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المجلة العراقية لبحوث السوق
وحماية المستهلك

USING A SCANNING ELECTRON MICROSCOPE IN DIAGNOSING OF CLAY MINERALS IN SOME IRAQI RICE SOILS

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ABSTRACT

The Middle Euphrates region, represented by the governorates of Diwaniyah and Al-Najaf, was chosen to conduct the current study. As the two governorates are famous for cultivating various varieties of rice crop. Two methods using to irrigated these soils during the growing season, and called locally wet and dry methods. The morphological features of clay minerals were studied using scanning electron microscope (SEM), to describe the changes that occurred to the mineralogical features of these minerals due to the influence of the irrigation patterns used during irrigation of these soils. The scanning electron microscope (SEM) results show that were many changes in morphological features occurs such as in size and shape of clay minerals, in particular to smectite minerals. The scanning electron microscope (SEM) figure of clays in Al-Najaf$_{(dry)}$ soil showed some of particles appeared as well-formed imperfect hexagonal shape, which revealed that these particles belong to the chlorite mineral. While the mica minerals were appeared in lath-shaped, and rounded flakes in clays of all studied soils. Whereas the montmorillonite particles appear as a thin, webby crust, and have a flat, perming morphology. The variation in the size of the montmorillonite particles was adopted as a basis for the occurrence of the Mg-hydroxide layer within the interlayers of montmorillonite.

Keywords: Clay Minerals, Formal appearances, Rice Soils.

استخدام المجهر الألكتروني الماسح في تشخيص المعادن الطينية في بعض ترب الرز العراقية

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الخلاصة

تم اختيار منطقة الفرات الأوسط ممثلة بمحافظتي الديوانية والنجف لإجراء الدراسة الحالية. حيث تشتهر المحافظتان بزراعة أصناف مختلفة من محصول الرز. هناك طريقتان تستخدمان لري هذه التربة خلال موسم النمو، وتسمى بالطرق الرطبة والجافة محليًا. تمت دراسة المظاهر الشكلية للمعادن الطينية باستخدام، المجهر الإلكتروني الماسح، لوصف التغيرات التي حدثت في المظاهر المعدنية لهذه المعادن بسبب تأثير أنماط الري المستخدمة أثناء ري هذه التربة. تظهر نتائج المجهر الإلكتروني الماسح أن العديد من التغييرات في المظاهر الشكلية تحدث مثل حجم وشكل معادن

[*]**The research is taken from a master's thesis by the first researcher.**

الطين، ولا سيما معادن السمكتايت . أظهرت صور المجهر الإلكتروني الماسح (SEM) للطين في تربة النجف أن بعض المعادن ظهرت بشكل جيد سداسي الشكل غير كامل، مما أظهر أن هذه الجزيئات تنتمي إلي معدن الكلوريت. بينما ظهرت معادن الميكا في شكل رقائق دائرية الشكل في الطين من جميع الترب المدروسة. في حين أن دقائق معدن المونتموريلونايت تظهر على شكل قشرة رفيعة متشابكة، ولها شكل مسطح ومتعرج . تم اعتماد التباين في حجم دقائق معدن المونتموريلونيت كأساس لحدوث طبقة Mg-hydroxide داخل الطبقات البينية للمونتموريلونيت.

الكلمات المفتاحية : معادن الطين ، المظاهر الشكلية ، تربة الرز.

INTRODUCTION

The formation and transformation of clay minerals and their various characteristics are among the important processes in most soils in general, including the soils of rice farms in particular. As the nature of their presence, content and different characteristics they show, reflect the states of oxidation and reduction in those soils, just as their degree of crystallization, surface area and charge density affect their surfaces in the soil of rice farms (Churchman **&** Velde, 2019). Clay mineralogical studies using other methods such as X-ray, DTA, and IR are still scarce **(Al-Jibury & Essa; 2016, Yang *et al*; 2018; Al-Khalil & Essa, 2020; Al-Shamary & Essa, 2020).**

There are many studies have been done on Iraqi soils, which were used both types of electron microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM) for diagnosis of clay minerals. In a study conducted by (**Al-Shamary, & Essa, S.K. 2020)** she used SEM to diagnose some of clay minerals and found that the montmorillonite particles appeared in cloudy-shape with indistinct edges, while the mica particles were appeared as lathe elongated-shape. Also, (**Shahad, 2021)** during their study on Iraqi rice soils, were used a SEM to study the morphological features of clay minerals, and they found that the edges of mica particles appeared in a pale color that distinguishes them from the surface of lath-shaped particles.

