Combined effect of salts and Groundwater Fluctuation in Clay Mineral Transformations of some sites in Babil and Al-Diwaniyah Provinces, Iraq

Sarah H.A. Al-Amidi[1,2*] , Raid S Jarallah[2]
[1]Department of Soil Sciences and Water Resources, College of Agriculture, Al-Qasim Green University, Babylon 51013, Iraq.
[2]Department of Soil Sciences and Water Resources, College of Agriculture, University of Al-Qadisiyah, Iraq.
[*]Corresponing authors e-mail: : agr23.ph01@qu.edu.iq
E-mail: raid.jarallah@qu.edu.iq

Abstract
To investigate the mineralogical transformations of clay minerals induced by groundwater table fluctuations and to identify the dominant clay minerals in Babil and Al-Diwaniyah provinces, one representative site was selected in each province. The selected sites were affected by accumulated salts and had been previously modified through land leveling and elevation relative to the surrounding lower adjacent soils and nearby roads. This configuration enabled direct visual observation of groundwater table fluctuations over a period more than 15 years.

The study sites were located at coordinates (N 31°57′3.76″, E 44°48′16.47″) in Babil Province and (N 32°19′2.21″, E 44°22′20.73″) in Al-Diwaniyah Province. Three soil samples were collected from each site: the upper-layer sample represented soils minimally affected by salinity; the intermediate-layer sample represented the zone influenced by fluctuating groundwater movement; and the lower-layer sample represented soils continuously saturated with saline groundwater.
The results obtained from the soils of Babil Province indicated the presence of regular-type chlorite (true chlorite) and revealed mineralogical transformations characterized by the formation of smectite with increasing groundwater salinity influence. In addition, illite and kaolinite minerals were detected. Gypsum was detected exclusively within the groundwater salinity saturation zone and was absent in the other soil layers. Moreover, an increase in palygorskite content was observed with increasing depth, corresponding to conditions of prolonged saturation.
The results obtained from the soils of Al-Diwaniyah Province indicated the presence of swelling chlorite, along with mineralogical transformations involving smectite and montmorillonite. Additionally, palygorskite was identified; however, its intensity and relative abundance decreased with increasing depth, in contrast to

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the trend observed in the soil samples from Babil Province. This behavior can be attributed to the lower concentration of calcium ions in these soils, as calcium is the key ion controlling gypsum formation rather than sulfate ions. The results also confirmed the presence of illite and kaolinite minerals.

Keywords: Groundwater fluctuation; Babil; Al-Diwaniyah; Gypsum; Swelling chlorite; True chlorite

Introduction

Groundwater commonly contains dissolved salts, particularly in arid and semi-arid regions, where salt concentrations may be relatively high. When groundwater approaches the soil surface, it rises through capillary action, and upon evaporation, salts are deposited at the surface. Over time, these salts accumulate within the soil profile. (1) reported that groundwater evaporation increases as the water table approaches the soil surface, thereby enhancing salt accumulation on and within the soil. (2) further emphasized that the upward movement of saline groundwater is intensified in areas where the groundwater table is shallow and evaporation rates are high, leading to an accelerated rate of soil salinization.

(3) demonstrated a direct effect of groundwater-induced salinization in soils characterized by a shallow groundwater table. Their study examined both groundwater depth and salinity, concluding that reliance on groundwater depth alone, without considering salt concentration, does not yield reliable results. They further clarified that groundwater salinity exerts a stronger influence on soil salinization than groundwater depth.

Mineralogical properties exert a pronounced influence on the chemical characteristics of saline soils. Such soils are typically dominated by montmorillonite clay minerals rather than kaolinite and are characterized by neutral to alkaline reaction conditions, often approaching sub-alkaline levels, along with high base saturation at exchange sites. In contrast, calcite and gypsum contents are generally lower than those in non-saline soils. Moreover, high concentrations of exchangeable sodium govern ion-exchange processes and the relative solubility of sodium and calcium salts (4).

Clay minerals represent the most significant fraction among soil colloids due to their high contribution to soil surface charge. This importance is particularly evident in 2:1 clay mineral, which possess permanent negative charges, whereas 1:1 clay mineral predominantly exhibits pH-dependent charges (5).

