



# Solubility of Iron (Fe II) in the Long-Term Vegetable Growing in Calcareous Soils

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## ABSTRACT

In the calcareous soils, iron solubility is very limited because of the high pH and calcite values. In this study, the solubility and partition coefficient iron in the variety of vegetable cultivated calcareous soils from Sulaymaniyah province, Iraqi Kurdistan, was studied. The objective of the current study was to determine the Fe mineral that controls the solubility of iron and the range of iron adsorbing in these soils. A total of 21 soil samples were incubated for 56 days, and periodically at (1, 7, 14, 28, and 56), the soluble iron was determined. The main results indicated that the solubility of iron in the studied soils was saturated to siderite in all the studied soils and incubation period with slight fluctuation. Soluble iron is negatively correlated with total Fe in soil and pH values but highly correlated with dissolved organic carbon and total dissolved carbon. The  $K_d$  values low which ranged from  $0.11 \pm 0.04$  to  $1.90 \pm 0.03$  for iron was recorded indicating low adsorption capacity for iron by soils this is because the presence of the high amount of the organic matter in the studied soils. A high correlation was found between Log  $K_d$  values with dissolved organic carbon and total dissolved carbon in all soils. From the results, it is concluded that siderite is the dominant iron mineral in Iraqi Kurdistan calcareous soils and soil organic matter has the main role in increasing Fe availability.

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Keywords: Iron status, Solubility, Iron minerals, Availability, Partition coefficient.

## 1. Introduction

### Background

In the earth's crust, Iron (Fe) is the fourth most abundant element<sup>[1]</sup>. However, Fe concentration in the natural soil ranged from 7 to 42 g kg<sup>-1</sup><sup>[2]</sup>. Iron is generally represented in primary minerals and several phyllosilicates in its ferrous (Fe<sup>II</sup>) state<sup>[3]</sup>. They reported four main Fe groups in soil, including (i) Fe<sup>2+</sup> in primary minerals, (ii) Fe<sup>3+</sup> in secondary minerals (crystalized and (hydro) oxides Fe), (iii) exchangeable and soluble Fe, and (iv) Fe bounded to the organic matter in insoluble and soluble forms. But, calcareous soil is geologically present in arid and semi-arid regions because of minor leakage<sup>[4]</sup>, which covers about 30% of the earth's surface, and low solubility of Fe reduces plant growth and reproduction<sup>[5, 6]</sup>.

Plants growing deficiency called 'Iron chlorosis' occurs in this type of soil even though the total amount of Fe in this type of soil is higher<sup>[7]</sup> than the soluble amount of Fe essential for normal plants growth, which is about  $10^{-8}$  M in the soil solution<sup>[8]</sup>.

Iron availability and solubility are very limited due to some soil conditions, such as high pH value, soil redox, and low organic matter in calcareous soil<sup>[2, 9, 10]</sup>. Moreover, in this type of soil, due to high CaCO<sub>3</sub>, active CaCO<sub>3</sub> and clay contents with high surface area, are the main factors that controls Fe fixation<sup>[11]</sup>. The soil solution Fe decreases due to the adsorption of Fe on the CaCO<sub>3</sub> surface, clay minerals, and Al/Fe-oxides<sup>[12]</sup>. At the pH range between 7.4-8.5, the inorganic form of Fe is at minimum concentration, and solubilization of Fe is a slow process regulated by dissolution-precipitation phenomena and the pH of Fe minerals<sup>[3]</sup>. Chen and Barak<sup>[13]</sup> reported that in the pH range of 7-9 the Fe species of Fe(OH)<sub>2</sub><sup>+</sup>, Fe(OH)<sub>3</sub><sup>0</sup>, and Fe(OH)<sub>4</sub><sup>-</sup> are the most abundant inorganic Fe species in soil solution, which does not exceed  $10^{-10}$  M Fe L<sup>-1</sup>, but Kumar *et al.*<sup>[12]</sup> reported that at high pH values, Fe precipitates as insoluble amorphous and FeSiO<sub>4</sub>, which reduces Fe availability. Furthermore, soil properties such as soil texture, chelating agents, and time which mainly limit the availability<sup>[14]</sup>. However, Priyadarshini *et al.*<sup>[9]</sup> reported that organic manure is beneficial to improve Fe availability in soil. Kosegarten and Koyro<sup>[8]</sup> reported that Fe solubility increased in the rhizosphere due to the production of the chelating compound by micro-organisms in additional to root exudates.

