Clay Minerals Identification of Different Soil Orders in Northern Iraq

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Abstract

One of the main criteria for determining soil formation and its features is mineralogical composition of clay components. However, the relationships among clay minerals, soil orders, and soil profile development in the calcareous soils of the semi-arid regions of northern Iraq have not been thoroughly investigated. In this study, the analysis of clay mineral types in Vertisols, Mollisols, and Aridisols is conducted using X-ray diffraction. Additionally, physicochemical characteristics of soil are estimated. A series of 30 soil samples was selected at two depths (0-30 cm and 30-60 cm) and analyzed for clay mineral content, cation exchange capacity, carbonate concentration, organic matter, and exchangeable nutrients. Clay minerals found in the analyzed soils include illite (100%), kaolinite (100%), palygorskite (96.6%), vermiculite (93.3%), chlorite (83.3%), and smectite/montmorillonite (53.3%). The predominant clay mineral in Vertisols is smectite (montmorillonite) with the largest cation exchange capacity (29.20 cmolc/kg). Illite, kaolinite, vermiculite, and the occurrence of palygorskite in the studied soil orders may reflect the effect of parent material composition, carbonate-rich conditions, and high evaporation or alkalinity. It may also be a neoformation or inherited material from the soil solution, whereas smectite (montmorillonite) was largely restricted to the Vertisols and, to a lesser extent, the Aridisols. Statistical analysis showed that soil orders are more influential for mineralogical differences than soil depth. The cation exchange capacity of the soils was more closely associated with clay mineral assemblages than with carbonate content alone. It is concluded that inheritance from parent materials and carbonate-induced pedogenesis are the key factors determining clay mineral associations and soil functions in the study area.

Keywords: Clay minerals, X-ray diffraction, Total carbonate, Semi-arid, Mollisols, Vertisols, Aridisols.

1. Introduction

The soil is considered a dynamic natural system that can sustain agricultural productivity, manage biogeochemical cycles, and ensure ecosystem stability greatly depends on the nature and reactivity of the clay fraction. Among soil components, clay minerals have the highest level of reactivity due to their high specific surface area, surface chargeability, and high adsorption capacity. These properties control the basic processes occurring in soil, such as cation exchange, retention of nutrients and water, aggregation, and immobilization of contaminants. Thus, assemblages of clay minerals are widely used as important soil quality and pedogenic indicators [1, 2].

The genesis of clay minerals is controlled by mineral inheritance, transformation, and neoformation that reflects the effects of parent material, climate, topography, biology, drainage, and time on soils. The presence and proportions of minerals like smectite, illite, kaolinite, chlorite, vermiculite, and palygorskite represent evidence of the processes of soil formation under different environmental conditions. Expandable 2:1 type clay minerals, particularly smectite, usually provide high cation exchange capacity, water retention, and

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nutrient availability, while non-expandable minerals, like kaolinite, show low chemical reactivity and nutrient-holding capacity. In this way, differences in the assemblages of clay minerals not only reflect the past history of pedogenic evolution but also define the functional behavior and productive ability of soils [3]. Modern studies in the field of soil mineralogy showed that the assemblages of clay minerals have a great impact on soil functionality and ecosystem services. For example, [4]. noted that parent material and geomorphological features are the main factors determining the distribution of clay minerals, while [5]. proved that the mineralogical composition determines the reaction of soil to the management practices by controlling the dynamics of nutrients and structural stability. Similarly, [6, 7]. stated the importance of clay minerals in controlling the adsorption of nutrients and contaminants through ion exchange and surface complexation processes. In this way, these studies indicated that the assemblages of clay minerals are the integrative factor of soil genesis and environmental processes. However, the majority of previous studies were conducted separately for clay mineralogy, soil chemistry, or soil classification without considering the evolution of the assemblages of clay minerals in different soil orders and different soil depths.

Semi-arid regions can serve as ideal grounds for this purpose, since low leaching processes, high evapotranspiration and carbonate deposition result in retention of specific clay minerals that characterize the process of formation [8]. The semi-arid northern part of Iraq and specifically Duhok and Mosul Governorates consist of various geological strata, which have formed different soil orders despite similar climate. This area provides an ideal base to study how the diversity in parent materials and formation processes affects the process of development of clay minerals and the corresponding properties of soils. However, in this region, mineralogical researches were predominantly concerned with descriptive identification of clay minerals or physicochemical investigation, and complex studies that correlate mineralogical assemblages with soil order and development are still rare [9].

The proposed conceptual model builds upon the body of work already available on the fact that soils containing high levels of smectite and calcareous material in semi-arid regions are characterized by high cation-exchange capacity, effective nutrient storage, and strong horizon development, which is controlled by pedological mechanisms such as illuviation, calcification, and lessivage. The current study proposes to use soil order, profile depth, and clay mineralogy to expand the above-mentioned results to the case of the semi-arid calcareous soils of northern Iraq [10].

This study seeks to address the gap in the mineralogical composition of the soil orders, as there is no modern study of the investigated locations in this field. This paper explores the following research questions: In what way does soil order and soil depth control the clay mineral assemblage, and how do the resulting differences in the mineralogy affect the physicochemical properties of soil and its development in the semi-arid calcareous soils of northern Iraq? The null hypotheses are: (i) Different parent material and pedogenic environment result in different clay mineral assemblages in different soil orders; (ii) Smectite-rich assemblages have a higher cation exchange capacity and nutrient holding capacity compared to assemblages with lower reactivity minerals; and (iii) Variations in soil quality vertically and laterally depend mainly on the total carbonate accumulation, active carbonate, organic matter, clay content in each soil order, CEC, and clay mineral assemblage distribution. Therefore, the objectives of this paper include identifying the types of clay minerals using XRD analysis and specifying their distribution in the studied soil orders in surface and subsurface soil layers and the impact of the relationship between clay minerals and statistical

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analysis of physicochemical properties on soil quality in the calcareous soils of the semi-arid region in northern Iraq.