X-ray diffraction results reported by (6) for the clay fraction revealed the presence of chlorite, illite, kaolinite,

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and palygorskite minerals. In contrast, (7) indicated that the mineralogical composition of Iraqi alluvial soils consists mainly of montmorillonite, mica, chlorite, and minor amounts of quartz, palygorskite, vermiculite, and calcite, in addition to smectite groups and mixed-layer minerals. Based on the aforementioned findings, the present study aims to investigate the role of groundwater fluctuation in driving clay mineral transformations in soils exposed to prolonged groundwater movemen

Materials and Methods

The study area was selected within agricultural lands located in Babil and Al-Diwaniyah provinces. One representative soil site was chosen from each province based on the following criteria:

Site 1: Babil Province

A soil site affected by accumulated salts resulting from groundwater movement was selected. The site is located near a depression and adjacent to a roadway, which facilitated direct visual observation of groundwater table fluctuations over a period exceeding 15 years. The site is located at coordinates (N 31°57′3.76″, E 44°48′16.47″). Based on the observed groundwater fluctuation pattern, three distinct zones were identified, from which soil samples were collected as follows:
(a) an upper-layer sample representing soils minimally affected by salinity;
(b) a middle-layer sample representing the zone influenced by fluctuating groundwater movement; and
(c) a lower-layer sample representing soils continuously saturated with saline groundwater.

Site 2: Al-Diwaniyah Province

Soil samples were collected from a site located at coordinates (N 32°19′39.85″, E 44°22′4.06″). Sampling was conducted using the same methodology applied at the Babil Province site.

All soil samples were air-dried, gently crushed using a wooden hammer to break aggregates, and sieved through a 2-mm mesh. The samples were thoroughly homogenized and stored in plastic containers for subsequent chemical analyses (Table 1). In preparation for X-ray diffraction (XRD) analysis, clay fraction samples were examined at the Scientific Research Authority / Department of Materials Science, Iraq, using an X-ray diffractometer (Shimadzu 6000, Japan).

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Table 1. Chemical analyses of soils affected by groundwater salinity in Babil and Al-Diwaniyah provinces.

SO_4^{2-} (Meq/L)	CO_3^{2-} (Meq/L)	HCO_3^- (Meq/L)	Cl^- (Meq/L)	K^+ (Meq/L)	Na^+ (Meq/L)	Mg^{2+} (Meq/L)	Ca^{2+} (Meq/L)	EC (dS/m)	pH	Soil affected by groundwater salinity	Site	No.
50.84	0.00	8.00	202.00	0.41	160.43	10.00	90.00	20.20	8.07	slight influence	Babil	1
298.38	0.00	16.00	1332.00	1.08	297.30	848.00	500.00	109.20	7.80	Moderately affected		2
290.26	0.00	9.00	891.00	1.13	619.13	290.00	280.00	184.70	7.16	Saturated with saline groundwater		3
49.14	0.00	23.00	315.00	0.48	296.65	70.00	20.00	61.50	7.73	slight influence	Al-Diwaniyah	4
57.48	0.00	17.00	435.00	0.87	326.61	82.00	100.00	76.10	7.17	Moderately affected		5
60.18	0.00	14.00	445.00	0.87	326.30	84.00	108.00	73.30	7.11	Saturated with saline groundwater		6

Results and Discussion

Clay minerals were identified based onط variations in their X-ray diffraction (XRD) patterns following treatments with magnesium saturation, ethylene glycol solvation, potassium saturation, and thermal heating, as described by (8).

X-ray diffraction results for the first site in Babil Province revealed the clay mineral assemblages of the soil samples representing the three groundwater salinity conditions (low salinity, fluctuating, and continuously saturated), as illustrated in Figures (1, 2), and (3), respectively. Figure (1) presents the XRD patterns of the clay fraction from the Babil site for the soil sample minimally affected by groundwater salinity. The results showed a basal reflection at 14.15 Å under both air-dried and ethylene glycol–treated conditions. This reflection persisted after heating to 350 °C but collapsed upon heating to 550 °C, indicating the presence of swelling chlorite.

