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BENTONITE AND SMECTITE-RICH SHALE DEPOSITS OF IRAQ

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Keywords: Bentonite; Volcanic ash; Smectite; Ca-montmorillonite; Iraq

ABSTRACT

Bentonite (*senso stricto)* is reported in the Mukdadiya Formation (Upper Miocene – Pliocene) in several localities within the Low Folded Zone of Iraq. The bentonites are beleived to have resulted from the alteration of volcanic ash in a fluvial system (lakes) environment. The mineralogy is dominated by Ca-montorillonite and quartz. The major chemical oxides are SiO_2, Al_2O_3, CaO and Fe_2O_3. The reserves were estimated by about 1.45 million tons, but the Na-activation processes showed little success and most of the reserves were exhausted in the native sulfur purification process at the Mishraq sulfur mines. On the other hand, the Late Maastrichtian – Danian phosphatic succession of the Iraqi Western Desert, include 5 to 6 smectite-rich shale beds, arranged in a transgressive cyclic sequences deposited in an open-subtidal reducing marine environment of the Maastrichtian Digma Formation (Safra Beds) and the Paleocene Akashat Formation (Traifawi Member, Danian). The mineralogy is dominated by smectites, rich in montmorillonite, with subordinate kaolinite, quartz, calcite, apatite, pyrite, palygorskite, chalcedony, opal-CT and organic matter. The dominant major chemical elements are Si, Al, Fe, Mg, Ti, Ca and P. Trace elements, normalized to average shale show high anomalies of Cd, Mo, Cr, Zn, V, U and Ni. The important factors controlling the mineral formation include intense chemical weathering in the source area, normal marine coupled with upwelling currents, precipitation of the authigenic phosphate-shale-silica facies association in a middle to outer system of shallow to deep marine environment within local inner to outer ramp semi-closed basin. Geochemical indices reflect the chemical weathering of the source rocks, which included metamorphic, felsic with a great influx of mafic rocks, and older sedimentary rocks of the Arabian Shield. Weathering, under warm and semi-humid climate, generated low lateritic pedogenic soils at the source area. The reserves were estimated by about 300 million tons in the Digma Formation deposits and about 23 million tons in the Traifawi Member deposits. The former is being mined for the past 25 years and used mainly as a drilling mud.

رواسب البنتونايت والسجيل الغني بالسمكتايت في العراق

ابراهيم قاسم محمد

المستخلص

يوجد البنتونايت (بمعناه الحرفي) في تكوين المقدادية (المايوسين الأعلى – الپلايوسين) في عدة مواقع ضمن نطاق الطيات الواطئة في العراق. يعتقد ان البنتونايت تكون من تحول الرماد البركاني في بيئة مائية (بحيرات) ويسود معدن المونتمورلونايت الكالسيومي والكوارتز على المكونات المعدنية وأكاسيد السيليكون والألمنيوم والكالسوم والحديد على المكونات الكيميائية. تم تقدير الاحتياطي بحوالي (1.45) مليون طن وبسبب صعوبة تنشيط البنتونايت بالصوديوم فقد تم استغلال معظم الاحتياطي في تنقية الكبريت الحر من الشوائب بعد تنشيطة بالحامض في مناجم كبريت المشراق. من جهة

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أخرى يتضمن التتابع الفوسفاتي لعصري الماستريختي الأعلى (تكوين الدگمة – طبقات الصفره) والدانيان (تكوين عكاشات – عضو الطريفاوي) في الصحراء الغربية العراقية على (5 – 6) طبقات من السجيل الغني بمعادن السمكتايت ترسبت في بيئة بحرية اختزالية تحت الى مدية. يحتوي السجيل على السمكتايت الغني بمعدن المونتمورلونايت فضلا عن نسب متفاوتة من الكاؤولين والكالسايت والأباتايت والبايرايت والبايغورسكايت والجالسيدوني والأوبال-سي تي والمواد العضوية. العناصر الكيميائية الرئيسية هي السيليكا والألومينا والحديد والتيتانيوم والكالسيوم والفوسفور والعناصر الشحيحة هي الكادميوم والموليبدينوم والكروم والزنك والفناديوم واليورانيوم والنيكل. تضافرت عدة عوامل جيولوجية في تكون رواسب السجيل الغني بالسمكتايت أهمها التجوية الكيميائية لصخور المصدر وظروف بحرية اعتيادية رافقتها تيارات بحرية صاعدة من الأعماق ساهمت في ترسب سحنات مترافقة من الفوسفات والسجيل والسيليكا في المنصتين الوسطى والخارجية لبيئة بحرية ضحلة الى عميقة مع وجود موقعي لأحواض شبه مغلقة ضمن المنصة. تشير الدلائل الجيوكيميائية الى التجوية الشديدة في صخور المصدر التي تكونت من صخور متحولة ونارية فلسباثية مع مساهمة من صخور قاعدية وصخور رسوبية قديمة في الدرع العربي والتي نتج عنها تربة لاتيريتية تحت ظروف مناخية دافئة وشبه رطبة. تم تقدير احتياطيات السجيل الغني بالسمكتايت بحوالي (300) مليون طن في تكوين الدگمة وحوالي (23) مليون طن في عضو الطريفاوي ويستعمل الأول ومنذ حوالي 25 سنة في تحضير سوائل حفر الآبار.