2. Material and Method

2.1 Study Area

The study area is located in northern Iraq and includes parts of Duhok and Mosul governorates within the northwestern segment of the Zagros Fold–Thrust Belt (Figure 1). The region has complex geological and geomorphological conditions due to the presence of tectonic activities in the northeastern margin of the Arabian Plate [11]. The study region is mainly covered by soil orders Mollisols, Vertisols, and Aridisols. Mollisols soils have high organic matter and fertility levels, whereas Vertisols soils are mainly composed of clay and shrink–swell soil texture. Aridisols are formed in arid and semi-arid climates. These differences in soil types are mainly attributed to parent material, climate, and topography. To capture spatial variability in the study region, 15 zones were selected based on land use and environmental conditions. Soil samples were collected at two different depths (0-30 cm and 30-60 cm) Five surface and five subsurface soil samples were collected from each studied soil order (Vertisols, Mollisols, and Ardisols), and a total of thirty single soil samples were collected (Figure 1). Chemical characteristics such as pH, nutrients, and organic matter were analyzed in the laboratory to assess soil quality.

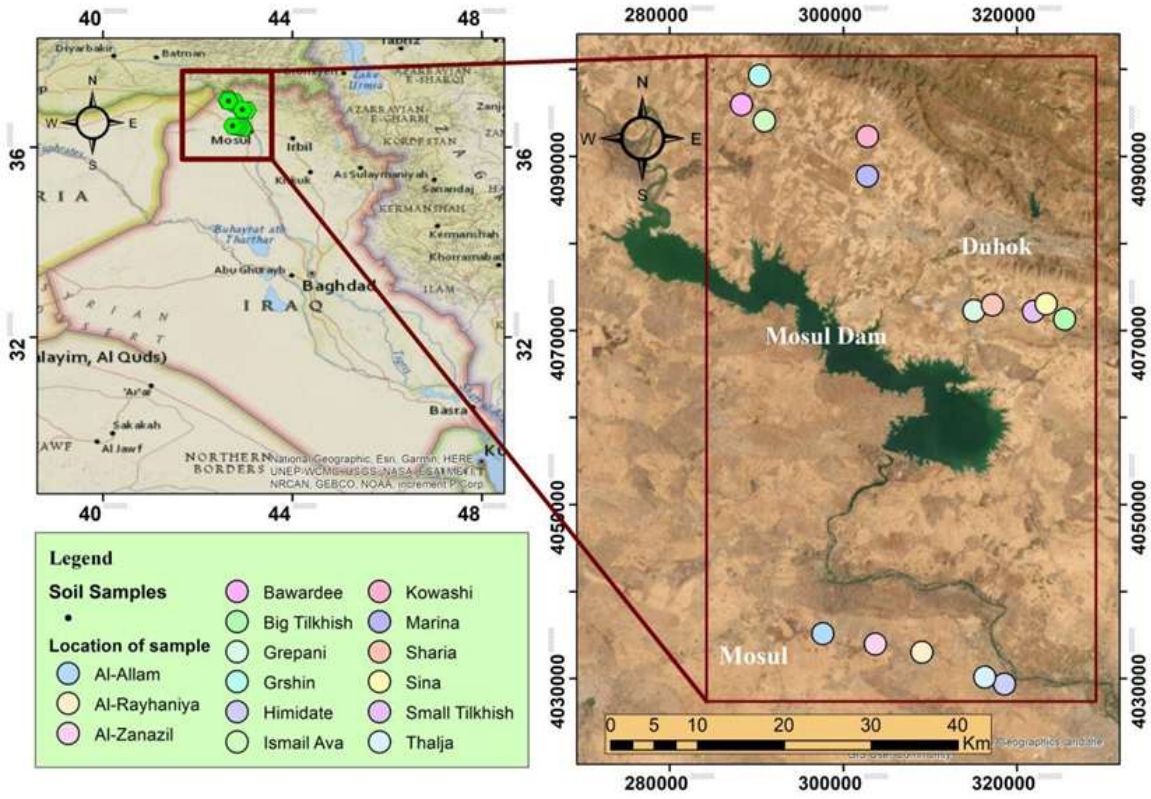


Figure 1. Map of Iraq showing the study area

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2.2 Climate and Vegetation of Study Area

The region under study (Duhok and Mosul governorates of northern Iraq) exhibits a Mediterranean semi-arid climate that is known for its cool and rainy winters and warm and dry summers. The average annual temperature is 19–21°C with a maximum of above 37°C during the summers, while the total annual rainfall ranges from 500 mm to 1000 mm (Mzuri et al., 2022). Remote sensing indices (MSAVI2, NDVI, EVI2) show that vegetation is denser in high-altitude areas and declines in lowlands due to moisture stress. A positive correlation with precipitation and a negative relationship with LST has been observed [12, 13]. In Mosul, vegetation dynamics are further affected by drought and anthropogenic disturbances, including land use changes and fire events, leading to reduced biomass and soil productivity [13].

2.3 Laboratory Analysis

A total of 30 soil samples from both the surface and subsurface layers of soil were air-dried, slightly crushed, sieved through a 2 mm sieve and analyzed according to standard soil laboratory methods. The soil pH and electrical conductivity (EC) were analyzed in a 1:2.5 soil/water ratio. The soil exchangeable cations (Ca^{2+} , Mg^{2+} , Na^{+} and K^{+}), Cation Exchange Capacity (CEC), calcium carbonate, organic matter, and free iron oxides were analyzed using internationally approved analytical techniques [14, 15, 16, 17].