Under ethylene glycol treatment, the basal spacing expanded to 17.38 Å, which is characteristic of montmorillonite. This reflection disappeared following potassium saturation and heating to 350 °C and 550 °C, suggesting that the mineral is inherited from mica. Additionally, a reflection at 10.48 Å was detected under ethylene glycol treatment, corresponding to palygorskite. The intensity of this reflection decreased after potassium saturation and heating to 350 °C and further declined upon heating to 550 °C. This behavior is attributed to the removal of channel water from the internal structure of palygorskite as temperature increases (9).

Illite was identified by a stable basal reflection at 10.19 Å observed across all treatments, which remained unchanged even after heating to 550

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°C. Furthermore, a reflection at 7.09 Å was detected under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, but disappeared after potassium saturation and heating to 550 °C. This reflection corresponds to kaolinite.

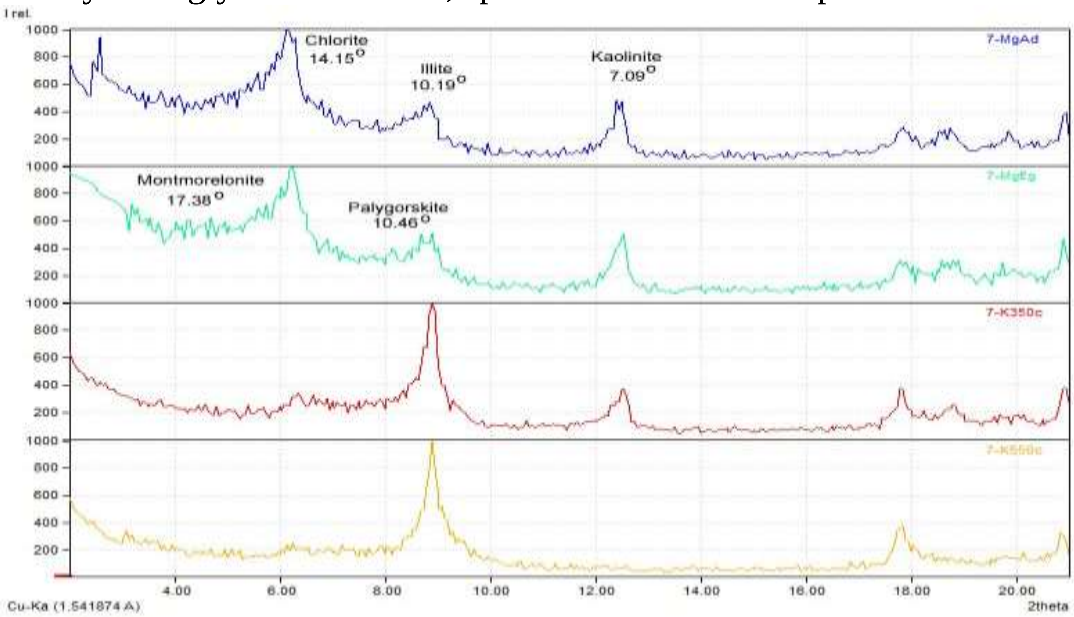

Figure 1. XRD patterns of the clay fraction from the Babil site for the soil sample minimally affected by groundwater salinity.

Figure (2) illustrates the X-ray diffraction (XRD) patterns of the clay fraction from the first site in Babil Province for the soil sample affected by fluctuating groundwater salinity. The results revealed a basal reflection at 14.14 Å corresponding to chlorite under magnesium-saturated and air-dried conditions. Under ethylene glycol treatment, this reflection expanded to 17.50 Å, indicating the presence of montmorillonite, which disappeared following potassium saturation and heating to 350 °C and 550 °C. This behavior suggests that the mineral is inherited from mica.

A reflection at 10.45 Å was also identified under ethylene glycol treatment and is attributed to palygorskite. Illite was identified by a basal reflection at 10.22 Å observed consistently across all treatments, remaining stable even after heating to 550 °C. In addition, a reflection at 7.10 Å was detected under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, but disappeared after potassium saturation and heating to 550 °C, which is characteristic of kaolinite.