INTRODUCTION

Exploration for bentonite was made in the Qara Tappa vicinity (Himreen Mountain Range) on the basis of old (historic) workings in the area. The deposit, together with neighboring Zarloukh, Tayawi and Emgarin deposits and occurrences were investigated in the seventies by GEOSURV geologists (Zainal and Jargees, 1972 and 1973 and Al-Maini, 1975) and small reserves of bentonite were discovered and assessed in the Mukdadiya Formation (Upper Miocene – Pliocene).

Smectite-rich shale beds are the main lithological facies in the Late Maastrichtain – Danian phosphatic succession of the Iraqi Western Desert. The Digma Formation (Maastrichtian) and the Traifawi Member (Danian) of the Akashat Formation (Paleocene) are the main smectite-bearing rock units, developed west of the Hauran Uplift (Al-Bassam *et al.*, 1990). The pioneer work of Mohammad (1985) highlighted for the first time the presence of these smectite-rich shale deposits in the Western Desert of Iraq. His findings encouraged Iraq Geological Survey to carry out systematic mineral exploration in the area extending from Akashat in the North to Wadi Bashira in the South (Khudair *et al.*, 1988; Al-Bassam and Saeed, 1989; Al-Bassam *et al.*, 1991). Moreover, smectite- and palygorskite-rich deposits of the Western Desert of Iraq have attracted the attention of numerous researchers in Iraqi universities and in scientific research institutes (e.g., Ali, 1985; Al-Bassam and Al-Saadi, 1985; Hasson, 1986; Aba-Hussian, 1987; El-Khafaji, 1989; Al-Malah, 1988; Kas-Yunnan, 1989; Mohammed, 1988 and 1993; Mohammed and Jassim, 1992; Mohammed and Al-Rubies, 1995; Aswad *et al.*, 2000; Al-Haza'a, 2001 and Mohammed and Lawa, 2017).

This article provides an overview of the available information on bentonite and smectite-rich shale deposits of Iraq with special emphasis and new information presented for the first time by the author on the Maastrichtian – Danian smectite-rich shale deposits of the Iraqi Western Desert. The scarcity of data on the Neogene bentonites limited the data presented here on their mineralogy, geochemistry and origin. Whereas, the extensive work on the Cretaceous and Paleocene smectites are shown here in presenting detailed information, especially with respect to mineralogy, geochemistry and provenance. Moreover, these montmorillonite-rich smectites are the source of activated “bentonite” for the oil industry in Iraq for several decades now.

THE BENTONITE DEPOSITS

Bentonite deposits were discovered in the Himreen Mountain Range in the early seventies. Two deposits were investigated within the Mukdadiya Formation (Upper Miocene – Pliocene). These are Zarlukh and Qara Tappa. Both sites show some old workings and excavations. The bentonite is found as lenticular bodies in association with tuffa, sandstone and siltstone.

