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ARTICLE

Study of Environmental and Geochemical Effects on The Distribution and Transformations of Iron Oxides in Some Soils

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ABSTRACT

This study was conducted to determine the content, distribution and transformation of iron oxides in the soils of the Middle Euphrates regions in Iraq. The study included four sites: Tuwairij area in Karbala Governorate, College of Agriculture at the University of Kufa in Najaf Governorate, College of Agriculture at the University of Qadisiyah in Diwaniyah Governorate, and the Nile District in Babylon Governorate. The results showed that the soils of Najaf and Qadisiyah were superior in terms of their content of total free iron oxides (Fet) compared to the soils of Karbala and Babylon. The relative distribution of free iron oxides was generally close among the studied sites, with a homogeneous pattern in the distribution of these oxides within the soil horizons. As for silicate iron oxides (Fes), a homogeneous pattern was observed in the soil of Babylon with its content increasing with depth, while these patterns varied in the soils of Karbala, Najaf and Qadisiyah. Regarding the ratios of crystalline iron oxides (Fed/Fet), the study showed that the Babylon and Qadisiyah soils recorded the highest values, while these values were lower in the Najaf and Karbala soils. On the other hand, amorphous iron oxides (FeO) showed similar values in the Najaf and Qadisiyah soils. In general, these results clearly showed the effect of environmental and geochemical factors of the study areas on the distribution and transformations of iron oxides in the soil of the Middle Euphrates regions.

Keywords: Fed/Fet; FeO; Crystalline; Iron Compounds; Active Iron Ratio

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1. Introduction

Iron is one of the most common elements in the Earth's crust, with its total environmental content estimated at about 6%. It is found in many ores, often in the form of oxides and hydroxides. Iron oxides and hydroxides form a very important group of minerals, both economically (iron ore) and scientifically for soils (redox processes, soil formation processes)[1]. The most important of this group are: magnetite ($FeO \cdot Fe_2O_3$), hematite (Fe_2O_3), goethite ($\alpha FeOOH$), akaganite (β-FeOOH), lepidocrocite (γ-FeOOH), ferroxyheite (δ-FeOOH) and ferrohydrite [$Fe_5HO_8 \cdot 4H_2O$)]. Iron oxides found in soils and sediments can be divided into two main groups: the magnetic group and the color group, which differ based on their physical properties. The magnetic group includes magnetite (Mgt) and maghemite (Mgh), which are characterized by high magnetic sensitivity (MS). While the color group includes hematite (Hm) and goethite (Gt), which are characterized by their distinctive colors. The distribution and composition of each group in soils and sediments are controlled by weathering and genetic formation processes, under the influence of specific climatic conditions[2, 3]. Ref.[1] indicated that there are (6) iron oxides consisting of Fe and O: hematite (α-Fe_2O_3), magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), (β-Fe_2O_3), (ε-Fe_2O_3), and (FeO). In most of these compounds, iron is in the trivalent state, but FeO and $4Fe_3O$ contain Fe^{+2}. Iron oxide (Fe^{+3}) or iron(III) has four polymorphs, only two of which are found in nature as the minerals hematite (α -Fe_2O_3) and maghemite (γ -Fe_2O_3), but the other two (β -Fe_2O_3, ε Fe_2O_3) can be found only as synthetic nanoparticles. Ref.[4] found a clear variation in the content of free iron oxides in soils depending on the degree of their development and the presence of iron movement with other soil components, especially clay. There are 16 types of iron oxides with different crystalline structures, chemical compositions and valence state of iron. Iron oxides are classified into three different groups based on the oxidation state of Fe including: (a) Iron oxides containing only trivalent Fe(III), including ferrihydrite, goethite, hematite, lepidocrocite, etc. (b) Very rare iron oxides containing exclusively divalent Fe(II), including only Fe(II)O and $Fe(II)(OH)_2$. (c) Mixed-valent iron oxides containing both Fe(III) and Fe(II) in their structure, including magnetite and green rust (GR). Iron is mainly found as Fe(III) oxides, especially near the surface of the Earth's crust due to the presence of oxygen. However, under suitable redox conditions in aquatic or terrestrial environments, Fe(III) oxides can act as electron acceptors for microbial respiration or react with Fe(II) or other reducing agents to form mixed-valent Fe minerals such as magnetite And green rust[5].