Comparison between Figures (1) and (2) shows noticeable variations in the intensity, peak shape, and peak broadening of the 14.14 Å reflection. These changes indicate mineralogical transformations within

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the chlorite structure. This interpretation is further supported by variations in the illite reflections and the emergence of montmorillonite peaks, suggesting favorable conditions for montmorillonite formation (Table 1) (10). Additionally, broadening of the palygorskite peak was observed, reflecting changes in its crystallinity under fluctuating groundwater salinity conditions.

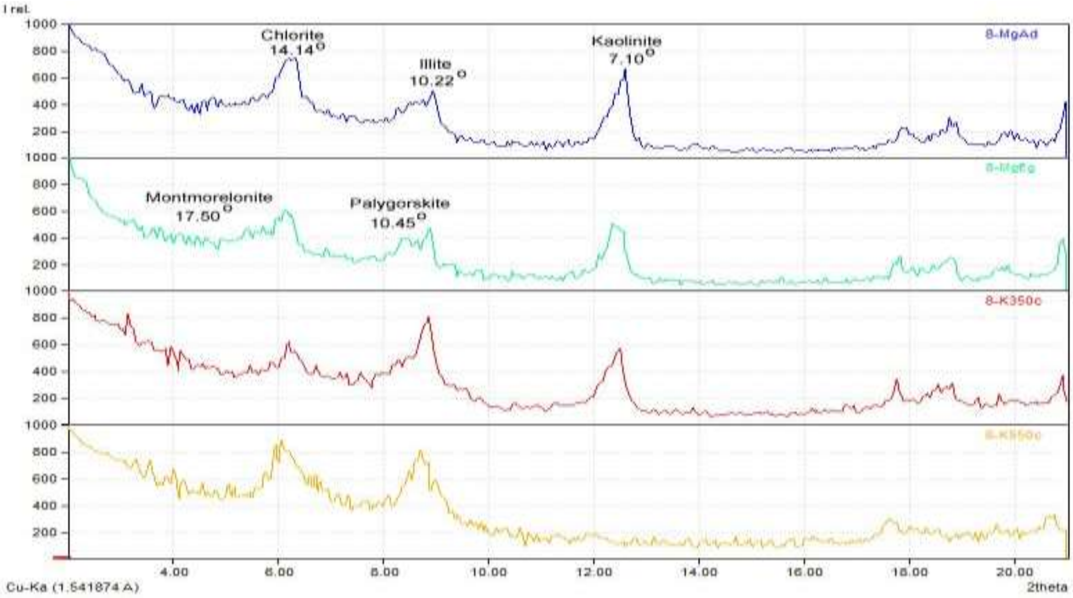

Figure 2. XRD patterns of the clay fraction from the first Babil site for the soil sample moderately affected by groundwater salinity.

Figure (3) presents the X-ray diffraction (XRD) patterns of the clay fraction from the first site in Babil Province for the soil sample continuously saturated with saline groundwater. The results revealed a basal reflection at 14.13 Å corresponding to chlorite under magnesium-saturated and air-dried conditions. Under ethylene glycol treatment, this reflection expanded to 17.42 Å and 17.01 Å, representing montmorillonite and smectite, respectively. These reflections disappeared following potassium saturation and heating to 350 °C and 550 °C, indicating that these minerals are inherited from mica.

These observations reflect mineralogical transformations associated with the 14.13 Å reflection and its conversion into other clay minerals. This interpretation is further supported by the reduction in intensity and peak broadening of the mica-related reflection, which was identified by a stable basal spacing at 10.22 Å across all treatments and remained unchanged even after heating to 550 °C.

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A basal reflection at 10.50 Å was detected under magnesium-saturated and air-dried conditions as well as after ethylene glycol treatment, corresponding to palygorskite. The intensity of this reflection decreased after potassium saturation and heating to 350 °C and further declined upon heating to 550 °C. Additionally, a reflection at 7.80 Å was identified under magnesium saturation and air-dried conditions and is attributed to gypsum. The occurrence of gypsum is associated with high concentrations of calcium and sulfate ions, which are essential for gypsum formation (Table 1), in addition to the prolonged exposure time and the arid to semi-arid climatic conditions characterizing the study area.