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A possible method to evaluate the soluble Fe in soil environments would be the study of the change in total Fe concentration and the minerals that control Fe solubility over time<sup>[14]</sup>. The current study explores the impact of incubation time, soil pH value, CaCO<sub>3</sub> content, and soil organic matter content on Fe solubility in vegetable grown calcareous soils in Iraqi Kurdistan. Thus, the aim of this work is to (i) Identify mineral that control over Fe solubility, (ii) Estimate the impact of incubation time on Fe solubility, and (iii) Determine the availability and capacity of the studied soil to adsorb Fe through solid/liquid partition coefficient.

## 2. Methods and Materials

### Sample collection and preparation

A total of 21 soil samples were collected from different sites at (0-30 cm) depth which was cultivated with vegetables in Sulaymaniyah province. The samples were transported to the laboratory and dried at room temperature for three days then sieved with a 2 mm stainless sieve. The samples were kept in a 100 mL universal tube for further analysis.

### Soil chemical analysis

The pH value of the soil was determined by soil-distilled water suspension (1:2.5 soil-water ratio) using the method reported by<sup>[15]</sup>. Soil organic matter was determined following the procedure reported by<sup>[16]</sup>. The soil calcite content (total CaCO<sub>3</sub>) was determined using the Collins Calcimeter method<sup>[17]</sup>. The extractable Fe determined followed the procedure reported by<sup>[15]</sup>.

**Table 1:** Iron mineral phases, reaction, and K<sub>sp</sub> used in the constructing of a ferrous solubility.

Mineral	Formula	Equation	K <sub>sp</sub>
Iron oxide	FeO	FeO + 2H <sup>+</sup> ----- Fe <sup>+2</sup> + H <sub>2</sub> O Log Fe <sup>+2</sup> = 13.48-2pH	13.48
Iron hydroxide	Fe(OH) <sub>2</sub>	Fe(OH) <sub>2</sub> + 2H <sup>+</sup> ----- Fe <sup>+2</sup> + 2H <sub>2</sub> O Log Fe <sup>+2</sup> = 12.9-2pH	12.9
Siderite	FeCO <sub>3</sub>	FeCO <sub>3</sub> + 2H <sup>+</sup> ----- Fe <sup>+2</sup> + CO <sub>2</sub> (g) + H <sub>2</sub> O Log Fe <sup>+2</sup> = 7.92-2pH	7.92

### Statistical analysis

SPSS software (version 26) for Windows and Microsoft Excel (2016) was used to make standard statistical tests through analysis of variance (one-way ANOVA). The Least Significant Difference (LSD) was operated to determine the significant difference between samples under (p≤0.05).

## 3. Results and Discussion

### General soil characteristics

The essential data for pH, OM, calcium carbonate, and elemental concentration in the studied soil are summarized in (Table 2). The

**Table 2:** Descriptive statistical associations for soil pH, OM, CaCO<sub>3</sub>, and total metal concentrations (n=21).

parameters		Average	Median	Minimum	Maximum	±SD
pH		8.03	8.13	7.33	8.27	0.22
OM	%	8.91	8.80	4.80	12.0	1.99
CaCO <sub>3</sub>		25.5	27.1	5.58	41.0	10.7

The DOC and TDC in the filtered soil solution were determined using the shimadzu TOC-V CPA/CPN Total Organic Carbon analyzer (Model TNM-1). Soil multi-element analysis determined followed the procedure reported by<sup>[18]</sup> using ICP-MS at the University of Nottingham, UK.