▪ Geological Setting of the Low-Folded Zone

The Low Folded Zone (Fig.1) is mostly covered by Neogene formations; mainly Fatha (Middle Miocene), Injana (Upper Miocene), Mukdadiya (Upper Miocene – Pliocene) and Bai Hassan (Pliocene – Pleistocene). Neogene clastic rock units, sometimes with anhydrite and carbonates, fill the synclinal plains in the SE. In the NW part, Early and Middle Miocene carbonates and anhydrites form the core of short anticlines. The prominent structures of Sinjar and Qara Chouq are generally built by Paleogene carbonates (Jassim and Buday, 2006). The area was the depocenter of the Neogene molasses. It was a subsiding area throughout the Mesozoic, Paleogene and Neogene (Jassim and Buday, 2006). The paleogeographic facies configuration was briefly described by Buday (1980) as follows:

– **Oligocene:** Area of no deposition (or erosion) in the north, neretic (reef – forereef) in the southern and northern flanks of the basin and basinal facies in the center. However, GEOSURV geologists discovered later that Oligocene rocks cover the Pila Spi Formation. (Eocene) almost everywhere in the Kurdistan Region and even in the Low Folded Zone (Sissakian and Al-Jibouri, 2012 and Sissakian and Al-Jibouri, 2014).

– **Early Miocene:** Evaporites (Dhiban Formation) in the middle and carbonaceous marine rocks without evaporites (Euphrates Formation) in the flanks of the depositional basin.

– **Middle Miocene:** Lagoonal evaporitic deposition (Fatha Formation) in most of the area.

– **Late Miocene – Pliocene:** Fluvial clastics, Bai Hassan Formation in the middle and Injana and Mukdadiya formations in the flanks of the basin.

▪ Characterization of the Bentonite Deposits

The bentonite deposits and occurrences occure in lenticular shape in association with tuffa and fluvial clastic sediments of sand and clay of the Mukdadiya Formation (Upper Miocene – Pliocene) they are exposed in a narrow NW – SE stretch parallel to the strike of the Himreen Anticline (Fig.1).

– **Qara Tappa Deposit:** Situated about 17 Km SW of Qara Tappa town within the Himreen Mountain Range. The deposit is part of the Mukdadiya Formation. The deposit, as described by Zainal and Jargees (1972) consists from bottom to top as basal gray sandstone, overlain by thin bed of marl, followed by gray medium grained sandstone. The main bentonite horizon occurs within the overlying (6 – 9) m thick marl bed, and consists of up to 2 m thick (average about 1 m), irregular and lenticular in form; overlain by white and light gray tuffaceous horizon. The mineral analysis showed up to 80% Ca-montmorillonite with impurities of illite, chlorite and quartz. The chemical composition is shown in Table (1). The reserves (Table 2) were estimated by about 0.4 m.t. on all categories (Zainal and Jargees, 1972).

– **Zarloukh Deposit:** Situated 18 Km west of Qara Tappa town. The investigated area is about 0.3 Km^2 extending about 1400 m along the strike (NW – SE). The wall rocks are composed of cross-bedded sandstones of the Mukdadiya Formation. The bentonite “bed” is composed of gray or brown montmorillonite-rich horizon, (0.5 – 1.8) m thick, overlain by 2.7 m thick gray or white sandy-clayey horizon, with montmorillonite admixture. The overlying bed consists of (1 – 3) m thick dark gray sandstone. The montmorillonite is Ca-based, and its content vary in the deposit from (3 – 93) %. The chemical composition is shown in Table (1) and the reserves (Table 2) were estimated by about 0.3 m.t. (Zainal and Jargees, 1973).

– **Emgarin – Tayawi Deposits:** Located within the NE limb of the Himreen Mountain Range, about 140 Km NE of Baghdad, along Kirkuk – Baghdad highway. The deposit is 1.5 Km^2 in area and includes two neighboring locations: Abu Gharib and Ausija. All of which are within the Mukdadiya Formation. The bentonite-bearing sequence consists of basal sandstone, dark gray and cross bedded, overlain by light brown claystone, which is topped by (0.8 – 3.2) m thick montmorillonite-rich horizon. The bentonite is brown, compact and light weight. The overlying unit consists of tuffaceous sandy material, white, current bedded and laminated, topped by siltstone, mudstone and sandstone. The mineral analysis showed (8 – 63) % montmorillonite content, with impurities of illite, kaolinite and quartz. The chemical composition is shown in Table (1). The reserves (Table 2) were estimated by 0.37 m.t. (Al-Maini, 1975).