Furthermore, a reflection at 7.09 Å was observed under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, but disappeared following potassium saturation and heating to 550 °C. This reflection is characteristic of kaolinite. These findings are consistent with those reported by (11).

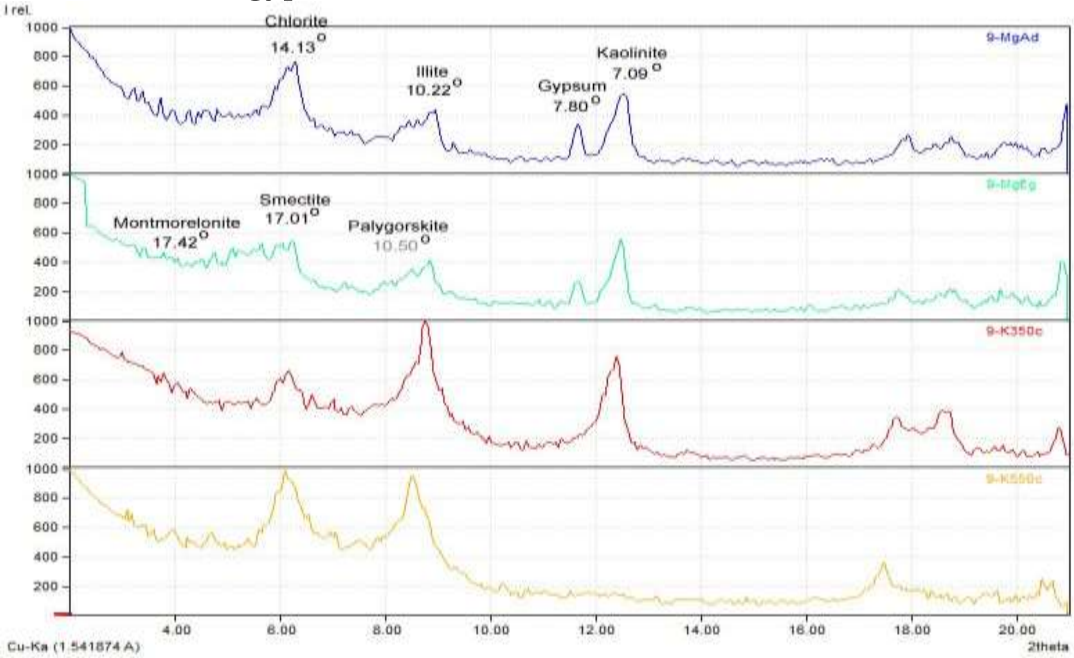

Figure 3. XRD patterns of the clay fraction from the first Babil site for the soil sample continuously saturated with saline groundwater.

Based on the above results, prolonged exposure of soils to groundwater (Sample 3) over an extended period (approximately 15 years) led to the formation of swelling-type chlorite under heating treatments at 350 °C and 550 °C, as indicated by the increased intensity and relative abundance of the corresponding basal reflections. In

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contrast, soils minimally affected by salinity (Figure 1) exhibited chlorite that disappeared under these heating treatments. However, in the moderately saline (Sample 2; Figure 2) and Saturated with saline groundwater soils (Sample 3; Figure 3), the persistence of chlorite-related XRD reflections suggests the presence of regular (true) chlorite. This transformation is attributed to increased deposition of internal carboxyl interlayers within the mineral structure, leading to enhanced thermal stability of chlorite (12).

The results further indicate that this mineralogical change occurred in chlorite originally present in the soil rather than through neoformation. This interpretation is supported by the disappearance of the 7.09 Å reflection upon heating to 550 °C, confirming that this peak corresponds to kaolinite rather than the second-order chlorite reflection, thereby indicating the absence of newly formed chlorite at this site.