The presence of these oxides in the soil depends on many factors, the most important of which are high temperatures, long dry periods, and the degree of soil development[6]. Iron oxides are widely distributed in terrestrial systems, and are highly concentrated near the surface as a result of air weathering of iron-bearing minerals[7]. The nature of its distribution varies according to the prevailing pedological processes and the parent material from which the soil was formed. It has been shown that soils formed from magnetite parent material have a higher proportion of quartz and granite due to their high content of ferromagnesian minerals[8]. The basic structural unit of all iron oxides is octahedral. Depending on whether it is a true oxide or hydroxide, each Fe atom is surrounded by six O atoms or by O and OH ions. Tetrahedral coordination occurs only in the structure of magnetite and maghemite oxides. The most common anion network in iron oxides is hexagonal close-packed (hcp). This term refers to a type of crystal arrangement in which the atoms are packed very closely together in a hexagonal pattern. In this arrangement, the atoms form layers that are stacked so that each atom in one layer is surrounded by six other atoms in a hexagonal pattern. The layers are stacked so that the atoms in each layer occupy the spaces in the layer before it. Both goethite and hematite exhibit this structural stacking, and for this reason, they are called α-phase. Close-packed cubic (CCP) This term refers to a crystal arrangement in which atoms are densely packed in a cubic pattern. In this arrangement, the atoms are arranged so that each atom occupies a position very close to its neighbors in a cubic lattice. This arrangement is also known as the face-centered cubic (FCC) structure pattern in lepidocrocite and maghemite, and is called the γ-pattern. Structural differences between iron oxides are primarily in the octahedral arrangement. In iron oxides, corners, edges, and faces are shared between the octahedral lattices[9]. All iron oxides consist of compact oxygen clusters, where the cavities of the octahedral and/or tetrahedral units are partially filled with iron (Fe^{+3} and Fe^{+2}). Different mineral species differ in the way in which the $Fe(O/OH)_6FeO_6$ or FeO_4 in the vacuum by sharing it at the corner, edge or face[10]. The study "Geochemistry of the Heavy Metals in the Tansrift Mine (Atlas of Beni Mellal, Morocco)" assesses the pollution levels caused by hazardous heavy metals (Cd, Cr, Cu, Pb, Zn, Fe, Ni) in the mining district. Using geochemical analyses and pollution indexes, it identifies significant copper contamination in the area, with environmental impacts on air, water, and soil due to mining activities[11–13].

Iron oxides are considered an essential component of soil composition, greatly affecting its physical and chemical properties, which affects its agricultural productivity. With climate change and environmental degradation in many areas, including the Middle Euphrates regions in Iraq, there is an urgent need to understand how environmental and geochemical factors affect iron oxides. There is also growing concern that changes in the distribution and transformation of iron oxides may lead to deterioration of soil quality and fertility, which poses a threat to agricultural sustainability. In addition, iron oxides can contribute to the absorption and release of heavy metals and pollutants. Therefore, our study aimed to focus on how environmental and geochemical factors affect iron oxides to understand the chemical reactions that occur in soil. We also seek to determine how iron oxides are distributed and transformed based on the prevailing climatic conditions and geographical location of some soils from the Middle Euphrates regions in Iraq.

2. Materials and Methods

Soil samples were taken from each horizon of the study soils, and placed in plastic bags after being numbered and transferred to the laboratory for the purpose of conducting laboratory analyses[14]. Total free iron oxides were extracted using sodium citrate-bicarbonate-dithionite according to[15]. Amorphous free iron oxides were extracted using acidified ammonium oxalate (PH = 3) according to[16, 17]. Crystalline free iron oxides were estimated from the difference between total free and amorphous iron oxides (crystalline iron oxides = total iron oxides - amorphous iron oxides). Total iron was determined by digesting the soil sample (wet method) using 60% perchloric acid $HClO_4$ and concentrated nitric acid HNO_3 according to the method described in[18, 19] and was determined by atomic absorption spectrophotometer. Finally, silicate iron was calculated by subtracting the values of total iron oxides from total iron, then converted to Fe_2O_3 by multiplying by 1.41.