2.4 Mineralogical Analysis

Clay fraction less than 2 μm was separated through sedimentation process after dispersion with sodium hexametaphosphate as a dispersing agent to separate soil particles by fractionation process following extraction of organic matter, carbonates and iron oxides from soil using chemical materials that do not affect the crystalline structure of clay minerals [18, 19]. Clay samples oriented under Mg2+ and K+ saturated states were investigated after solvation with ethylene glycol of the Mg-saturated sample and heating at 350 and 550 °C for K saturated samples based on their characteristic behavior of expansion and collapse [20, 21]. The mineral composition of the clay samples was determined by X-ray diffraction (XRD) technique using a Shimadzu XRD-6000 diffractometer with Cu-Kα radiation, and determination of mineral phase was carried out on the basis of basal reflections with Bragg's law [22].

2.5 Statistical Analysis

Descriptive statistics such as mean, standard deviation, and coefficient of variation were used for the summary of soil characteristics.

3. Result and discussion

3.1. Physicochemical Characteristics of Soils Across Soil Orders

Descriptive statistics for the investigated soil properties are shown in Tables 1 and 2. The studied soils have great variability in terms of chemical properties, which is associated with dissimilarities in parent materials, pedogenesis, and development of these soils in semi-arid regions of northern Iraq. This variability created particular environmental conditions for the formation of different assemblages of clay minerals over time. Properties related to carbonates are characterized by unique pedogenic features (Table 1). Concentration of total carbonates varies from 149.6 to 361.2 g kg-1 and shows rather weak variability (CV = 21.06%), which reflects the common presence of carbonate parent materials in the study region. On the other hand, the concentration of active carbonates (83.5–250 g kg-1) and soil organic matter (3.2–24.8 g kg-1) shows much stronger variability (CV = 56.86% and 56.43%, respectively) (Table 1). It reflects differences in carbonate mobility, biological activity, and weathering rate of soils. The presence

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of a small amount of organic matter is in accordance with semi-arid climate conditions in which low amounts of precipitation and high evapotranspiration do not allow accumulation of biomass and organic matter in the soil.

Table 1. Descriptive Statistics of Soil Physical and Carbonate-Related Properties Across the Studied Soil Samples

Parameter	Min	Max	Mean	Std. Dev.	CV %	Skewness	Kurtosis	Shapiro p
Total Carbonate (g.kg^{-1})	149.6	361.2	24.97	5.26	21.06	-0.25	-0.36	0.41
Active Carbonate (g.kg^{-1})	83.5	250	2.75	1.56	56.86	0.75	1.32	0.08
Organic Matter (g.kg^{-1})	3.2	24.8	1.158	0.65	56.43	0.73	-0.65	0.01

Footnote: *SD = standard deviation; CV = coefficient of variation. Soil properties exhibiting CV values greater than 35% indicate substantial spatial variability among sampling locations. Shapiro–Wilk p-values lower than 0.05 Indic deviation from normal distribution.*

The investigated soils also displayed contrasting chemical characteristics (Table 2). Exchangeable Ca, Mg, and CEC exhibited low to moderate variability (CV = 16.74–27.81%), suggesting relatively stable exchange complexes associated with carbonate-rich soils. Conversely, exchangeable Na, available N, and particularly available P exhibited high spatial variability (CV > 50%), indicating localized differences in nutrient cycling, weathering intensity, and pedogenic development. The relatively consistent CEC values, despite substantial variability in nutrient availability, suggest that cation-retention capacity is primarily influenced by differences in clay mineral assemblages rather than by carbonate abundance alone. This observation highlights the importance of mineralogical composition as a major regulator of soil chemical behavior.

Table 2. Descriptive Statistics of Soil Chemical Properties Across the Studied Soil Samples

Parameter	Min	Max	Mean	Std. Dev	CV%	Skewness	Kurtosis	Shapiro p
Ca^{+2} ($cmol_c$ kg^{-1})	4	12.80	7.90	2.20	27.81	0.005	-0.432	0.695
Mg^{+2} ($cmol_c$ kg^{-1})	4.6	16.60	12.82	3.18	24.80	-1.093	0.901	0.005
Na^{+} ($cmol_c$ kg^{-1})	0.24	1.88	0.56	0.31	55.72	3.113	11.293	0
K^{+} ($cmol_c$ kg^{-1})	0.22	0.68	0.41	0.13	31.23	0.368	-0.919	0.154
CEC ($cmol_c$ kg^{-1})	17.30	29.20	21.61	3.61	16.74	0.086	-0.517	0.688
Available N (%)	0.40	2.20	0.68	0.36	53.42	2.877	10.476	0
Available P (ppm)	0.003	0.19	0.02	0.03	98.9	4.174	20.067	0

Footnote: *SD = standard deviation; CV = coefficient of variation; CEC = cation exchange capacity. Variables with Shapiro–Wilk p-values < 0.05 indicate non-normal distribution. The coefficient of variation was classified as low (<15%), moderate (15–35%), and high (>35%) variability.*

Analysis of normality revealed that some parameters, including soil organic matter, exchangeable magnesium and sodium, available nitrogen, and available phosphorus, were not normally distributed ($p < 0.05$). In addition, total carbonates, exchangeable calcium and potassium,

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and cation exchange capacity were found to be normally distributed. The above differences in the shape of distribution are indicative of non-homogeneous conditions for soil formation, which are influenced by both parent material and carbonate dynamics. Despite the common semi-arid climate under which these soils have been formed, chemical properties of the soils greatly varied depending on the order and depth of soil.