Additionally, the occurrence of the 7.80 Å reflection corresponding to gypsum was observed exclusively in the permanently groundwater-saturated sample (Figure 3), whereas this reflection was absent in the other samples (Figures 1 and 2). This finding highlights the critical role of groundwater in gypsum precipitation and crystallization. The lack of gypsum formation in the minimally and intermittently affected soils may be attributed to rapid groundwater movement and exposure to drying conditions, as gypsum requires the presence of structural water within its chemical composition. Such conditions are predominantly met in the soil layer continuously saturated with saline groundwater.

X-ray diffraction results from the Al-Diwaniyah Province site revealed clay mineral assemblages for soils with low, fluctuating, and high groundwater salinity (Figures 4, 5, and 6), respectively. As shown in Figure (4), a basal reflection at 14.4 Å representing chlorite was detected under magnesium-saturated and air-dried conditions. Under ethylene glycol treatment, this reflection expanded to 17.4 Å, indicating montmorillonite. The reflection diminished following potassium saturation and heating to 350 °C but reappeared upon heating to 550 °C at 13.75 Å, indicating the presence of thermally stable true chlorite. This behavior suggests that the mineral is inherited from mica, consistent with the findings of (7).

A stable basal reflection at 10.07 Å observed across all treatments confirms the presence of illite. Additionally, a reflection at 7.80 Å was identified under magnesium saturation and air-dried conditions, corresponding to gypsum. The high intensity and sharp peak of this reflection are attributed to elevated concentrations of calcium and sulfate ions in this sample (Table 1),

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reflecting favorable conditions for gypsum formation. Furthermore, a reflection at 7.14 Å was observed under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, corresponding to kaolinite. The persistence of this reflection after potassium saturation and heating to 550 °C indicates that it represents the second-order reflection of chlorite (7.30 Å), following identification of the primary chlorite reflections at 14.4 Å and 13.75 Å.

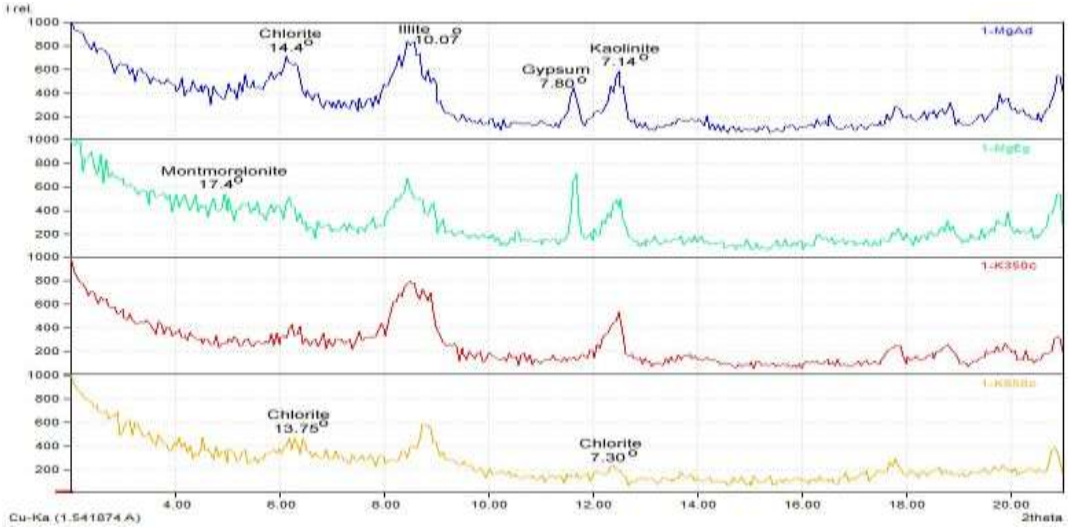

Figure 4. XRD patterns of the clay fraction from the Al-Diwaniyah site for the soil sample minimally affected by groundwater salinity.

Figure (5) illustrates the X-ray diffraction (XRD) patterns of the clay fraction from the Al-Diwaniyah Province site for the soil sample affected by fluctuating groundwater salinity. The results revealed a basal reflection at 14.04 Å corresponding to chlorite under magnesium-saturated and air-dried conditions. Under ethylene glycol treatment, this reflection expanded to 15.24 Å, indicating the presence of montmorillonite. The reflection disappeared following potassium saturation and heating to 350 °C and 550 °C, suggesting that the mineral is inherited from mica, in agreement with the findings of (13).