### Iron solubility experiment

A duplicate of 21 soil samples was used to determine Fe solubility by incubating 2 g of each soil at 50% water holding capacity at room temperature (20±2) °C for a period time of 56 days. At days 1, 7, 14, 28, and 56, the solubility of Fe was considered by suspending 2 g of samples in 20 mL of 0.01 M calcium nitrate and shaking for two days, followed by centrifugation (2500 rpm for 15 min) filtered with 0.22µm filter and acidification of a 5 mL aliquot with 0.2 mL 50% nitrate for measurement of Fe by ICP-MS. A split aliquot of the un-acidified supernatant solution was used to determine pH and total dissolved carbon (TDC) and dissolved organic carbon (DOC).

### Iron solubility

Log-free Fe<sup>2+</sup> ion activities versus hydrogen potential (pH) were used to create the solubility diagram for Fe minerals. It was expected that Fe<sup>2+</sup> activity was regulated by pH established on distributed K<sub>sp</sub> values for Zn minerals is expected that Fe solubility in the calcareous soils is regulated by the phase of Fe minerals presented in (Table 1)<sup>[19]</sup>.

studied soils were slightly alkaline since their pH values ranged between 7.33 and 8.27. The results indicated that the all studied soils were varied from calcareous soil because their calcium carbonate contents ranged between 5.58% and 41.0%. The soil organic matter content ranged from 4.80% to 12.0%. The average ratio of macro in (g kg<sup>-1</sup>) and micronutrients in (mg kg<sup>-1</sup>) in all studied soils were 14.7 (Mg), 102 (Ca), 9.19 (K), 26700 (Fe), 633 (Mn), 14.5 (Co), 97.0 (Ni), 24.3 (Cu) and 93.1 (Zn), suggesting that all in the range reported for calcareous soils.

<b>Mg</b>	g kg <sup>-1</sup>	14.7	16.0	6.86	21.3	3.89
<b>Ca</b>		102	107	39.0	162	38.2
<b>K</b>		9.19	8.81	5.56	12.7	2.03
<b>Fe</b>		26.7	25.6	19.5	36.4	5.57
<b>Mn</b>	mg kg <sup>-1</sup>	633	579	309	960	183
<b>Co</b>		14.5	14.2	8.58	19.8	3.43
<b>Ni</b>		97.0	105	44.6	140	29.3
<b>Cu</b>		24.3	25.2	16.9	35.8	4.59
<b>Zn</b>		93.1	92.9	43.4	187	37.1

\*SD = Standard deviation

### Iron solubility in soils

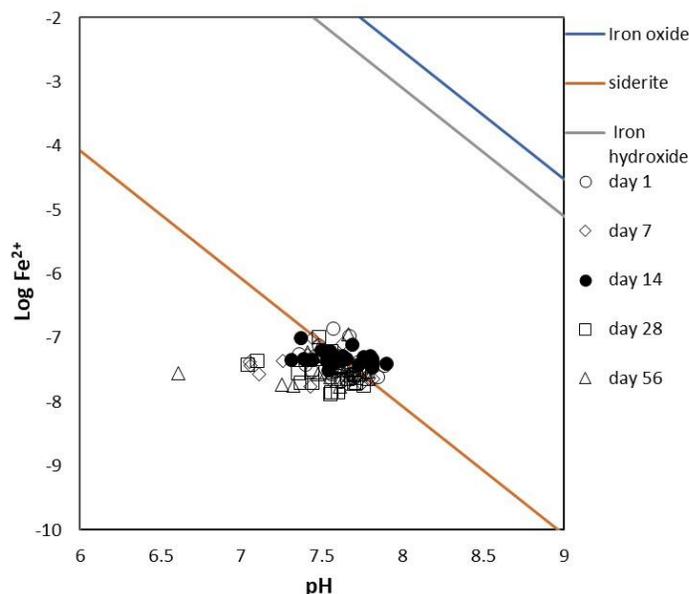
The Fe solubility is generally low in calcareous soils because of rapid precipitation<sup>[20]</sup>. The initial mineral Fe formed in calcareous soil is iron oxide from the original iron hydroxide, which is then converted to siderite<sup>[3]</sup>. A stability diagram for Fe minerals equilibria was developed by plotting Fe potential (-log Fe<sup>2+</sup>) against soil hydrogen potential (pH). The activity of Fe<sup>2+</sup> and H<sup>+</sup> (pH) used to develop this diagram, were determined using the measured value of Fe in soil solution followed by speciation using WHAM-7. This approach can indicate the likelihood of specific Fe minerals being presented in the soil at different pH values. It can be used to estimate Fe<sup>2+</sup> activity maintained by several minerals as a function of pH, CO<sub>2</sub> (g), and redox<sup>[21]</sup>.