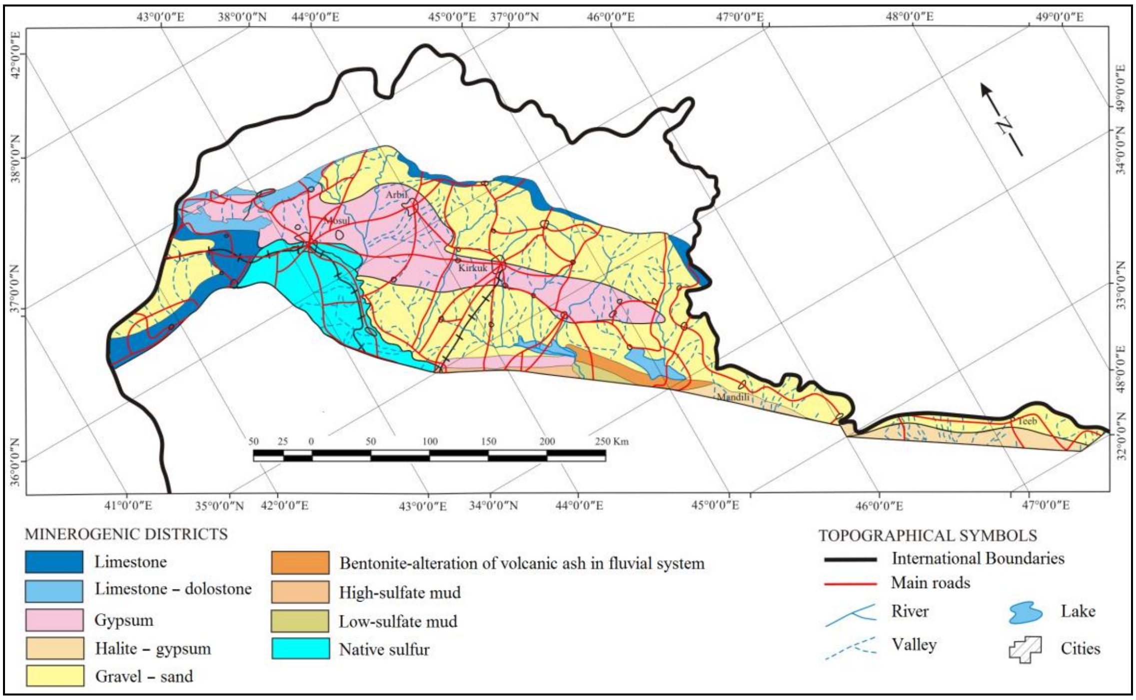

Fig.1: Minerogenic districts of the Low Folded Zone (after Al-Bassam, 2007)

Table 1: Chemical analysis of the investigated bentonite deposits (wt.%)

Oxides	**Zarloukh** [1]	**Qara Tappa** [2]	**Emgarin – Tayawi** [3]
SiO_2	57.79	57.47	57.30
Al_2O_3	16.53	19.63	16.00
Fe_2O_3	3.20	3.58	3.30
CaO	4.40	4.25	4.70
MgO	7.00	5.07	6.15
K_2O	0.12	n.a.	0.70
Na_2O	0.17	0.21	1.00
H_2O^+	10.30	10.51	9.50

[1]Zainal and Jargees (1973), [2]Zainal and Jargees (1972), [3]Al-Maini (1975)

Table 2: Bentonite reserves in the Mukdadiya Formation

Deposit	**Formation**	**Reserve C2 (ton)**	**Reserve C1 (ton)**	**Reserve B (ton)**	**Reserve A (ton)**	**Reference and report number (GEOSURV)**
Qara Tappa*	Mukdadiya		62141	120911	220212	Zainal and Jargees, 1972 (rep. no. 546)
Zarloukh*	Mukdadiya	440000	250000			Zainal and Jargees, 1973 (rep. no. 587)
Emgarin-Tayawi*	Mukdadiya	370000				A-Maini, 1975 (rep. no. 697)
Emgarin	Mukdadiya		268847			Al-Badri *et al.*, 1991 (rep. no. 1977)
Al-Awsagy	Mukdadiya		44718			Al-Badri *et al.*, 1991 (rep. no. 1977)

(*) Exhausted reserves

- **Genesis of the Bentonite Deposits**

Volcanism in the neighboring regions (NE of Iraq) was reported as a result of subduction of the Arabian Plate in the Late Neogene beneath Iranian Plate (Zainal, 1977). Airborne volcanic ash may travel for long distances, but finally it settles down and precipitates. Such event is recorded in several places within the fluvial clastics of the Mukdadiya Formation in Himreen Range. True “bentonite” is derived from the alteration of volcanic ash. Remnants of glass shards were identified in thin sections in the Qara Tappa bentonites (Zainal, 1977) and were taken as evidence on the origin of these bentonites, which were noticed in several other localities in the Low Folded Zone in association with tuffaceous rocks, within the Mukdadiya Formation, probably marking the same volcanic event. Among those localities, Na Salih village (west of Kifri town) is the outstanding one (Sissakian, 1978).