A basal reflection at 10.42 Å was detected under ethylene glycol treatment and attributed to palygorskite. The intensity of this reflection decreased after potassium saturation and heating to 350 °C and further declined upon heating to 550 °C, as increased thermal treatment leads to the removal of channel water from the internal structure of palygorskite (9). Illite was identified by a stable basal reflection at 10.24 Å observed across all treatments, remaining unchanged even after heating to 550 °C.

Additionally, a basal reflection at 7.81 Å corresponding to gypsum was

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observed under magnesium-saturated and air-dried conditions. The relatively low intensity of this reflection reflects the fact that this sample represents a salt-transit zone rather than a zone of salt accumulation, with salts preferentially accumulating in the surface soil layer (Sample 4). Furthermore, a reflection at 7.24 Å was detected under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, corresponding to kaolinite. The persistence of this reflection after potassium saturation and heating to 550 °C indicates that it represents the second-order reflection of chlorite, following identification of its primary basal reflection at 14.04 Å (14).

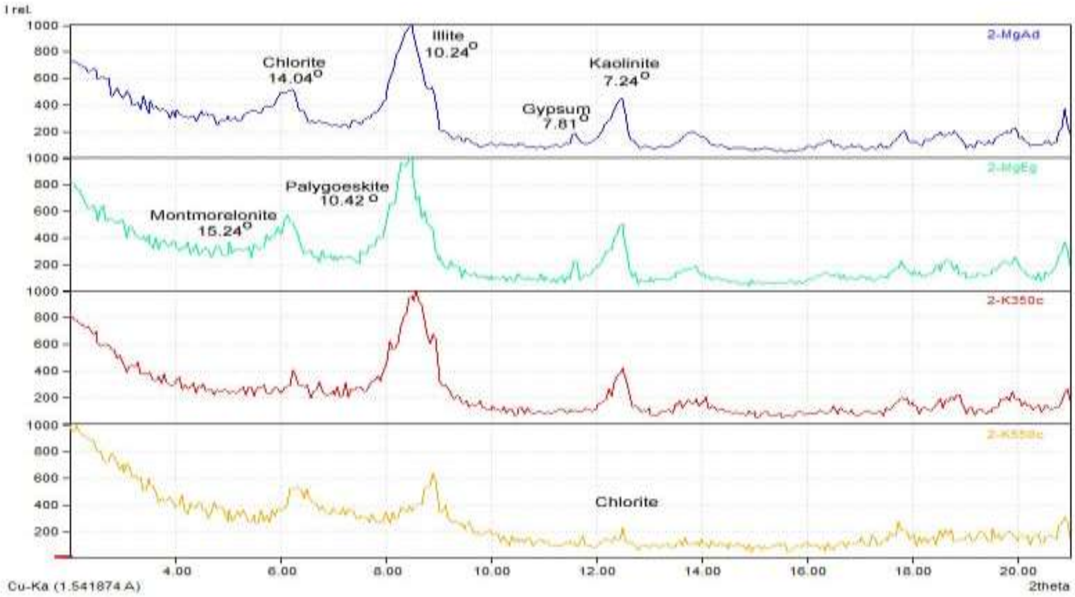

Figure 5. XRD patterns of the clay fraction from the Al-Diwaniyah site for the soil sample moderately affected by groundwater salinity.

Figure (6) presents the XRD patterns of the clay fraction from the Al-Diwaniyah Province site for the soil sample continuously saturated with saline groundwater. The results revealed a basal reflection at 14.15 Å corresponding to chlorite under air-dried conditions. Under ethylene glycol treatment, this reflection expanded to 17.30 Å and 17.00 Å, representing montmorillonite and smectite, respectively. These reflections disappeared following potassium saturation and heating to 350 °C, indicating that these minerals are inherited from mica.