In the present study, an attempt has been taken to characterize the clays in the rice soils of Middle Euphrates region using SEM technique, and due to the lack of adequate studies dealing with the study of the effect of variation in irrigation patterns within the soil of rice farms on the nature of the presence and properties of clay minerals, the importance of the current study came to diagnose the characteristics and type of clay minerals by using SEM .

MATERIALS AND METHODS

The Middle Euphrates region represented by the governorates of Al-Diwaniyah and Al-Najaf was chosen to conduct the current study (Table-1). The two governorates are famous for cultivating multiple varieties of the rice crop. The study soils are exploited by rice crop and irrigated by two methods (Flooded and Dry) during the growing season.

Table (1): some of chemical properties of the study soil

Organic matter	Active lime	Carbonate minerals	$EC_{1:1}$ Desi siemens $^{-1}$m	$pH_{1:1}$	Depth cm	irrigation method	location
$^{-1}$g kg							
8.1	50.0	175.82	٣,٣	7.0	٣٠ - ٠	dry ١	Diwaniyah
9.6	57.5	87.91	٦,٣	٧,٣	٦٠ - ٣١		
6.0	50.5	219.78	٢,٨	٧,٥	٣٠ - ٠	dry ٢	Diwaniyah
6.5	60.0	197.80	٢,٩	٧,٣	٦٠ - ٣١		
8.1	47.5	246.15	١,٨	٧,٦	٣٠ - ٠	wets ١	Diwaniyah
7.0	50.5	250.55	١,٧	٧,٤	٦٠ - ٣١		
5.0	50.0	261.54	١,٢	٧,٥	٣٠ - ٠	wets ٢	Diwaniyah
4.0	80.0	221.98	٢,٢	٧,٦	٦٠ - ٣١		
16.6	70.0	248.35	٧,١	٧,٠	٣٠ - ٠	control	Diwaniyah
13.6	85.0	259.34	٦,٩	٧,١	٦٠ - ٣١		
8.3	60.0	217.58	١,٠	٧,٢	٣٠ - ٠	dry ١	Najaf
9.6	70.0	237.36	١,٨	٧,٢	٦٠ - ٣١		
11.6	60.0	224.18	١,٩	٧,٦	٣٠ - ٠	dry ٢	Najaf
5.0	85.0	204.40	٢,٧	٧,٥	٦٠ - ٣١		
4.0	55.5	123.08	١,٥	٧,٥	٣٠ - ٠	wets ١	Najaf
3.0	75.0	228.57	١,٤	٧,٦	٦٠ - ٣١		
5.0	70.0	175.82	١,٤	٧,٦	٣٠ - ٠	wets ٢	Najaf
4.0	85.0	197.80	٢,٠	٧,٥	٦٠ - ٣١		
6.11	55.0	224.18	٢٠	٧,١	٣٠ - ٠	control	Najaf
5.8	90.0	263.74	١٨,٣٠	٧,١	٦٠ - ٣١		

The flooded or (wet) method is the traditional method used to grow rice crop, in which the soil is flooded throughout the growing season of the rice crop, while the (dry) method has been applied recently in Iraq to irrigate the rice crop, in order to water consumption, so that the soil is flooded for three days, and followed by three days drying till mid of season, and then soil flooded till end of season. Two sites were chosen in each governorate, as the first site represents: soil grown with the rice crop and irrigated by wet method. Whereas the second site

represents a soil grown with rice crop and using the dry method to irrigate it. An additional site was chosen in each governorate for uncultivated soils, which were considered as comparative soils. Soil samples were taken from all sites at depths of 0-30 and 31-60 cm, and air dried, crashed and passed through 2 mm sieve. The air-dried soil samples <2mm were dispersed in distilled water, and then the binding materials were removed, using NaOAc- at pH 5 for removing $CaCO_3$, while the organic matter was removed by treating the soil samples with NaOCl- 14%, and the free oxides were removed with citrate bicarbonate dithionate (C.B.D). The sand fraction was separated from the clay and silt fractions using a 50 µm sieve. While the clay fraction < 2 µm was separated from the silt by repeated sedimentation and syphoning based on the Stocks Law, taking into account the separation conditions of temperature and the size of the particles as stated in (**Jackson, 1979**). After the completion of the separation processes, the clay samples was taken and air-dried, and scanned under a scanning electron microscope SEM of the type Inspect S50. SEM imaging was carried out using Inspect S50 scanning electron microscope. The SEM was operated at 20 KV.