- **Bentonite Resources**

The limited extensions of the bentonite deposits and their lenticular character within the fluviatile Mukdadiya Formation have constrained the estimation of large reserves. In addition the difficulty in the Na-activation of the montmorillonite was not encouraging to continue exploration in other localities or to extend the reserves of the already explored ones. The total reserves have been estimated in two localities only: Qara Tappa and Zarloukh in the Himreen South Range. The total reserves in these two localities amounted to 1.46 million tons (Table 2); about 1 million tons of which is already consumed in the native sulfur processing at thr Mishraq sulfur mines.These bentonites proved to be excellent filter-aids after acid activation.

MONTMORILLONITE-RICH SMECTITE DEPOSITS

The Maastrichtian – Paleocene succession in the Iraqi Western Desert forms essentially a north – south trending exposed belt extending from the western rim of the Ga’ara Depression in the north and Wadi Traifawi and Wadi Bashira in the south (Fig.2). The Safra Beds of the Digma Formation (Maastrichtian) and the Traifawi Member (Danian) of the Akashat Formation (Paleocene) are the main smectite-rich shale-bearing rock units in the western part of the Iraqi Western Desert (Al-Bassam *et al.*, 1989a; Al-Bassam *et al.*, 1990). The deposits are originally black shales (Al-Bassam and Al-Haba, 1990), deposited in marine environment, influenced by frequent upwelling currents, in association with phosphorites and biogenic silica (Al-Bassam, 1982).