Figure (1) shows the soil solution composition with common Fe minerals in the studied soils. The Fe data fell undersaturated with respect to iron hydroxide and saturated with respect to siderite across all the studied soil and in all incubation periods (1-56 days). The results indicate that siderite (FeCO<sub>3</sub>) could be considered of as the main solid phase governing Fe solubility in the studied soils. A similar result was reported by<sup>[22]</sup>. Results in the current study also indicate that a siderite mineral is a stable form of Fe mineral in the studied soils. This agrees with the results report by<sup>[21]</sup>, who found that the siderite mineral is the main Fe mineral that controls Fe solubility in agricultural calcareous soils.

The minerals of Fe transformed to a more stable form in the presence of high amounts of calcium carbonate and high pH value in the calcareous soil. This indicates that Fe solubility in the studied soils is low; this may be due to high pH values ranging from 7.33 to 8.27 in the studied soils. Lindsay and Schwab<sup>[21]</sup> reported that in the pH range between 7.4 and 8.5, the Fe oxides achieve their minimum solubility. They also reported that Fe uptake is linearly correlated to Fe<sup>2+</sup> in soil solutions above 10<sup>-9.2</sup> M. Thus, the Fe<sup>2+</sup> form is the major source of plant Fe uptake in the studied soils because the Fe<sup>2+</sup> in the soil solution in the current study remains above 10<sup>-8</sup> M all over the incubation time (1-56 days) (Fig. 1). It is also can be concluded that in the studied soil Fe deficiency slightly occur due to remain of Fe<sup>2+</sup> activity slightly under level of 10<sup>-7.7</sup> M, this is because this level is critical. However, the actual critical level differs depending on some reasons including plant species, presence high amounts of chelating agents, acidifying effects of roots, reductive capacities

of roots. Moreover, release Fe<sup>2+</sup> oxides to Fe<sup>3+</sup> and precipitate as secondary Fe<sup>3+</sup> oxides because, since the Fe<sup>2+</sup> minerals are mostly variable in soil and slow weather in the presence of atmospheric oxygen<sup>[23]</sup>.

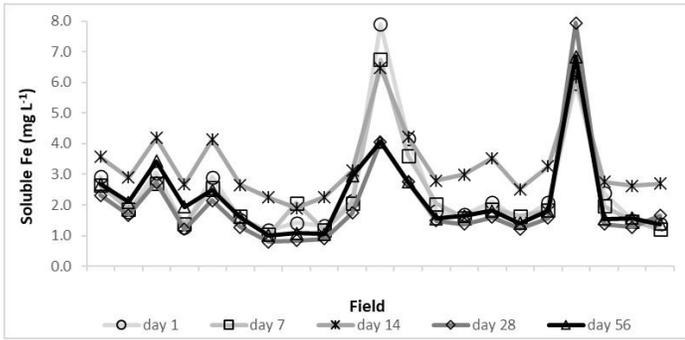
Chen and Barak<sup>[13]</sup> reported that organic matter application to soil causes an increase in the water-soluble Fe in soil solution. Organic acid and complex polymers can form soluble complexes



**Figure 1:** Iron (II) solubility in the studied soils about crystal Fe minerals; iron oxide, FeO; siderite, FeCO<sub>3</sub>; and iron hydroxide Fe(OH)<sub>2</sub>: Equilibrium lines for mineral phases were calculated based on the Ksp values reported by (Lindsay, 1979). All 21 soil samples were incubated for (○) 1 day, (◇) 7 days, (●) 14 days, (□) 28 days, and (△) 56 days.

with Fe<sup>2+</sup> or act as chelating agents and increase the availability of Fe<sup>[5]</sup>.