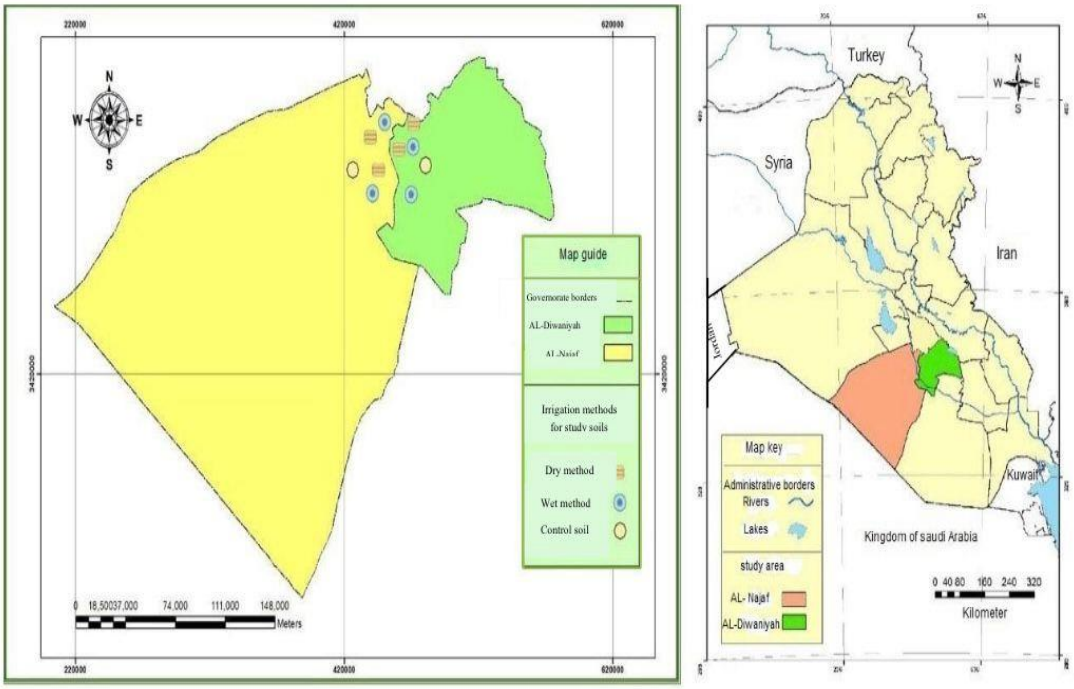


Figure (1): The locations of study area.