3. Results and Discussion

3.1. Free Iron Oxides in the Study Soils

3.1.1. Total Free Iron Oxides (Fet)

The results of **Table 1** showed the content of total free iron oxides (Fet) and ranged between (0.92–3.79) g kg^{-1}, and the highest value appeared in Najaf soil at the surface depth, and the lowest value in Karbala soil at the third depth. Also, Najaf and Qadisiyah soils outperformed Karbala and Babylon soils in terms of their content of total iron oxides, as they ranged between ((3.22–3.79), (1.95–3.02)) in Najaf and Qadisiyah respectively, and between ((0.92–1.30), (2.04–2.46)) g kg^{-1} in Karbala and Babylon respectively, **Table 1** and **Figure 1**. The results confirmed that the soils of Najaf and Qadisiyah contain larger amounts of free iron oxides compared to the soils of Babylon and Karbala. This is attributed to several reasons, including the increased intensity of weathering processes as a result of the increased rainfall rates, which led to the release of large amounts of iron from silicate minerals, or to the difference in the amount of iron-bearing minerals and the degree of development of these soils, which was confirmed by[20]. To prove this, the ratio between the amount of total iron in the soil and the amount of silicate iron in the soil (Fes/FeT) was calculated, **Table 2** and **Figure 1**. The results showed that the distribution of the ratios was close or similar between the different soils, which indicates a relative stability in the weathering processes and chemical transformations over time in these areas, with a slight increase observed in some layers, and the reason may be attributed to water activity or the decomposition of light materials, which leads to different effects on the redistribution of oxides, but in general there was a homogeneous pattern in the distribution of these ratios within the studied soil layers. It began to decrease with depth, except for the last depth of the Qadisiyah soil, and its percentages ranged between (0.87–0.93)%, as the lowest value appeared in the Najaf soil at the third depth, while the highest value appeared in all soils at the first depth, which indicates the presence of larger quantities of iron within the silicate minerals **Table 1** and the insufficiency of weathering to release them, unlike the soils whose horizons showed lower values as a result of their exposure to more severe weathering conditions with a longer period of time after their formation, which led to a difference in the quantities of iron silicates within the study pedons, and this was confirmed by[21, 22]. In terms of the vertical distribution of these oxides (Fes) within the study soils, which ranged between (7.64–53.11) g kg^{-1} **Table 1** and **Figure 1**, the distribution did not take a specific direction, as it took a homogeneous pattern throughout the soil of Babylon Governorate, which ranged between (17.76–27.51) g kg^{-1}, as it began to increase with depth, and in contrast to the rest of the study soils (Karbala, Najaf and Qadisiyah) ((7.64–16.78), (25.53.11), (15.15–40.77)) g kg^{-1} respectively, which increase once and then decrease again within depth,While Saleh and Qahtan, 2017, explained this to two reasons, the first is related to the increase in the percentage of separated clay, as a highly significant positive relationship was observed between the amount of free iron oxides and the percentage of clay, as the increase in clay means an increase in the accompanying oxides, while the second reason is due to the effect of weathering and washing factors, as a gradation in the percentages of oxides and the method of their distribution within the horizons of the soils under study was observed.