The soluble Fe from incubated soils is presented in (Fig. 2). Although the data were slightly affected by incubation time and the change was insignificant. During incubation, the amount of soluble Fe remained stable over incubation time. Especially, at day 14, which was the soluble amount of Fe increased and then decreased at days 28 and 56. The stability of soluble Fe overall incubation time indicates the equilibrium achieved.

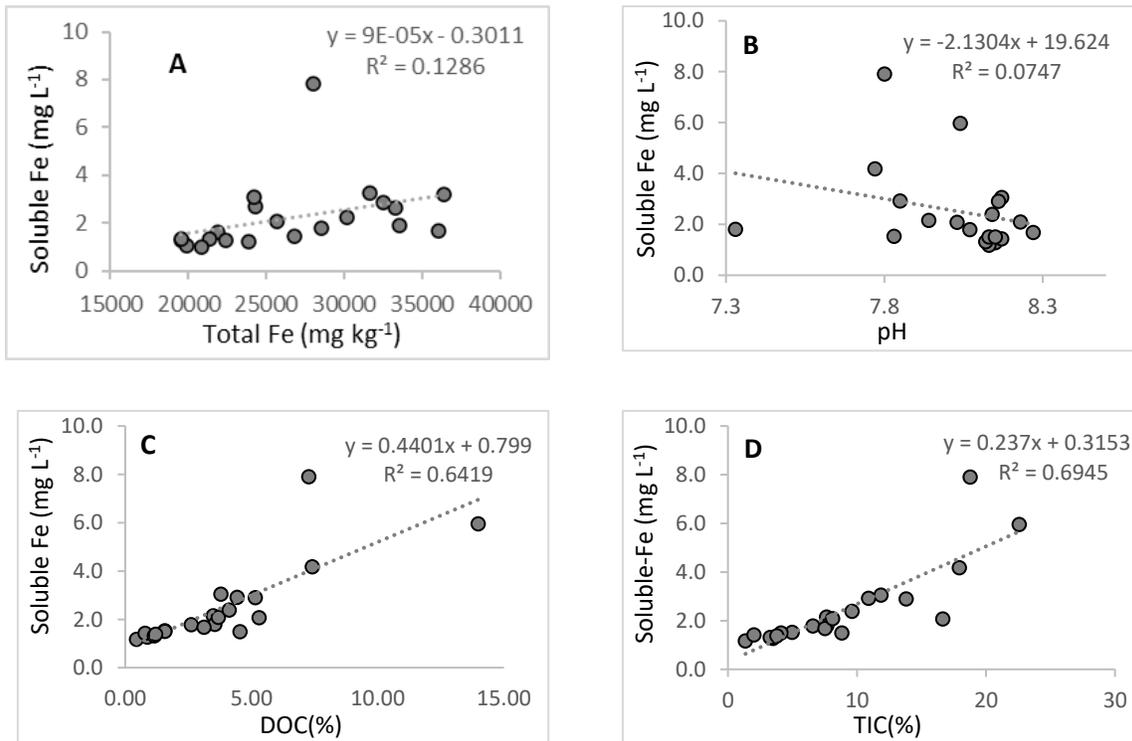


**Figure 2:** Average variation in soluble Fe ( $\text{mg L}^{-1}$ ) throughout the incubation for 1, 7, 14, 28, and 56 days.