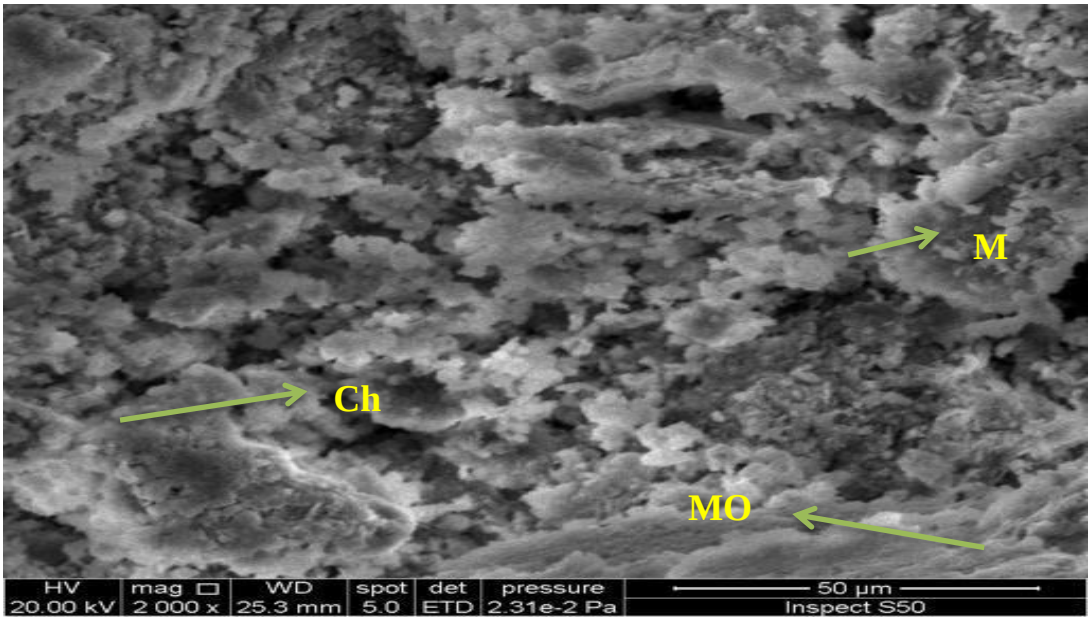
RESULTS AND DISCUSSION

SEM Inspection

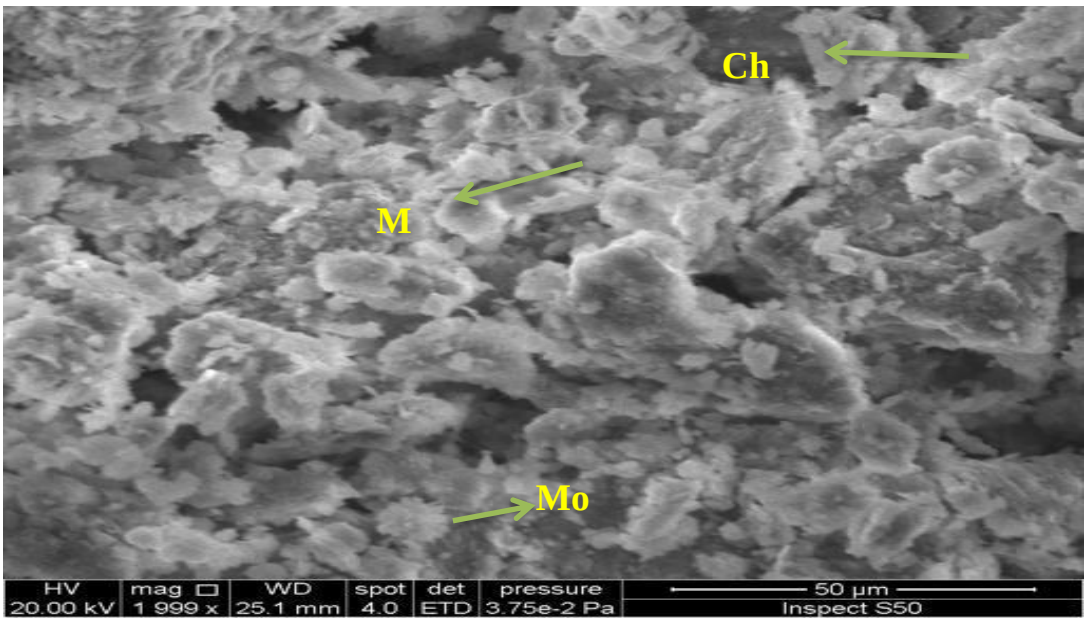
The SEM was used to studying the morphological features of clay minerals in the studied soils, to investigate the changes that occurred to the mineralogical structure of these minerals, due to the impact of the irrigation patterns used in the irrigation of the study soils.