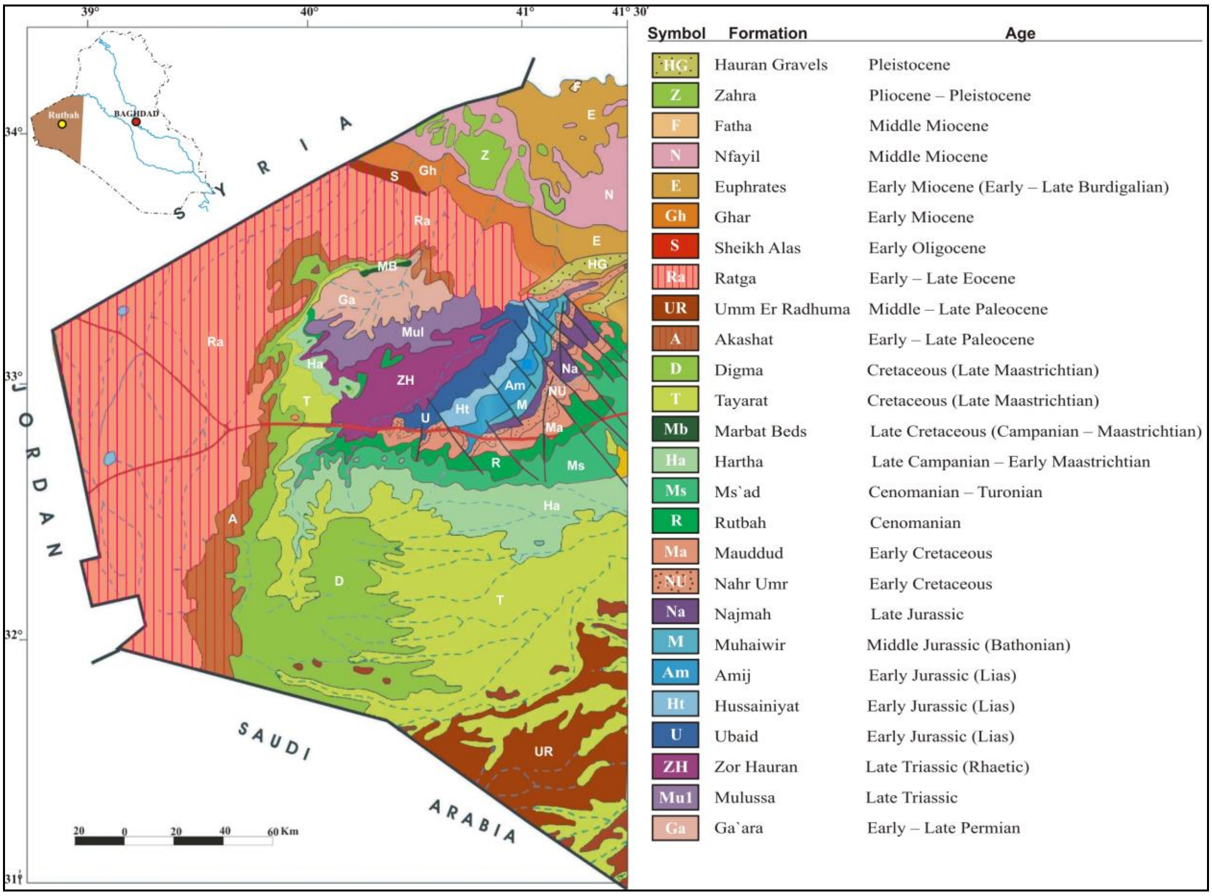

Fig.2: Geological map of the Iraqi Western Desert
(After Sissakian, 2000)

- **Geological Setting**

– **Tectonic Setting and Structure:** The Western Desert of Iraq is tectonically situated in the Inner Platform of the Arabian Shelf according to the recent scheme adopted by Iraq Geological Survey (Fouad, 2012). The region is characterized by two main sets of faults and several arches, the most pronounced of which is the Hail – Rutbah Arch. The main geomorphological feature in the area is the Ga'ara Depression, which is an oval dome underwent erosion. The available data point out that the Hail Arch is a broad up-warp that extends from the northern margin of the Arabian Shield northward to northwest Iraq affecting large parts of western Iraq, northeastern Jordan and southeastern Syria. The arch itself has a series of highs such as Rutbah, Ga'ara and Khleissia separated by several lows. The main fault systems in the Western Desert are described by Fouad (2007) as follow:

(1) The NW – SE Trending Faults; (Hauran Strike – Slip Fault System): These are NW – SE trending faults, well developed in the central part of the Western Desert. Al-Mubarak and Amin (1983) described these faults as a vertical horst and graben forming normal faults that are partly associated with horizontal displacement (Fig.2). **(2)** The N – S trending faults; (Nukhaib Fault System): These are N – S trending faults are restricted to the southern part of the Western Desert. Al-Mubarak and Amin (1983) described them as a set of normal faults with (80 – 150) Km long traces and clear vertical displacement. Some of these faults, however, have been deleted in the recent Geological Map of Iraq (Sissakian and Fouad, 2012).

– **Lithostratigraphy of the Main Shale-Bearing Units:** The Maastrichtian–Danian sequence in the western part of the Western Desert (Fig.3) is comprised of limestone, shale and phosphorite with occasional chert nodules and silica geodes associated with diatomaceous silica (porcelanite). Silicification is common in several horizons and the sequence show cyclicity (Fig.2). This sedimentary package is characteristic of almost all Tethyan phosphorite-bearing sequences of the region. Many controversies related to the previous stratigraphic nomenclature was corrected including the name “Jeed Formation” suggested by Buday and Hak (1980) to include the Upper Maastrichtian – Lower Paleocene sequences in one formation. The sequence was described by Jassim *et al*., (1984) and is recognized by Jassim and Buday (2006) to include two separate formations: the Digma Formation (Upper Maastrichtian) and the Akashat Formation (Lower – Upper Paleocene) which includes the Danian part of the sequence as the Traifawi Member. Whereas, Mohammed (1993) and Mohammed and Lawa (2017) suggested to include the Late Maastrichtian and Danian suceesions in one formation.

The stratigraphy of the Upper Cretaceous – Paleogene sequence was reconstructed after the detailed (1: 25 000 scale) mapping of the area west of the Hauran Uplift, extending from the Syrian borders to the Saudi Arabian borders with Iraq (Al-Bassam *et al*., 1990). The following information on the shale-bearing Maastrichtian and Danian sequences in the area is a summary based on the detailed geological survey of the phosphorite-bearing sequence in the Iraqi Western Desert (Al-Bassam *et al*., 1990) and the excellent review on the stratigraphy of the area provided by Sissakian and Mohammed (2007), which was highlighted recently by Al-Bassam (2017).