Soluble Fe of the soils correlated negatively with total Fe content but negatively with soil pH (Fig. 3A and B). The above results suggest that Fe solubility decreased with increasing total Fe and pH value of the soils portably due to the adsorption of Fe by clay minerals and Fe/Al oxides and formation of ferric minerals due to aeration in soil, and it causes to reduce the amount of soluble Fe. A similar result was reported by<sup>[12, 24, 25]</sup>. Chen and Shenker<sup>[26]</sup> reported that the most important soil characteristic is pH which reduces Fe solubility in soil. The increase in the value of soil pH causes a decrease in soil solution Fe; this is due to the adsorption of Fe ion by  $\text{CaCO}_3$ , Fe/Al oxides, organic matter, and clay minerals<sup>[12]</sup>. The amount of soluble Fe increases in the soil solution by decreasing soil pH<sup>[3, 20]</sup>. A negative correlation

between soluble Fe and total Fe content in selected soil may be returned to that in calcareous soils in the presence of oxygen; most of Fe is in the form of Insoluble-Fe and also oxidizing to  $\text{Fe}^{+3}$  form.

Moreover, soluble Fe significantly and positively correlated with DOC and TDC (Fig. 3c and d). The results suggest that the soluble Fe complexes form due to the reacting of each DOC and TDC with Fe; this is cause inhibits the Fe from fixation or precipitation components. Najafi-Ghiri et al.<sup>[28]</sup> indicated that more than 90% of soluble Fe was fixed by organic complexes. While, Lindsay<sup>[22]</sup> believes that organic matter influences Fe solubilization throughout  $\text{Fe}^{+3}$  reduction to  $\text{Fe}^{+2}$ ; this is due to the decomposition of organic matter. A sufficient amount of soluble Fe in selected soils may be due to the presence of moderate to high amounts of inorganic and organic carbon in these types of soils, as shown a significant positive relationship between soluble Fe and DOC and TIC levels in the studied soils. Hochmuth<sup>[5]</sup> reported that in soil solution, Fe might present in its reduced state ( $\text{Fe}^{+2}$ ) due to the presence of organic matter in the soil. This also suggests that during organic matter breakdown and other inorganic carbon sources react with Fe and form soluble Organic-Fe and adsorbing Fe by inorganic carbon, which prevents the Fe from precipitation. Ahmad et al.<sup>[14]</sup> reported that the solubility of Fe increases due to the chelates of Fe by various organic complexes. However, a negative correlation was found between soluble and total Fe in all 21 studied soils.



**Figure 3:** Correlation between (A) soluble Fe and total Fe, (B) soluble Fe and soil pH, (C) soluble Fe and DOC, and (D) soluble Fe and TDC in all 21 studied soils.

**Available Fe and Solid-liquid partition coefficient**

Most soils have adequate quantities of Fe to suffer plant needs, but its availability depends on chemical and environmental factors<sup>[20]</sup>. The average available Fe concentration in ( $\text{mg kg}^{-1}$ )

ranged from 7.68 to 133 for F18 and F14, respectively, as shown in (Table 3); a significant difference was found between soils a lower amount of DTPA-Fe of  $4.2 \text{ mg kg}^{-1}$  for calcareous soils was reported by<sup>[8]</sup>. Kumar et al.<sup>[12]</sup> investigated the distribution of the available Fe, and its relation with several soil characteristics in

calcareous soil cultivated with rice in India and reported that DTPA-Fe varied from 6.09 to 32.14 mg kg<sup>-1</sup>. Also, Meena *et al.*,<sup>[29]</sup> reported DTPA-Fe between 4.01-29.3 mg kg<sup>-1</sup> for ten soil samples in India. However, Najafi-Ghiri *et al.*<sup>[27]</sup> reported DTPA-

Fe ranged from 2.20-90.0 mg kg<sup>-1</sup> for 59 calcareous soil samples in Iran. Compared to those reported for calcareous soils, the available Fe in the current study are in the range reported globally.