The SEM of the clays sample at depth of 0-30 cm in Al-Najaf$_{(dry)}$ soil (Plate 1A) revealed that some of particles appeared as well-formed imperfect hexagonal shape, which confirms that these particles belong to the chlorite mineral. The scanning results in (plate 1A) showed that the chlorite particles appeared in different sizes, and some of their edges were exposed to weathering processes. These results are in agreement with finding of many studies **(Alam *et al*, 1999; Rajkumar *et al*, 2014; Perri, 2020 & Laird, 2001)**. Also, the lath-shaped, rounded, and semi defined flake edges that have been diagnosed in (plate 1A) were belong to the mica minerals, as diagnosed in many previous studies (**Bohor & Hughes, 1971; Dixon et al, 1977; Essa & Al-Sheikhly, 2001**). In addition, the results of scanning in (plate 1A) showed that some of mica edges appeared in pale color, which was completely surrounds the particle or part of it. These morphological features of mica edge particles can interpret into two possibilities. First, the pale color is result of exposing these particles to the weathering processes, perhaps within the locations of their sources, or during transport and sedimentation (**Meunier & Velde, 2004; Almashhadani & Al-Hasanay, 2023**). These results came in agreement with several studies (**Al-Dahi, 2009; Majeed, 2017**) which were conducted on Iraqi soils. While the second possibility is exposure of these particles to the weathering in current sites, as a result of the effect of alternating wet-dry cycles, these finding consistent with (**Shahad, 2021**) during his study of rice soils in some governorates of the Middle Euphrates in Iraq.

Results of (plate 1A) also, showed the presence of montmorillonite particles, which appeared as cloudy shaped fluffy mass flaks. While some of the montmorillonite particles appears as a thin, webby crust, and have a flat, perming morphology **(Islam *et al*, 2022).** Most of these particles were interstratified with the mica particles, which may indicate a stage of shifting the mica minerals towards the expanded 2:1 minerals group. In general, through the results of the (plate 1A), it can be observed that the presence of montmorillonite and mica minerals simultaneously has more abundance of montmorillonite than mica. Moreover, montmorillonite particles were found individually, and have large size compared to the size of other mineral particles in the sample. Results of scanning in (Figure 1A) also, reveled some particles have well-formed six-sided flakes (Hexagonal shape), with a prominent elongation in one direction. From our point of view these six-sided flake particles are 1:1 type antigenic (well-crystallized) kaolinite minerals, according to the diagnosis given by **(Dixon *et al*, 1977).**



A



B

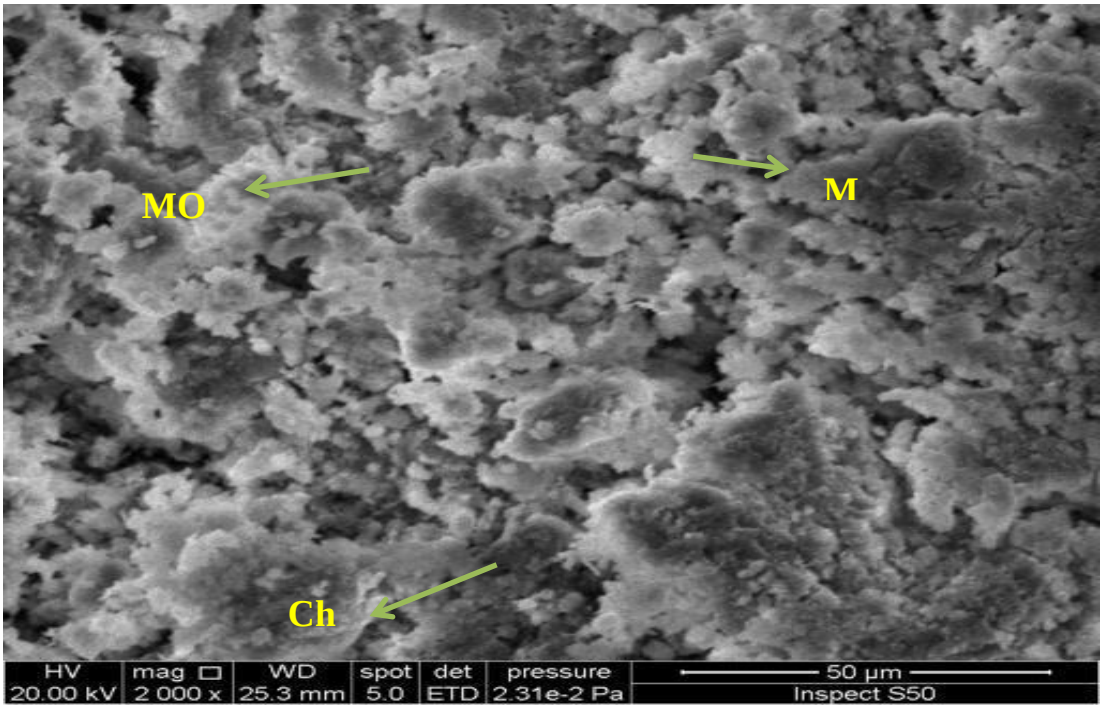
Figure (1): Scanning electron microscopy (SEM) images at magnification (X2000) of Najaf soil at depth of 0-30 cm, irrigated by, A. Dry method, and B. Wet method.
M = Myca, Mo = Montmorillonite, Ch = Chlorite