The degree of transformation of free iron oxides in soil can be determined by calculating the ratios Fed/Fet (the ratio of crystalline iron oxides to total iron oxides) and Fe^{+2}/Fe^{+3} (the ratio of iron(II) to iron(III)), since iron is present in the primary minerals of parent rocks in the form of Fe^{+3}, and over time the ratios Fed/Fet and Fe^{+2}/Fe^{+3} gradually decrease to approach zero, as indicated by Al-Fatlawi (2016) on the authority of Leigh (1996). To calculate the state of soil development (weathering index or iron transformations), the ratio of crystalline iron oxides to total iron oxides (Fed/Fet) is calculated. The results in **Table 2** and **Figure 1** showed that the highest values were found in the soils of Babylon and Qadisiyah, ranging between ((0.53–0.61), (0.48–0.57)) g kg^{-1} respectively, compared to the soils of Najaf and Karbala, which were lower, ranging between ((0.41–0.50), (0.39–0.45)) respectively. These results confirm the effect of climatic conditions and biological factors on weathering processes and iron transformation in the soil. The relatively low values also indicate that all these soils are considered relatively young, as the severity of weathering increases with time, especially in similar climatic conditions, as explained by[22, 23]. In general, the results confirmed that the variation in the content and distribution of iron oxides in soil is related to several factors, including weathering, which greatly affects the release of iron oxides from silicate minerals into the soil. Areas that experience severe weathering often contain higher levels of iron oxides. The variation in the quantity and distribution of iron-bearing minerals in the soil affects the total iron oxide content, and climatic conditions such as rainfall and temperature also play an important role in weathering processes and the distribution of iron oxides. Therefore, it can be said that understanding the variation in the content of iron oxides in soil requires considering the effect of these factors on the soil.

Table 1. Content of different forms of iron oxides in the study soils.

				Fet	Feo	Fed	FeT	Fes
location	depth cm	horizon	serious					
								g kg^{-1}
	0–30	Ap		2.04	0.95	1.09	29.55	27.51
BABIL	30–60	C1	MP9	2.23	0.88	1.35	24.18	21.95
	60–130	C2		2.46	1.09	1.37	20.22	17.76
	0–30	Ap		1.17	0.64	0.53	17.95	16.78
KARBALA	30–60	C1	MW8	1.3	0.79	0.51	14.09	12.79
	>60	C2		0.92	0.54	0.38	8.56	7.64
	0–30	Ap		3.79	2.13	1.66	56.9	53.11
NAJAF	30–80	C1	DM97	3.22	1.98	1.3	35.91	32.69
	80–113	C2		3.61	1.82	1.79	28.67	25.06
	0–22	Ap		3.02	1.59	1.44	43.79	40.77
	22–62	C1		2.74	1.39	1.35	28.71	25.97
QADSIAH	62–83	C2	TM1166	1.96	0.87	1.09	17.11	15.15
	83<	C2		2.18	0.93	1.25	24.29	22.11

Table 2. Content of different forms of iron oxides in the study soils.

				Fes/FeT	Feo/Fet	Fed/Fet	Feo/Fed
location	depth cm	horizon	Serious				
							$10^{-2}*$
Babil	0–30	Ap	MP9	0.93	0.47	0.53	0.87
	30–60	C1		0.91	0.39	0.61	0.65
	60–130	C2		0.88	0.44	0.56	0.8
Karbala	0–30	Ap	MW8	0.93	0.55	0.45	1.21
	30–60	C1		0.9	0.61	0.39	1.55
	>60	C2		0.89	0.58	0.41	1.42
Najaf	0–30	Ap	DM97	0.93	0.56	0.44	1.28
	30–80	C1		0.91	0.61	0.41	1.52
	80–113	C2		0.87	0.5	0.5	1.02
Qadsia	0–22	Ap	TM1166	0.93	0.53	0.48	1.11
	22–62	C1		0.9	0.51	0.49	1.03
	62–83	C2		0.89	0.44	0.56	0.8
	83<	C2		0.91	0.43	0.57	0.74