Illite was identified by a stable basal reflection at 10.25 Å observed across all treatments, remaining unchanged even after heating to 550 °C. Additionally, a basal reflection at 7.83 Å corresponding to gypsum was detected under magnesium-saturated and air-dried conditions. The intensity of this reflection was higher than that observed in the intermediate sample shown in Figure (5), reflecting increased gypsum accumulation under continuous groundwater saturation.

Furthermore, a reflection at 7.14 Å was observed under magnesium saturation, air-dried conditions, ethylene glycol treatment, potassium saturation, and heating to 350 °C, but disappeared following potassium saturation and heating to 550 °C (15).

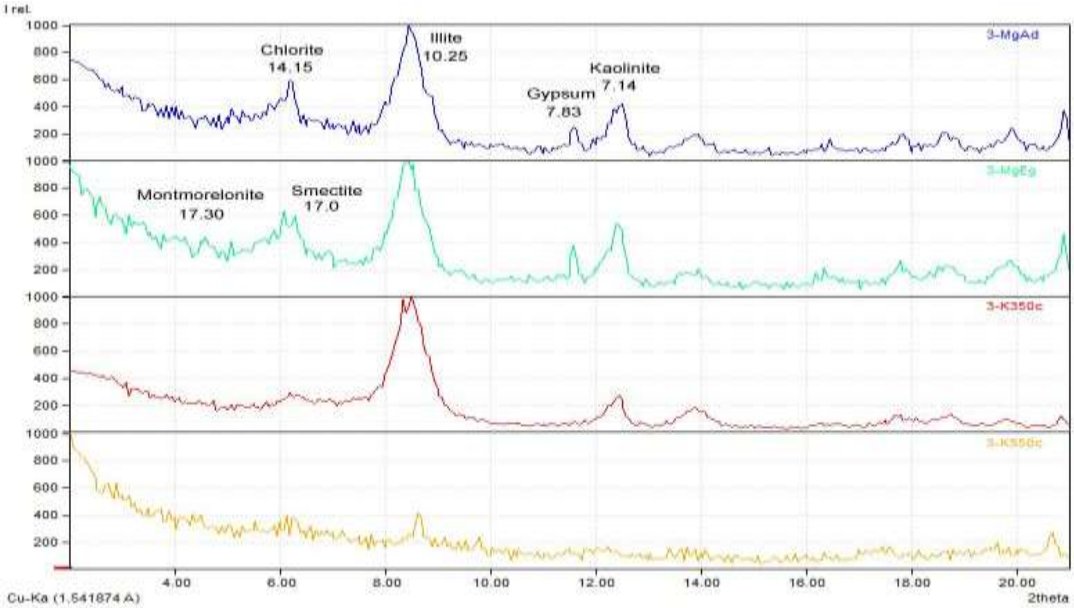

Figure 6. XRD patterns of the clay fraction from the Al-Diwaniyah site for the soil sample continuously saturated with saline groundwater.

Based on the above results, mineralogical transformations involving 2:1 clay mineral were observed, leading to the formation of smectite and montmorillonite under favorable conditions for their development, as summarized in Table (1). The intensity of these minerals increased with increasing groundwater salinity saturation. In addition, palygorskite was detected; however, its intensity decreased and it eventually disappeared in the soil sample continuously saturated with saline groundwater. This trend reflects the combined influence of drying and prolonged groundwater salinity, which promotes palygorskite accumulation in surface soils minimally affected by groundwater salinity.

In contrast, gypsum formation was most pronounced in surface soils with slight influence, followed by the permanently saline-saturated zone, and was least abundant in the fluctuating groundwater zone. This distribution may be attributed to gypsum accumulation at the soil surface as a result of evaporation, as well as its presence in the saturated zone, whereas its reduced content in

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the fluctuating zone is likely due to salt translocation without accumulation, resulting in lower gypsum concentrations.

The persistence of the 7.24 Å reflection after heating to 550 °C indicates the second-order reflection of chlorite, providing evidence for the formation of swelling chlorite. This transformation is attributed to the deposition of internal carboxyl interlayers between the sheets of 2:1 clay mineral. This interpretation is further supported by the high concentrations of magnesium ions reported in Table (1) and the XRD patterns shown in Figures (4), (5), and (6).

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