The skewness of all the chemical properties in Tables 1 and 2 except for total carbonate and Mg was more than zero, implying a positively skewed distribution. On the other hand, total carbonate and Mg were negatively skewed. Generally, all the chemical properties in Tables 1 and 2 were moderately skewed. An excess kurtosis value above zero (positive) of soil properties (active carbonate, Mg, Na, N, and P) implies a higher frequency of outliers, while negative excess kurtosis of soil properties (total carbonate, organic matter, Ca, K, and CEC) implies a lower frequency of outliers compared to a normal distribution. The statistical test showed that the soil properties (total and active carbonate, organic matter, Mg, Na, K, N, and P) had p-value of less than 0.05 ($p < 0.05$), meaning the deviation from normality is significant, except for Ca and CEC which had p-values of more than 0.05 ($p > 0.05$), implying normal distribution (Tables 1 and 2).

3.2 Clay Mineral Assemblages Identified by X-ray Diffraction

3.2.1 Dominant Clay Minerals in the Studied Soils

According to X-ray diffraction (XRD) data, the clay mineral fraction of the studied soils includes illite, smectite (montmorillonite), chlorite, vermiculite, kaolinite, and palygorskite (Figure 2). Abundances of these minerals showed some variations within the orders of soil and soil depth (Table 3); however, the overall composition was quite similar throughout the region under investigation. Such mineral associations can be interpreted as evidence of complex paths of mineral transformations in the soils, although the soils retained the mineralogical inheritance formed from the sedimentary carbonatic rocks as parent materials. These mineral compositions are not typical for individual mineral phases, but rather reflect the combination of effects caused by the parent material, weathering, accumulation of carbonates, and the development of a vertical soil profile in a semi-arid environment.

Illite proved to be the most resilient mineral, occurring in all the studied soil samples (100%), regardless of soil order or sampling depth. The wide occurrence of illite suggests that this mineral is the main heritable clay mineral in the study area, found almost everywhere in the Vertisols, Mollisols, and Aridisols (Tables 3 and 4). This phenomenon can be explained by the fact that the sediments composing the geological parent material specific to the Zagros Fold-Thrust Belt are rich in this mineral. It also means that the process of chemical weathering has not been strong enough to convert the heritable minerals such as micas into their secondary counterparts. Similar prevalence of illite is typical of calcareous Mediterranean and semi-arid soils because the lack of leaching and the presence of alkaline soils do not allow for mineral transformation [23, 24, 25].

The smectite (montmorillonite) was dominantly found in Vertisols and occasionally in Mollisols and Aridisols. In Vertisols (53.3%) (Tables 3 and 4), the expandable clay mineral was confirmed to be montmorillonite, which indicates the presence of swelling 2:1 clay. The dominance of smectite (montmorillonite) in Vertisols is consistent with the diagnostic mineralogy of the Vertisols, which is formed through the pedogenic processes associated with highly saturated with bases, alkaline soils with low desilication. Under semi-arid climatic conditions, where there is a limited leaching process, silica preservation leads to the preservation or neoformation of expandable clay minerals (montmorillonite) with a shrink–

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swell property. Furthermore, the high level of smectite (montmorillonite) explains the relatively high values of the cation exchange capacity (CEC) found in the Vertisols, as 2:1 expandable clay mineral contain much higher surface charge compared to non-expandable clay minerals. These relationships between the presence of smectite, high values of CEC, and the shrink–swell property of soil are reported for Vertisols developed under semi-arid conditions [26, 27]. Previous studies show that smectite formation is promoted in the alkaline soils with a high content of calcium and magnesium under the conditions of limited leaching [28]. The lower abundance of smectite in Mollisols is an indicator of the relatively moderate clay transformation processes.

Chlorite was found to be present in most soil samples tested (83.3%), except for some Aridisols soils, which show either low quantity or structural changes of chlorite during weathering (Tables 3 and 4). The presence of chlorite in these samples indicates that these minerals have not yet completely transformed, while the decrease in chlorite indicates its transformation due to prolonged weathering under an alkaline environment. Similar trends have also been observed in calcareous soils where chlorite gradually converts to vermiculite with increasing weathering [29].

The occurrence of vermiculite was widespread in most of the examined soil orders (93.3%) which shows that the weathering of primary mica minerals has taken place in the area under investigation. Since vermiculite forms through partial weathering of illite or biotite minerals, the distribution of this mineral can be regarded as an indication of the intermediate stage of mineral formation and not the complete weathering process. The occurrence of both illite and vermiculite indicates that there is a large amount of inherited minerals while the pedogenic transformation takes place under semi-arid conditions. Kaolinite was found in all the soil samples tested (100%) despite the expected low degree of weathering of calcareous soils. This can be explained by the inheritance of this mineral in old weathered sediments rather than the neoformation of this mineral (Tables 3 and 4). The simultaneous occurrence of illite, vermiculite and kaolinite points to the fact that the mineral association in the studied soils is formed via different weathering processes. Such a mineral composition was found by [30] in semi-arid soils with moderate weathering conditions. Kaolinite was observed in all the studied soil samples; however, the maximum abundance of this mineral was less than that of illite and smectite. The presence of kaolinite indicates the hydrolysis and weathering of silicate minerals; however, the low abundance of kaolinite points out to the fact that there are no advanced weathering processes in the study area. [31] Lowe et al. found similar results for the semi-arid soils developed under weak desilication.

Palygorskite occurred in almost all soils (96.6%) regardless of their order or depth and represents the characteristic clay mineral in the studied semi-arid landscapes (Tables 3 and 4). The occurrence of this fibrous clay mineral can be well associated with the alkaline nature of the soils, abundant calcium carbonate, and minimal leaching processes. Previous studies have shown that palygorskite either forms or exists in a stable state in calcareous environments where there is more evaporation than precipitation and where the soil solution is highly charged with calcium, magnesium, and silica [32, 33, 34].