– **The Digma Formation (Maastrichtian):** Al-Bassam *et al.* (1990) described the Digma Formation, which they called the "Safra Beds", as white to creamy limestone, dolostone with phosphorite horizon and green to ocher papery shale, with oyster shell horizon. Hassan (1998) divided the formation in the Ga'ara vicinity into two units: **(1) Lower Unit** consists of white, creamy dolomicrite, with phosphorite horizon, overlain by green papery shale and topped by phosphorite horizon. **(2) Upper Unit** consists of oyster rich limestone alternated with marl horizons. Southwards the fossiliferous limestone increases in thickness and passes laterally to shelly limestone, with chert nodules and geodes in the middle and upper parts of this unit. Al-Haza'a (2001) divided this sequence in the Ga'ara – Akashat area into three parts: The lower part consists of dolostone with phosphorite horizon and porcelanite at the base. The middle part consists of ocher marl, occasionally with oysters, whereas the upper part consists of oyster rich limestone (lopha bed) which alternates with dolostone. Southwards the upper shelly part increases in thickness and passes to bivalves-rich limestone.

– **The Akashat Formation (Paleocene) – Traifawi Member (Danian):** The Akashat Formation is recently added to the stratigraphic column of Iraq (Jassim *et al*., 1984). Formerly, the succession was considered as Umm Er Radhuma Formation (Bellen *et al*. (1959). However, the differences in lithology, facial development and paleogeographical distribution between Akashat and Umm Er Radhuma Formations were recognized by Bellen *et al*. (1959) and Al-Naqib (1960). Buday (1980) and Buday and Hak (1980) called it the phosphatic facies of the Umm Er Radhuma Formation, Jassim *et al*. (1984) called it as Akashat Formation and Al-Bassam and Karim (1997) announced it officially as the Akashat Formation. It is worth to mention that GEOSURV geologists are using the name Akashat Formation instead of Umm Er Radhuma Formation since 1990 in the western part of the Western Desert of Iraq. The Akashat Formation is divided into three members (Al-Bassam *et al*., 1990); these are from older to younger: Traifawi, Hirri and Dwaima. The Traifawi

Member contains smectite-rich shale at the lower part and consists of cyclic alternation of lime mudstone, shale and phosphorite. The cycle, which is repeate (2 – 3) times, consists of coarse grained phosphorite, green to ocher, fissile, shale or marl, followed by creamy, siliceous lime mudstone, usually with small chert nodules, and marked by Thalassinoides burrows in firmground at the upper surface terminating each of the cycles (Mohammed and Aba-Hussain, 2018).