In (Figure 1B), which represent Al-Najaf$_{(wet)}$ soil, results of scanning showed, almost the same morphological features of clay minerals with some differences, the most important of

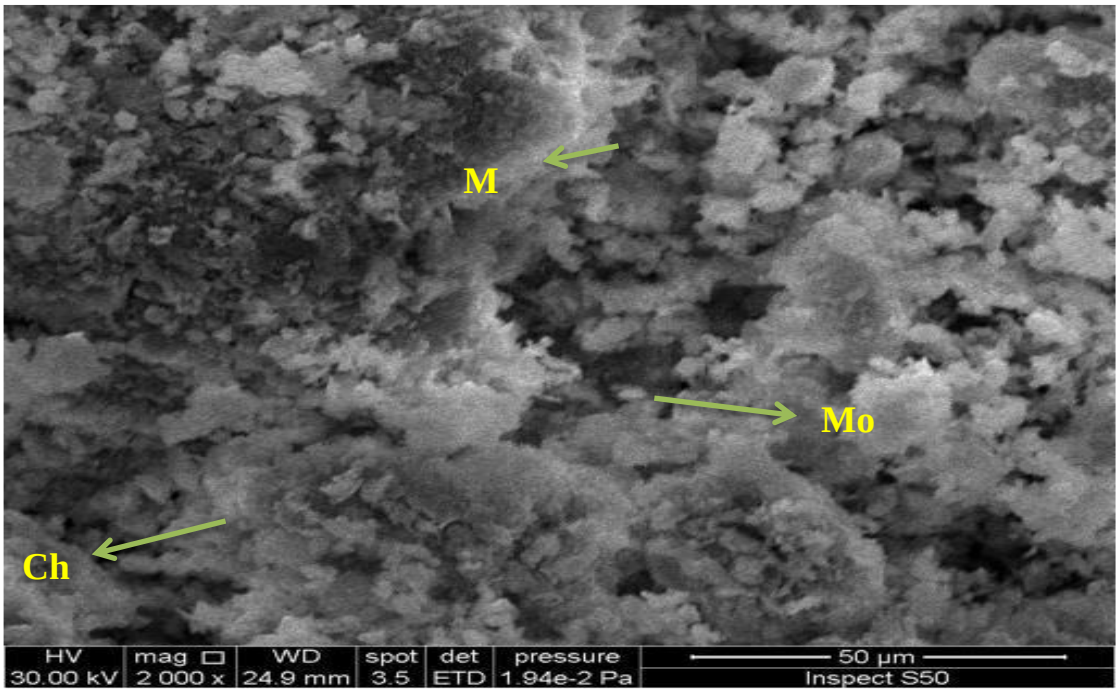
which is the size of the mineral particles, especially the size of montmorillonite particles, which seemed smaller in size than its counterparts in rice soil irrigated by dry method. It appears that the dry irrigation method, in which the soil is exposed to continuous cycles of wet-dry process, which encouraging the precipitation of Mg-hydroxide into Montmorillonite interlayers, which causes an increasing of surface area of mineral particles, and this increase depends on degree of filling for Mg-hydroxide **(Al-Watifi &Abbas Sabr Sarwan, 2012).**

The results of scanning in (Figure 2A, B) for the clay fraction of the Al-Diwaniyah$_{(dry)}$ soil, showed the presence of montmorillonite mineral particles, which appeared as cloudy shaped fluffy mass flaks. (Figure 2 A), and were found in two typs, the first overlapping with mica minerals, or in the type of individual particles secondly. (Figure 2A) also, shows the presence of mica minerals with weathered edges, which appeared in a pale color that distinguishes them from the surface of lath-shaped, rounded mica minerals. Further, the results of (Figure 2A) shows the presence of the chlorite mineral, which has an well-formed imperfect hexagonal shape.