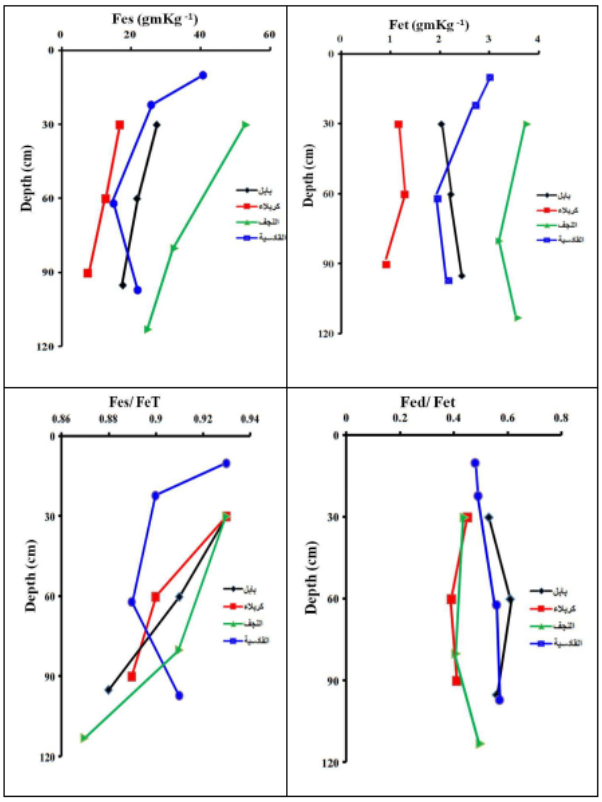

Figure 1. Vertical distribution of total and siliconized iron oxides in the study soils.

3.2. Amorphous Iron Oxides (FeO)

The results of **Table 1** showed the content of amorphous iron oxides in the study soils, where these percentages ranged between (0.54–2.13) g kg^{-1}, as the highest value appeared in Najaf soil at the surface depth, while the lowest value appeared in Karbala soil at the third depth. The results in **Table 1** indicate that the values of amorphous iron oxides in the soil are close between the soils of Najaf and Qadisiyah, where they ranged between ((1.82–2.13), (0.87–1.59)) respectively, except for the fourth depth where the values were low. As for the soils of Babylon and Karbala, the values were lower, where they ranged between ((0.88–1.09), (0.54–0.79)) respectively, with the exception of the third depth in the Babylon soil, which witnessed an increase in values. This variation in the values of amorphous iron oxides between the study soils can be explained, which is mainly due to the level of development of these soils. The more developed soil may contain iron oxides that differ in quantity and shape as a result of longer or more complex weathering, and to the organic matter that plays a role in this variation, as it works to inhibit the process of crystallization of iron oxides in the surface horizons of the soil. Organic compounds hinder the transformation of iron oxides into crystalline form, which leads to their accumulation in their amorphous form in these horizons.

The results also indicate that there is a heterogeneous distribution of amorphous iron oxides in the soils of Babylon and Qadisiyah, while the soils of Najaf and Karbala showed a homogeneous pattern in the distribution of iron oxides, **Figure 1**. This variation is attributed to the high content of organic matter in the surface horizon of these soils, as organic matter prevents the crystallization of iron oxides by reducing iron. Organic acids work to adsorb on the surfaces of iron oxides, forming complex bonds with iron, which breaks the bonds between iron and oxygen (Fe-O), thus increasing the amount of amorphous iron in the soil. As well as agricultural processes that contribute to reducing crystalline iron oxides and increasing amorphous oxides [24]. Or as a result of temporary anaerobic conditions that also prevent the crystallization of iron oxides, which leads to the accumulation of amorphous oxides [25]. The results also show that the behavior of amorphous iron oxides follows the same pattern as total oxides, showing a gradual increase in proportions from the soils of Najaf and Qadisiyah to Babylon and Karbala. This is attributed to the variation in weathering and soil development [26]. Or it is attributed to the presence of vegetation and high organic matter that enhances decomposition processes in the soil, especially in the surface horizons, where high temperatures and drought lead to inhibition of iron crystallization and an increase in the proportion of amorphous oxides. According to [27], organic acids adsorbed on the surfaces of iron oxides form complex bonds with iron, which increases the accumulation of amorphous oxides in the surface horizons that have a high content of organic matter. According to the study referred to by [28], amorphous iron oxides have effective surfaces for adsorption and fixation of organic compounds in the soil, which enhances their high adsorption capacity for organic compounds. The results showed that amorphous iron oxides gradually decrease with increasing depth in the soil, but by a small percentage. This decrease reflects the evolutionary state of the soil, where the more developed the soil is, the lower the percentage of amorphous iron oxides. This result supports what was confirmed by the study of[29], which found that the percentages of amorphous iron oxides decrease with soil development in the central and southern regions of Iraq. That is, over time, the soil is exposed to weathering and development processes, where amorphous iron oxides are transformed into more stable or crystalline forms. This process leads to a decrease in the percentage of amorphous oxides the deeper we go into the soil, as the surface layers are usually more exposed to environmental processes such as weathering and biological activity compared to the deeper layers. The greater the depth, the less the effects of surface factors such as weathering and biological activity, which leads to the percentage of amorphous oxides remaining lower in the deeper layers, where the soil is more stable.

