | 0.003 | 0.32 | 91.9 | 0.919 | 12.47 |
|       | 48       | 2.12                | 2.12           | 0.9984          | 0.013          | 125 | 0.05 | 0.09-0.29     | 0.9739 | 0.002 | 0.31 | 74.7 | 0.966 | 9.693 |
| 3     | 12       | 0.83                | 6.73           | 0.9584          | 0.105          | -101 | 0.00 | 1.09-1.46     | 0.9883 | 0.009 | 0.03 | 105 | 0.844 | 9.662 |
|       | 24       | 0.87                | 7.46           | 0.9903          | 0.045          | -213 | 0.00 | 1.21-1.14     | 0.9875 | 0.003 | 0.05 | 42.1 | 0.988 | 5.906 |
|       | 48       | 1.01                | 10.2           | 0.9467          | 0.100          | -455 | 0.00 | 1.21.13.15 | 0.9499 | 0.004 | 0.08 | 36.3 | 0.999 | 2.468 |
| 4     | 12       | 0.69                | 0.69           | 0.9685          | 0.105          | -18.6 | 0.00 | 0.13-0.78     | 0.9575 | 0.030 | 0.03 | 121 | 0.989 | 1.976 |
|       | 24       | 1.06                | 1.06           | 0.7619          | 0.225          | 87.7 | 0.01 | 1.37-2.48     | 0.7388 | 0.016 | 0.04 | 67.8 | 0.682 | 24.46 |
|       | 48       | 0.97                | 0.97           | 0.9739          | 0.071          | -333 | 0.00 | 0.56-0.89     | 0.9775 | 0.004 | 0.05 | 49.8 | 0.999 | 1.771 |

Figure 1. Linear form of Freunlich adsorption isotherm for all the studied soils

In the case of Langmuir model, the KL values ranged between 0.02-0.07 mg L⁻¹ for soil1, 0.020.06 for soil2, 0.00 for soil3 and 0.0-0.01 for soil4 Table (2) and Figure (2), which suggests the favorable condition of P adsorption by soils. The frequent KL is the similarity between soil colloids and P ions, which is a strength factor of soil adsorption [39], indicating the level of P adsorption of soil to a confident
amount. A positive K-value shows that the adsorption reaction is spontaneous at room temperature, and the significance of KL shows the spontaneous extent of the adsorption reaction. The larger the K-value, the greater the spontaneity and the more constant the products are, leading to weak P supply [40]. The KL-values were positive in all applied P concentrations and incubation times, indicating that the adsorption reactions were all spontaneous, the KL-value was higher for the soils from Erbil province compared with the soils form Sulaimanyah province, and no significant difference was detected among all the studied soils suggesting that the soil P adsorption was increased with increasing in the applied P rate and time of incubation. The adsorption capacity (b) ranged from 99.0 to 147 mg kg-1 regardless of the time of incubation. The RL ranged between 0-1 for soil 1 and 2 but increased to above 1 for soil3 and soil4 at 24 h of incubation. Moreover, the RL values indicate that the adsorption of P was more variable for lower concentration and time of incubation than high concentrations and time of incubations. This is maybe due to a decrease in the soil surface capacity to adsorb P at increasing phosphate concentration. This phenomenon is related to the saturation of high attraction binding sites of soil particles and rises in electrostatic barriers as negatively charged phosphates fix to the soil surface [32].

Figure (2) Linear form of Langmuir Adsorption isotherm for all the studied soils.

Moreover, the adsorption of P decreased with increasing OM content in all the studied soils. This is may be due to decrease in the surface charge of soil particles. Asomaning [34] reported that OM decrease P adsorption by two ways, first by reducing iron oxide crystallization and second by competing adsorption sites in the soil. But increased with increasing CaCO3 content. This is may be due adsorption
and reaction processes caused by CaCO3 in soils. Joarder et al. [35] studied P sorption in calcareous and non-calcareous soils and found that P sorption in calcareous soil was higher than non-calcareous soil. And also, the Temkin parameters showed in (Table 3) and (Figure 3). The minimum value of AT was 0.03 L mg-1 for soil4 at 12 h incubation time but the maximum value was 0.37 L mg-1 for soil1 at 24 h of incubation time. These results indicate physical adsorption of P by soil particles. The coefficient of determination (R2) was ranged from 0.7171 to 0.9986 for all the studied soils. It was found that the Temkin equation did not show a good fit to the data of the four studied soils compared to another model. The bt parameter in the Temkin the equation was larger in all the four soils, as compared with the other equation with values ranged from 42.1 to 121 regardless of soil type and time of incubation.

4. Conclusion
Time-depend adsorption behavior of P was investigated in this study for four soils, collected from two different geological sites in Iraqi Kurdistan. The results showed that P adsorption increased with the applied amount of P and time of incubation. Maximum adsorption was found with the highest applied P concentration and after 48h of incubation. This is indicating that all the studied soil had a high capacity to absorb applied P and not reached equilibrium. Furthermore, the oak forest soils adsorbed more P than uncultivated soils. And also, the Freundlich isotherm model was more suitable for describing the adsorption behavior for P in all the studied soils. The modles also better fitted to the oak forest soils than uncultivated soils. Results also indicate that presence high amount of calcium carbonate in the studied soil

Figure 3. Linear form of Temkin adsorption Isotherm for all the studied soils.
caused increase in the amount of adsorbed P. The study found a widespread range in P adsorbed on the studied soils. There is requirement for further study to recognize the responsibility and relations of soil properties on P adsorption for each site before fertilizer use.

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