The results of the scanning in (Figure 2B) of the clay sample in Al-Diwaniya$_{(wet)}$ soil, show the predominance of the cloudy-shaped montmorillonite mineral particles with indefinite edges, followed by the predominance of the mica minerals with lath-shaped, and the weathered edges of some of them, as these edges appeared in a pale color that distinguishes them from the surfaces of the mica minerals in the examined sample. In addition the montmorillonite particles in this sample, seemed smaller in size than its counterparts in rice soil irrigated by dry method.



A

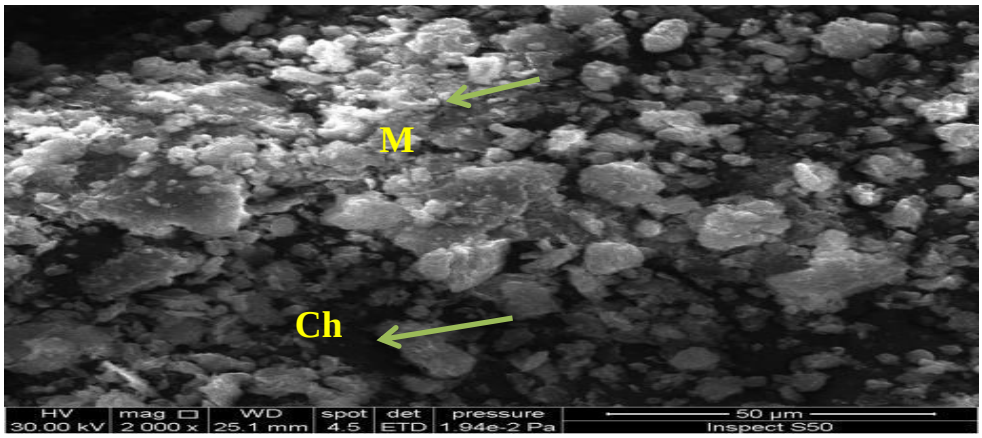


B

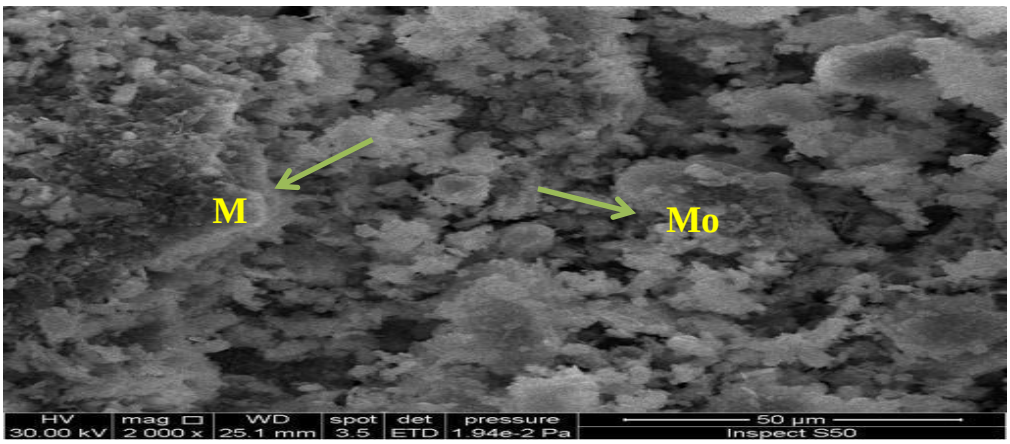
Figure (2): Scanning electron microscopy (SEM) images at magnification (X2000) of Al-Diwaniyah soil at depth of 0-30 cm, irrigated by, A. Dry method, and B. Wet method.
M = Myca , Mo = Montmorillonite , Ch = Chlorite