In general, the results indicate the effect of organic matter and environmental conditions on the distribution and transformation of iron oxides in the soil, as they prevent the crystallization of oxides in horizons rich in organic matter, which leads to the accumulation of amorphous oxides, a behavior similar to the distribution of total oxides in the soil. It also confirms that the decrease in the percentage of amorphous iron oxides with increasing depth reflects the development of the soil, as these oxides transform into more stable forms over time. In addition, the results show that the low values in some layers and the variation between the soils of Najaf and Qadisiyah on the one hand and Babylon and Karbala on the other hand reflect the effect of factors such as the degree of soil development and the presence of organic matter. Organic matter slows down the crystallization of iron oxides, which explains the accumulation of these oxides in their amorphous form in soils that contain a higher percentage of organic matter, especially in the surface layers. It also confirmed the effect of organic matter and environmental conditions on the distribution and transformation of iron oxides in the soil, as it prevents the crystallization of oxides in horizons rich in organic matter, which leads to the accumulation of amorphous oxides, which is a behavior similar to the distribution of total oxides in the soil.

3.3. Crystalline Free Iron Oxide (Fed)

The results of **Table 1** indicate that the content of crystalline iron oxides in the soils under study ranged between (0.38–1.79) g kg^{-1}. The highest value of these oxides appeared in Babylon soil at the third depth, and the lowest value in Karbala soil at the same depth. The results showed that the content of crystalline iron oxides was close to the content of total iron oxides (Fet), which is consistent with what was stated by [24], where he indicated that crystalline oxides are close to the total due to the transformation of amorphous oxides into crystalline ones, which are the most stable oxides in the soil. The reason for the transformation of iron oxides may be attributed to rainfall rates that affect chemical processes in the soil and enhance the transformation of oxides into crystalline form. Also, to the presence of vegetation cover that increases biological activity in the soil, which helps to stabilize Iron in its crystalline forms, the age of the soil and the original material, these factors helped to activate the processes in these soils, the older the soil, the more chemical transformation processes there are, which increases the amount of crystalline oxides. The results of **Table 1** also showed that the values of these oxides were high in the soils of Najaf, Qadisiyah and Babylon, where they ranged between ((1.30–1.79), (1.09–1.44), (1.09–1.37)) g kg^{-1} respectively, while they were low in the soil of Karbala where they ranged between (0.33–0.53) g kg^{-1}. The reason for the high values of crystalline oxides in these horizons is due to the amount of rainfall and the presence of vegetation. These factors helped in the occurrence of pedogenic processes and the increase of these proportions in the soil. This was confirmed by [1] that crystalline iron oxides are affected by the length of drought, temperatures, amount of precipitation, and degree of soil development. The results showed a significant increase in the values of crystalline iron oxides in the surface horizons of all study soils, and this is attributed to the climatic conditions in the Middle Euphrates regions, such as high temperatures in the summer. These results are consistent with what [29] reached, where she found that crystalline oxides are high in the surface horizons due to high temperatures that increase the state of crystallization in those horizons. In general, the variation in the content of crystalline iron oxides between different soils is attributed to environmental factors such as rainfall rates, vegetation cover, and temperatures. These factors play a crucial role in the transformation of iron from amorphous to crystalline forms, leading to increased stability of crystalline oxides in soils, especially in the surface layers where weathering and transformation processes are most active [30, 31].