Illite, kaolinite, palygorskite, and vermiculite demonstrate high dominance levels; chlorite demonstrates an elevated level; and smectite (montmorillonite) is present at a medium level in the soils of the analyzed orders. Altogether, the above-mentioned minerals show that the soils of concern contain inherited clay minerals and those formed during the pedogenic transformations. The combination of these two mineralogical types evidences a balance of mineral retention and mineral transformations, regulated

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by parent material composition, carbonate deposition, the level of weathering, and pedogenesis itself. It helps to describe the spatial variation of the physicochemical soil properties and provides direct evidence of the pedogenic transformations that occurred in the investigated soils. The predominance of illite, smectite, and palygorskite evidences the significant role of the semi-arid pedogenic conditions, calcareous parent material, and low leaching in the studied soils.

3.2.2 Comparison of Clay Mineral Assemblages Among Soil Orders

However, the frequency of clay minerals differed between the studied Vertisols, Mollisols, and Aridisols (Tables 3 and 4; Fig. 2), suggesting that the soil order plays a major role in defining mineralogical composition by means of pedogenesis, different drainage and parent materials. Despite the identical semi-arid climate of the region, clay mineral compositions of the soils indicate their separate evolutionary path, and not the impact of climate alone.

Vertisols possessed the most complicated and active mineral assemblages and included the combined presence of smectite (montmorillonite), illite, chlorite, vermiculite, kaolinite, and palygorskite. The widespread presence of smectite (montmorillonite) is the feature distinguishing Vertisols from the other two soil orders and providing the soils with unique properties of shrinking and swelling, as smectite (montmorillonite) is capable of retaining large amounts of water in winter and subsequent swelling, followed by water loss and shrinking during summer season in the wet-dry cycle. It represents one of the key features of the soil order formation in addition to relatively high cation exchange capacity of the soils provided by the large specific surface area of the smectite (montmorillonite) mineral. Combined presence of inherited illite along with pedogenically developed smectite and vermiculite reveals that these soils had experienced moderate weathering while maintaining favorable geochemical environment for expandable clay minerals. This type of mineralogical assemblage is typical for Vertisols forming in alkali soils with high calcium content and limited leaching [26, 27]. In contrast to Vertisols, mineralogically less diverse soils are characteristic for Mollisols, where smectite occurs in just one subsurface horizon, and illite, vermiculite, chlorite, kaolinite, and palygorskite are found at all horizons of this soil order. Based on the mineralogy, the process of pedogenesis in Mollisols has taken place as a result of gradual transformation of inherited micas and not neoformation of expandable clay minerals. The presence of vermiculite, alongside illite, implies moderate weathering processes and continued mineral transformation, while at the same time retaining relatively stable soil mineralogical properties.

As for Aridisols, they show mineral assemblages of illite, vermiculite, kaolinite, and palygorskite, with chlorite being present as weak reflections and smectite (montmorillonite) sporadically in this soil order. Palygorskite, which is often accompanied by carbonate minerals in the soils developed on calcareous substrates, indicates the long-term effect of alkaline weathering with low precipitation and evapotranspiration rates. Thus, mineral evolution of such soils is regulated not by chemical hydrolysis but by carbonate deposition and limited leaching, providing mineral preservation and preferential formation of fibrous clay minerals. Such mineral associations have been commonly observed in arid and semi-arid calcareous soils of the Mediterranean Basin and the Middle East [35, 36].

The comparative analysis of mineral assemblages of the surface (0-30 cm) and subsurface (30-60 cm) soil layers shows slight variations (Table 4). This means that the major types of clay minerals stay almost unchanged in the profiles under investigation. The similarity of mineral assemblages at different depths suggests that parent material is more important for

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determining the clay mineral assemblages than short-term surface weathering. Nevertheless, the occasional presence or absence of smectite (montmorillonite) and the decrease of chlorite in some of the subsurface layers prove the local mineral transformation process caused by pedogenesis within the soil profile. In general, the difference in the mineralogy demonstrates that the soil order is an efficient classification scheme used to describe the evolution of clay minerals in northern Iraq. Vertisols contain many 2:1 expandable clay. The Mollisols are intermediate forms of clay mineral transformation while Aridisols retain mineral assemblages typical for the environment with high levels of carbonate and water deficit. The different mineral assemblages are determined not only by the distinct parent material and the pedogenic evolution pathway but also explain the regional variation of cation exchange capacity, nutrient content and soil quality that was analyzed earlier when discussing soil chemical properties (Tables 1 and 2). Thus, the mineral assemblage represents the major link between soil formation and soil functioning in these calcareous soils.