In order to improve that presumption, three samples of clay were chosen from depth of 0-30 cm, which is the depth as a most affected by the fluctuation of the irrigation patterns used to irrigate the study soils. Also, the selected samples were represented each of the control unexploited Al-Najaf soil, and the second sample was representative of the Al-Najaf$_{(wet)}$ soil. While the third sample, was for Al-Najaf$_{(dry)}$ soil. All selected clay samples were scanned by SEM under one magnification (1990-2000X). The aim of that selection was to study the effect of variation in irrigation patterns on the morphological features of the montmorillonite, because of the deposition of Mg-hydroxide within the interlayer of the mineral. The variation in the size of the montmorillonite particles according to our point of view was adopted as a basis for the occurrence of the deposition process of Mg-hydroxide layer within the interlayers of montmorillonite, based on what was reported by many studies (**Dubbin, 1995., Goldbery *et al,* 2000**). which were conducted in this regard. They showed that because of the deposition of the Mg-hydroxide layer within the interlayers of montmorillonite, the d-spacing of the mineral increases due to the expansion of the particles size. Accordingly, the results of scanning in (Figure 3 A, B, C) showed a clear variation in the size of montmorillonite particles within the soil clay samples that were examined. Where the clay particles of montmorillonite in control soil were recorded the smallest size (Figure 3A), followed by the size of clay particles in Al-Najaf$_{(wet)}$ soil (Figure 3B), while the montmorillonite particles in clay sample of Al-Najaf$_{(dry)}$ (Figure 3C), were exhibited in the largest sizes.These results were consistent with what the

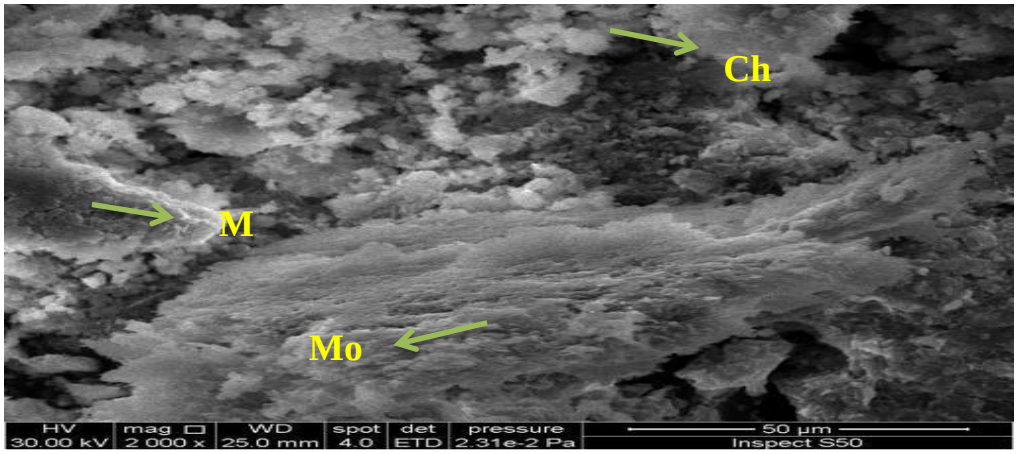
current study obtained from the results of the X-ray examination for the clay fraction of the study soils , which showed that the d-spacing of the montmorillonite varied according to the degree of filling of the Mg-hydroxide layer deposited within the interlayers of the montmorillonite, and the highest value of the d-spacing of montmorillonite containing an interlayer of Mg-hydroxide was recorded in Al-Najaf$_{(dry)}$ soil, accompanied by the retention of the montmorillonite diffraction at its d-spacing of 15.05 A^0 in the treatment of k-saturated and heated to the 350 and 550 ^{0}C, which reflects the effect soil subjected to successive of wet-dry cycles, which create suitable conditions for the Mg-hydroxide layer to prispetat within the interlayers of montmorillonite in the soils that are irrigated by the dry.



A



B



C

Figure (3): Scanning electron microscopy (SEM) at magnification (X2000) of Al-Najaf soil, A. control soil, B. irrigated by wet method, C. irrigated by dry method.
M = Myca, Mo = Montmorillonite, Ch = Chlorite

These results were identical to what was consisted with finding of (**Al-Watifi, 2012**) in his study of the phenomenon of chloritization within some of the Iraqi soils.

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