3.4. Active Iron Oxides (Feo/Fed)

The study deals with the transformations of iron compounds in the soil through the ratios of active iron (Feo/Fed) **Table 2** and **Figure 2**, which reflect the relationship between the most active forms of iron and non-silicate forms [22]. The ratios of active iron in the soils under study ranged between (0.65–1.55) %, where the highest value was recorded in Karbala soil at the second depth, At the second depth, which reflects a high activity of these oxides at this depth, it is an indicator of recent weathering and oxidation reactions, and the lowest value in the Babylonian soil at the same depth, and the lowest value in Babylon soil at the same depth [32]. The study noted that the distribution of active iron percentages was opposite to the distribution of crystalline iron oxides (Fed) in the soils under study. In the soils of Babylon and Qadisiyah, the active iron percentages were low, ranging between (0.65–0.87), (0.74–1.11))% respectively, except for the first depth of Qadisiyah soil, which witnessed an increase. This decrease is attributed to the high content of crystalline iron oxides in these soils. In contrast, the active iron oxide percentages were high in the soils of Karbala and Najaf, ranging between ((1.21–1.55), (1.02–1.52))% respectively. According to [29], this variation in active iron ratios is attributed to the conditions surrounding these soils, the most important of which are temperatures, which affect the speed of chemical processes, including iron transformation, and the nature of agricultural exploitation, which affects the composition of the soil and changes the distribution of chemical compounds in it, as well as plant density, which also affects the cycle of nutrients in the soil and contributes to the fixation or release of iron, in addition to the difference in the amounts of rainfall, which contributes to the dissolution of mineral elements in the soil and affects their transformations. In general, the variation in active iron ratios between different soils, as our results show, reflects the effect of environmental and climatic factors on the transformations of iron compounds. Soils containing higher levels of crystalline iron oxides show lower levels of active iron, while active iron ratios increase in soils that have the appropriate environmental conditions to transform iron into its more active forms [33–35].

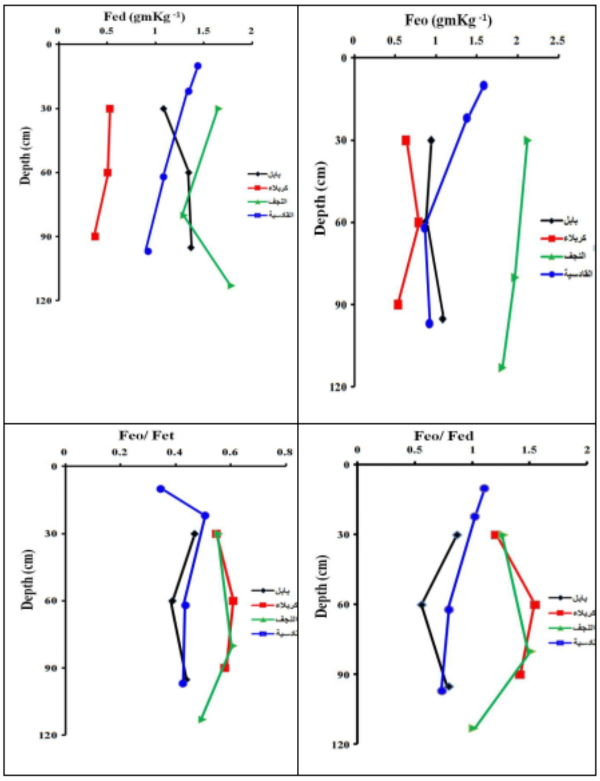

Figure 2. Vertical distribution of crystalline and amorphous iron oxides.

4. Conclusions

The study shows that free iron oxides in the soils of Najaf and Qadisiyah are higher compared to Karbala and Babylon, reflecting geological or environmental differences. The distribution of liberated iron oxides indicates stability among the soils, with local effects in certain layers. In Babylon, the distribution of silicate iron oxides increases with depth, while variations in other regions are due to local factors. The higher ratios of crystallized iron oxides in Babylon and Qadisiyah suggest ancient geological stability, while lower levels in Najaf and Karbala reflect ongoing environmental activity. Elevated non-crystallized iron oxides in Najaf and Qadisiyah indicate higher biochemical activity.

Author Contributions

Conceptualization, methodology, software, validation, formal analysis, investigation, resources, data curation, writing—original draft preparation, writing—review, editing, visualization and supervision R.H.A.A.-M., L.A.S.A.

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Conflicts of Interest

The authors declare no conflict of interest.

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