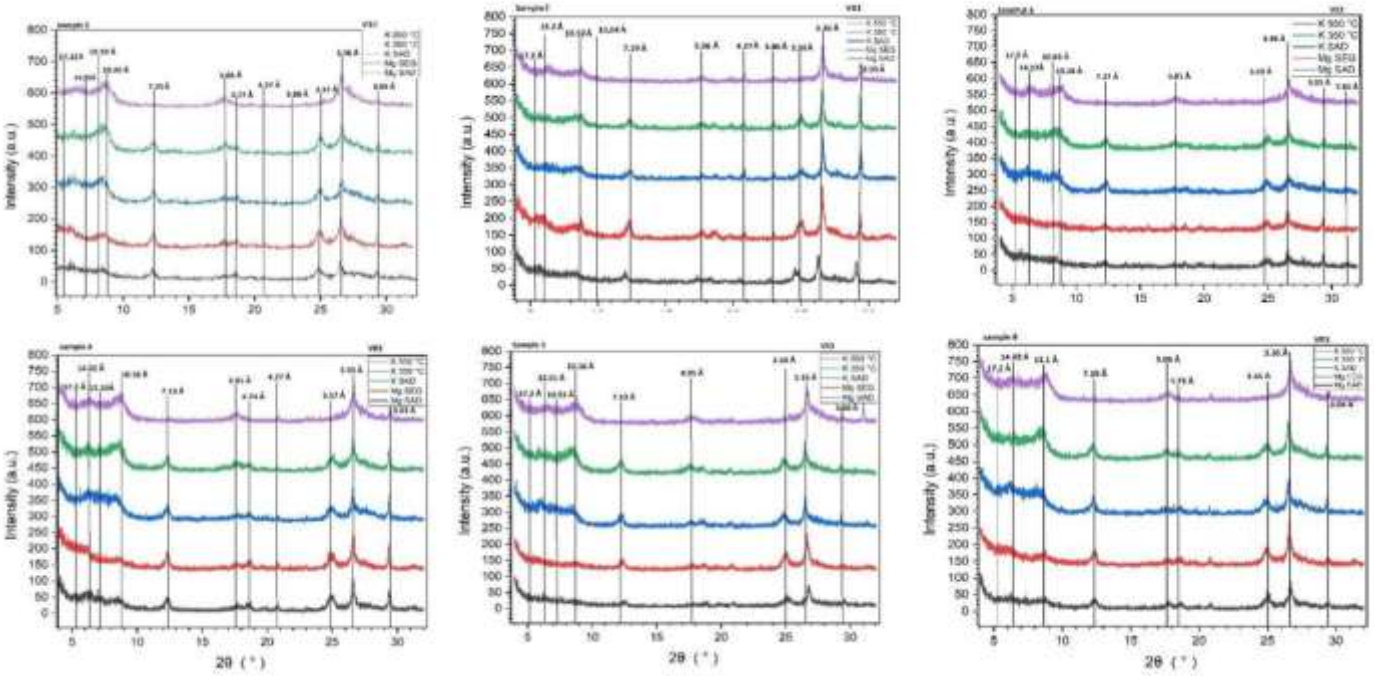


Figure 2. X-ray diffraction (XRD) patterns to identify clay mineral types in the investigated soil samples.

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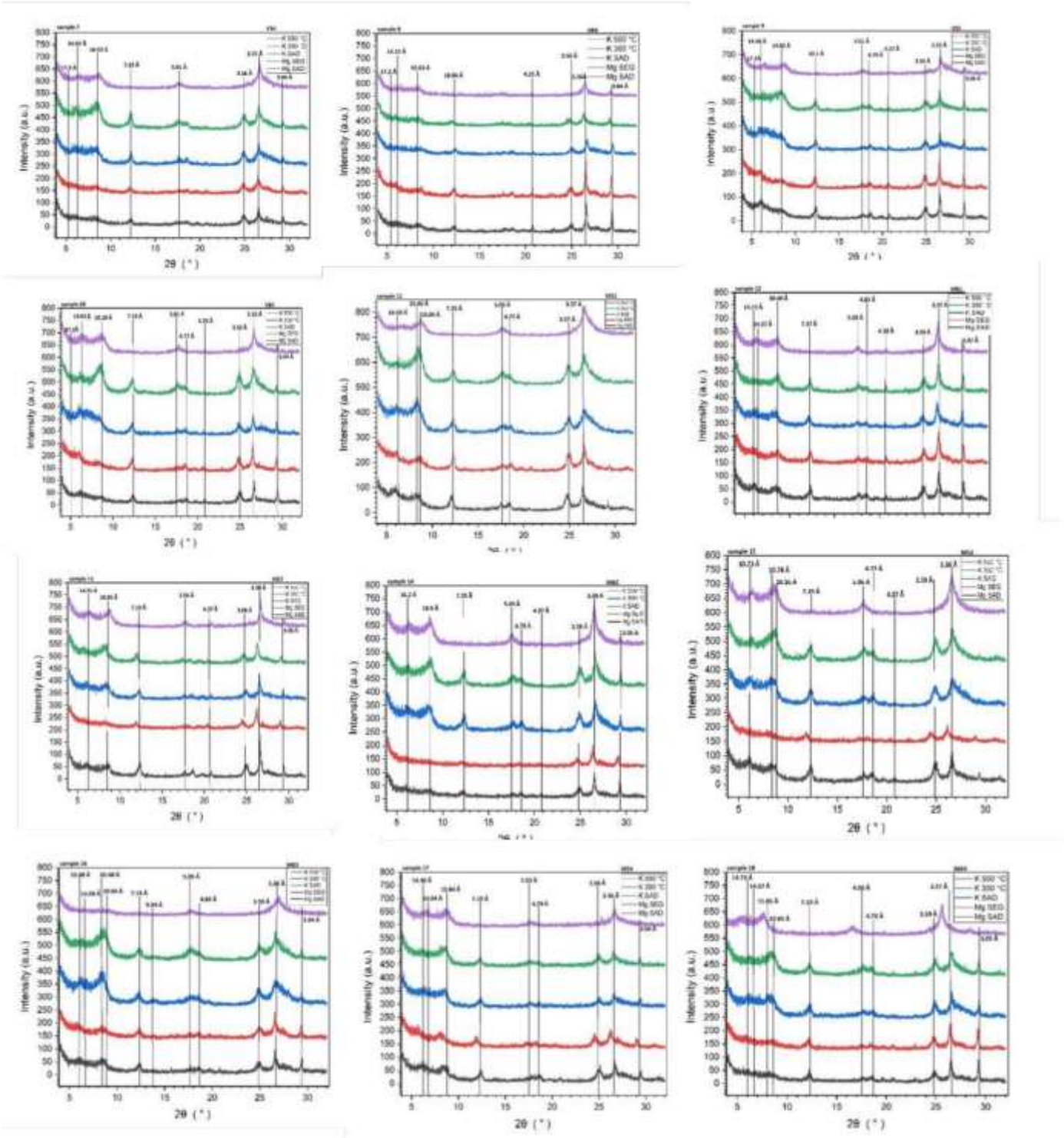