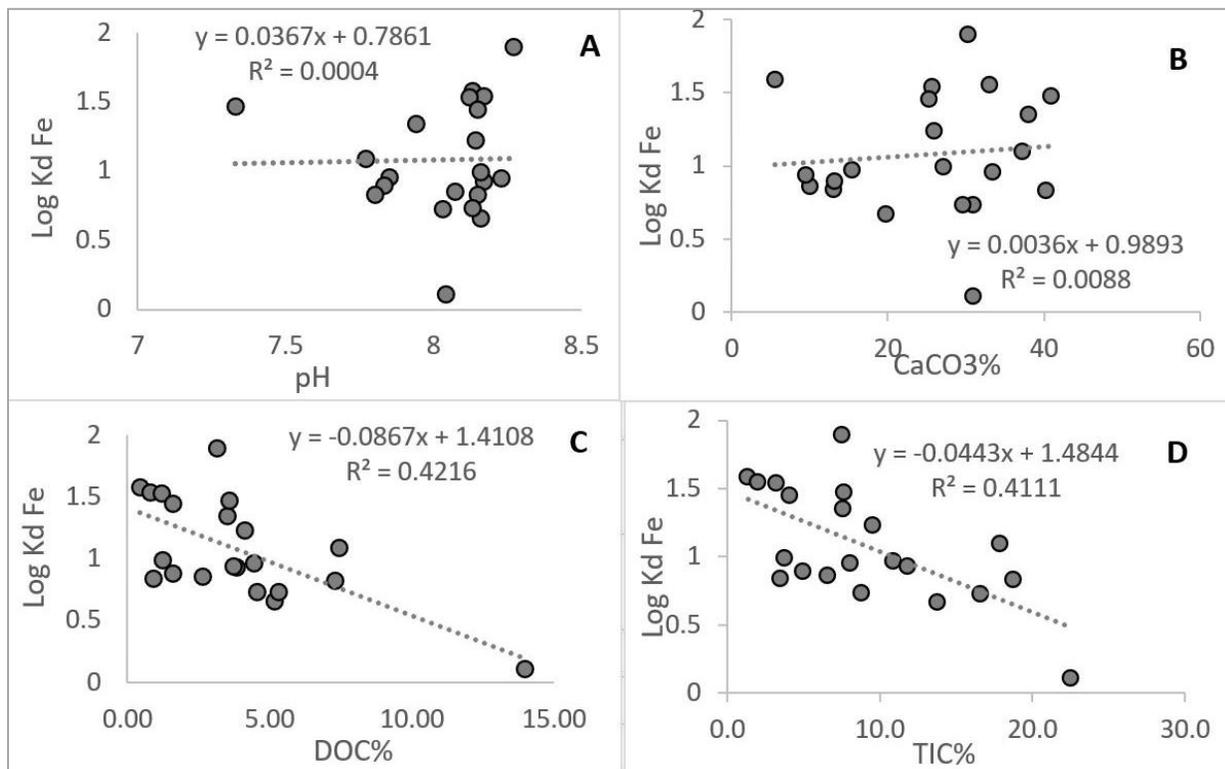
**Table 3:** The average values of available Fe (DTPA-Fe mg kg<sup>-1</sup>), soluble Fe (mg L<sup>-1</sup>), partition coefficient, and (Log K<sub>d</sub>) with its standard deviation in all the studied soils.

Field	DTPA-Fe (mg Kg <sup>-1</sup> )	Soluble-Fe (mg L <sup>-1</sup> )	Log K <sub>d</sub> Fe	±SD
F1	27.0	2.66	0.97	0.05
F2	12.9	3.18	0.86	0.04
F3	26.2	1.68	0.94	0.04
F4	8.80	1.89	0.84	0.01
F5	13.5	2.85	0.67	0.04
F6	12.0	3.25	0.90	0.04
F7	44.5	1.24	1.58	0.09
F8	48.9	1.30	1.55	0.15
F9	45.4	2.71	1.54	0.02
F10	53.5	3.10	1.35	0.02
F11	48.2	1.42	0.83	0.01
F12	51.9	1.64	1.09	0.01
F13	53.5	1.33	1.47	0.01
F14	133	1.01	1.90	0.03
F15	18.8	1.77	0.95	0.03
F16	8.12	1.08	0.73	0.02
F17	11.1	1.28	0.73	0.04
F18	7.68	1.35	0.11	0.04
F19	40.1	2.21	1.23	0.12
F20	42.6	2.09	1.45	0.04
F21	15.1	7.80	0.99	0.01

The solid-liquid partition coefficient (Log K<sub>d</sub>) is widely used in risk assessment as a guide to the environmental mobility of elements and pesticides<sup>[24]</sup> and a key factor influencing the accumulation<sup>[30]</sup>. Moreover, it has been used for the study of many nutrients, such as K<sup>+</sup>, Mg<sup>++</sup>, and NH<sub>4</sub><sup>+</sup><sup>[31]</sup>. Iron has not been studied this way, partly due to the challenge of determining the extremely low Fe concentration in the solution phase of calcareous soils. In the current study, the partition coefficient of Fe (Log K<sub>d</sub> Fe) was determined using ICP-MS, to assess the condition arising from the fields from the absence of Fe fertilizer application.

In the current study, the value of Log K<sub>d</sub> Fe was estimated for Fe adsorption in all the studied soils (C<sub>s</sub>/C<sub>sol</sub>), C<sub>solid</sub> (mg kg<sup>-1</sup>), and C<sub>solution</sub> (mg L<sup>-1</sup>) referring to DTPA-Fe and Fe<sup>+2</sup> ion concentration in solution determined following the speciation using the geochemical model WHAM-7 (Windermere Humic Aqueous Model, version 7). The partition coefficient suggesting the generally low soil adsorption strength for labile Fe ranged from 0.11±0.04 to 1.90±0.03 for F18 and F14, respectively, and showed a significant difference between samples under (p≤0.05), but more than half of values of log K<sub>d</sub> Fe under one log unit throughout all studied fields due to difference in soil ability for adsorbing available Fe (Table 3). The low values of Log K<sub>d</sub> Fe

indicate that the most Fe remaining in the soil solution were available plants uptake. This may be due to enough DOC and TDC in the soils, which prevent Fe from precipitation or fixation by soil components. Yaacob *et al.*<sup>[30]</sup> reported higher K<sub>d</sub> values for Pb and Cu for 5 different soils. Difference of Log K<sub>d</sub> Fe with soil pH, CaCO<sub>3</sub> content, DOC, and TOC is shown in (Fig. 4). A positive but non-significant correlation was found between Log K<sub>d</sub> Fe with soil pH, and CaCO<sub>3</sub> content in all studied soils, as might be expected although a narrow range of pH values and CaCO<sub>3</sub> content. Sohlenius *et al.*<sup>[32]</sup> studied the soil characteristic's effect on the K<sub>d</sub> value for several elements and found that the value of K<sub>d</sub> increased with increasing pH value of the soil. However, a high negative significant correlation was found between Log K<sub>d</sub> Fe with DOC and TOC contents in all studied soils. This may be due to the adsorption of Fe on particle surfaces<sup>[24]</sup>. A similar result was reported by<sup>[32]</sup>. Hochmuth<sup>[5]</sup> reported that organic matter present in the soil, in the soil solution, soluble Fe<sup>+2</sup> adsorbed onto soil particle surfaces.



**Figure 4:** Correlation between Log  $K_d$  Fe with (A) soil pH, (B)  $CaCO_3$  content, (C) DOC, and (D) TDC in all the studied soils.

**Conclusion**

The findings showed in the current study suggest that Fe solubility in the vegetable-grown calcareous soils in Iraqi Kurdistan was often controlled by the solid phase of the siderite Fe mineral. Moreover, the results of Log  $K_d$  values and high correlation between soluble Fe with DOC and TDC indicated that these soils had low capacity for Fe adsorption; this may be due to the high presence amount of organic matter in such soils, which is prevents Fe from precipitation of fixation by soil components. Results suggest that siderite is the dominant Fe mineral in the studied soils. Is also can be conclude that in the studied soil Fe deficiency slightly occur due to remain of  $Fe^{2+}$  activity slightly under level of  $10^{7.7}$  M.

**Conflict of interests**

The author declares that they have no conflict of interest

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