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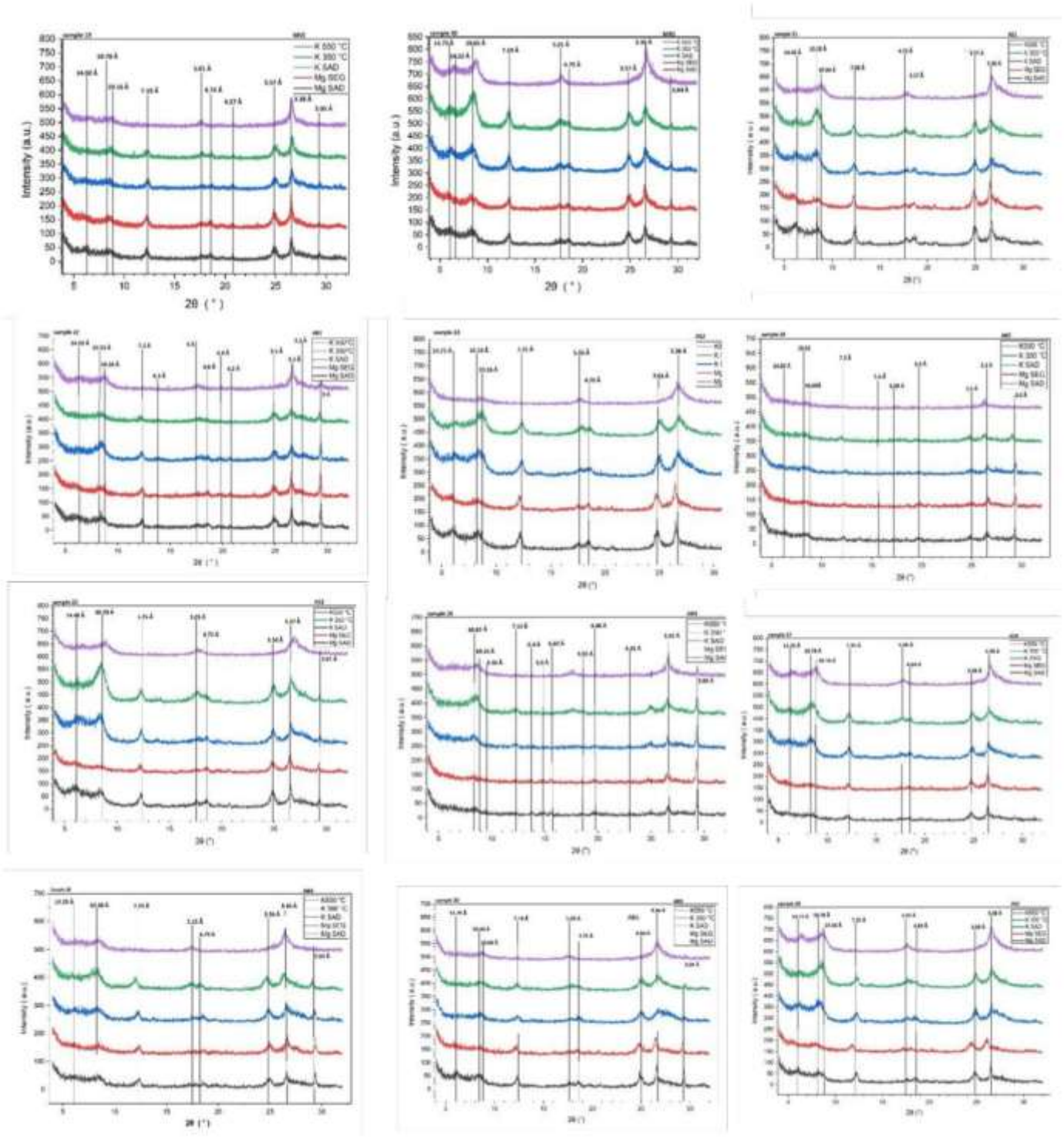


Figure 2. X-ray diffraction (XRD) patterns to identify clay mineral types in the investigated soil samples

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Table 3. Replication of clay mineral occurrence across all studied soil samples (n =30)

Mineral	Replication (n)	Occurrence (%)	Dominance level
Illite	30	100%	Very High
Vermiculite	28	93.3%	Very High
Kaolinite	30	100%	Very High
Palygorskite	29	96.6%	Very High
Chlorite	25	83.3%	High
Smectite (Montmorillonite)	16	53.3%	Moderate

***Footnote**: Replication was calculated based on presence/absence in XRD patterns across Vertisols, Mollisols, and Aridisols at two depth intervals (0–30 cm and 30–60 cm).*

3.2.3 Distribution of Minerals in Soil Orders

There were variations in the distribution of clay minerals among the Vertisols, Mollisols, and Aridisols, reflecting differences in the pedogenic processes, parent materials, and environments. Although many mineral types were common across the three soil types, variations were noted in the prevalence of vermiculite, smectite, chlorite, and palygorskite minerals. (Table 4).

Table 4. Existence of clay minerals across soil orders and depths in the studied area

Soil Order	Depth (cm)	Illite	Chlorite	Smectite (Montmorillonite)	Vermiculite	Palygorskite	Kaolinite
Vertisols	0–30	5	5	5	5	5	5
	30–60	5	5	5	5	5	5
Mollisols	0–30	5	5	0	5	4	5
	30–60	5	4	1	4	5	5
Aridisols	0–30	5	4	3	5	5	5
	30–60	5	2	2	4	5	5

Vertisols are defined by the abundance of smectite (montmorillonite) alongside illite, chlorite, vermiculite, palygorskite, and kaolinite. Smectite (montmorillonite) can be detected in all the surface and subsurface samples of Vertisols. Smectite (montmorillonite) forms one of the defining features of Vertisols, which is responsible for shrink–swell properties, cracking and cation exchange capacity. Similar mineralogical composition has been observed in the case of Vertisols developed in semi-arid environments and calcareous soils [28, 37]. Smectite (montmorillonite) along with illite indicates a moderate degree of soil weathering and a limited leaching environment.

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However, the amount of smectite (montmorillonite) in Mollisols is completely absent except in one subsurface sample. Illite, kaolinite, palygorskite, chlorite, and vermiculite are the common minerals in the soil. Smectite (montmorillonite) is not present in any Mollisol samples apart from one subsurface sample that shows a weak peak (refer to Figure 2 and Table 4). Absence of expansive clay minerals in Mollisols could be due to better drainage and increased weathering intensity, causing disillication and loss of silica (SiO_2), in addition to the effect of increased rainfall in the Mollisol location that led to leaching of basic cations, particularly Ca and Mg. The presence of illite and vermiculite shows that there is a moderate alteration in minerals under semi-arid environment. Similar findings were reported by Jackson [30], who noted that illitic mineral assemblages commonly dominate moderately weathered grassland soils.

The most characteristic feature of Aridisols is the extensive distribution of palygorskite together with illite and kaolinite. The clay mineral montmorillonite (smectite) is found in some soil samples, but less extensively than in Vertisols. The extensive occurrence of palygorskite in virtually all Aridisols is explained by arid climate, high rates of evaporation, and alkaline nature of soils. As mentioned by Singer (1989), the formation of palygorskite is related to arid conditions and the accompanying accumulation of silicon and magnesium in these soils. Furthermore, some Aridisol horizons contain weak peaks of chlorite which reflects the partial alteration of minerals that occurs due to low intensity of weathering.

It is important to state that the differentiation of soil orders by minerals is caused by the interconnection of climatic conditions, drainage, soil parent materials and pedogenesis. Vertisols have higher contents of smectite clay minerals, Mollisols consist mainly of illitic and kaolinitic assemblages, and Aridisols contain high amounts of palygorskite because of arid conditions of their formation. These facts provide useful information about genesis and mineral transformation in the study area. The distribution of clay minerals in deeper horizons of the studied soil orders is given in Table 4. Based on XRD analysis data, the surface (0-30 cm) and subsurface (30-60 cm) horizons of soil orders demonstrate insignificant differences in the mineral assemblages (Figure 2). This fact reflects the greater impact of the similarity of parent materials rather than surface weathering on mineralogy.

The presence of illite was significant in both layers in all soil orders studied and there was no clear vertical variability, thereby proving the continued presence of illite as the major inherited clay mineral in the study area (Tables 3 and 4). Likewise, vermiculite with the exclusion of two subsurface samples, and palygorskite with the exclusion of one surface sample of Mollisol were consistently present in all soil samples of the studied soil orders. Kaolinite was found in all surface and subsurface soil samples across the three studied soil orders, suggesting that this mineral is either an inherited component of the parent material or a stable secondary mineral formed during earlier stages of soil evolution. In contrast, chlorite also appeared in all surface soil samples in the studied soils except Aridisols. It exhibited a slight reduction in occurrence and disappeared in some soil samples of Aridisols (Table 4). In Aridisols, the slight decline in chlorite may indicate gradual alteration during pedogenic development of the studied soils.

The smectite (montmorillonite) clay mineral prevailed in all surface and subsurface soil samples of the Vertisols soil order with only slight variations in some particular surface and subsurface horizons. Though such minor variations do exist, they point to the occurrence of local mineral transformations depending on the soil depth. Perhaps, the abundance of smectite in Vertisol samples is associated with the retention of expandable 2:1 clay mineral in a relatively stable

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geochemical environment. However, the general homogeneity of mineral compositions at different soil depths proves that vertical clay mineral differentiation does not take place in the studied calcareous soils. At the same time, smectite (montmorillonite) is not present in any sample of the Mollisols soil order except for one subsurface sample. The smectite (montmorillonite) mineral appears in three surface and two subsurface samples of the Aridisols soil order, whereas it is absent in other samples of this soil order (Table 4). Such results prove that the variability of minerals with soil depth is much lower than their variability in soil orders.

Conclusion

The clay mineral assemblages are closely associated with soil orders and are a key factor controlling the behaviour of calcareous soils in the semi-arid region of northern Iraq. Illite, kaolinite, palygorskite, vermiculite, chlorite, and smectite (montmorillonite) were the dominant minerals. respectively. Smectite (montmorillonite) occurs mainly in Vertisols and contributes to higher exchange capacity. Soil order exerted a stronger influence on mineral distribution than soil depth, reflecting differences in pedogenic processes and soil evolution. The dominance levels of illite, kaolinite, palygorskite, and vermiculite are very high, whereas chlorite is high and smectite (montmorillonite) is moderate in the studied soil orders. Vertisols contained all clay minerals regardless of their amount, whereas smectite (montmorillonite) was absent in approximately all samples of mollisols. Chlorite had less of an appearance in aridisols.

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