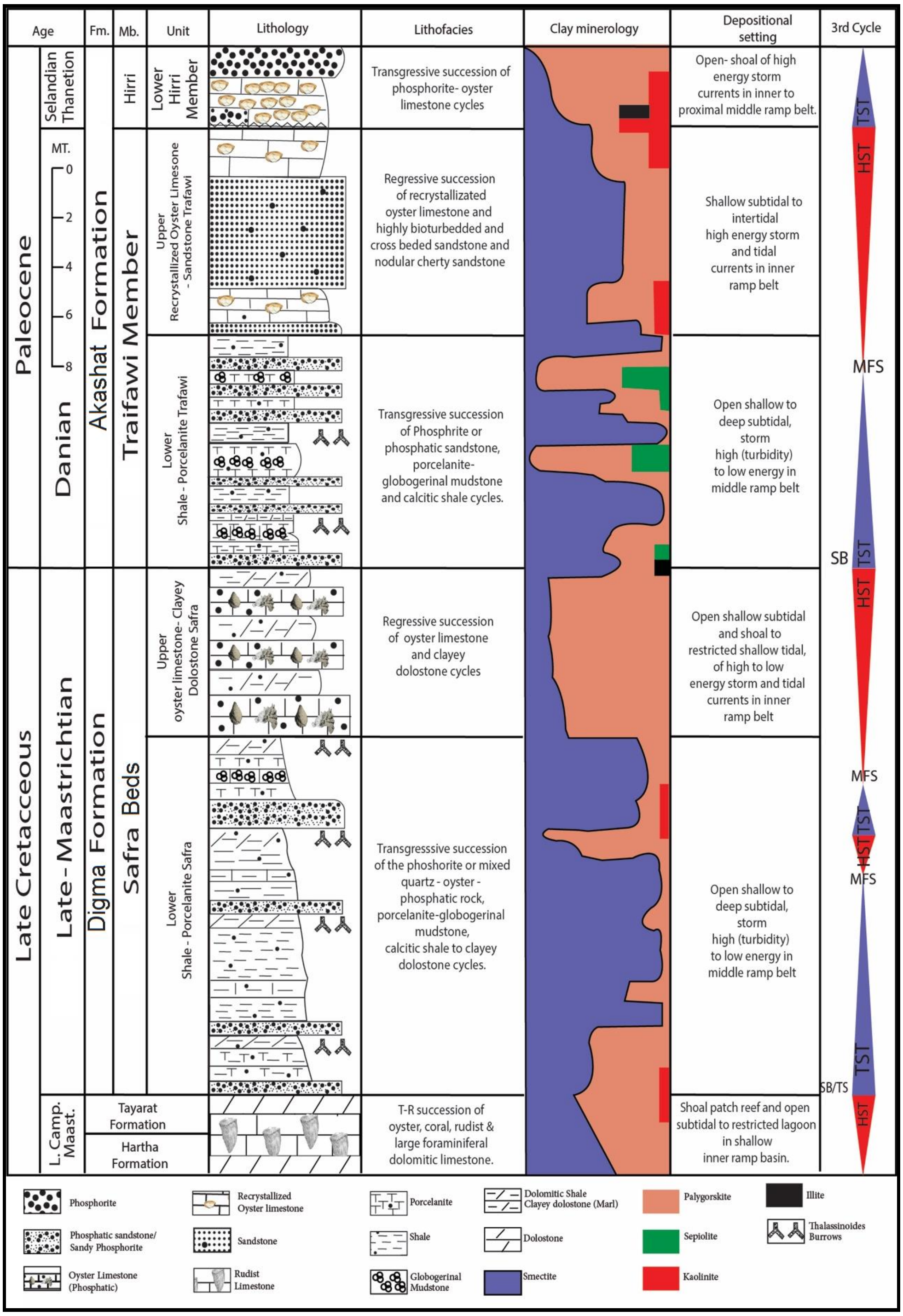

Fig.3: Lithostratigraphy, clay mineralogy, depositional setting and 3rd cycles subdivisions of the Late Maastrichtian – Danian succession, H3 area, west of the Rutbah town (present work)

MINERALOGY

▪ Bulk Rock Mineralogy

The mineral assemblages identified from optical microscopy, X-ray diffraction (XRD) and infra red spectroscopy (IR) include calcite, dolomite, quartz-chalcedony, opal-CT, apatite (francolite), organic matter and clay minerals as the main mineral consistuentds. Minor to trace amounts of gypsum, anhydrite, halite, pyrite, iron oxides and locally metatyamunite (secondary uranium mineral). The clay minerals in the shale beds consist mainly of smectitic in association with kaolinite, palygorskite and locally sepiolite, and illite in minor amount.

▪ Heavy Minerals

The heavy minerals (HM) composition is rich in tourmaline – staurolite assemblage mixed with different proportions with rutile and zircon as well as minor to trace amount of kyanite, chlorite, and hornblende of rounded to subrounded and less abundant angular and prismatic shape and Some HM grains shows pits and corroded outlines. The heavy mineral assemblage indicates mixed low to medium grade metamorphic rocks with contribution from felsic and mafic igneous rocks of proximal and distal provenance.

▪ Clay Minerals

– **XRD and IR:** Smectite peaks in the XRD and IR analysis show that smectite-rich smectite/ illite mixed layers is the major clay mineral and consists of (85 – 100) % smectite layers, mixed with appreciable amount of discrete smectite. The XRD main peak of smectite shows at (14.7) Å, which indicate that the studied smectite is Ca or Mg rich or Ca-Mg type. Locally in some scattered samples, especially those in the succession near Akashat and west of the Ga’ara Depression, where there is a clear halite association, the basal peak appeared at (12.6 – 12.8) Å, indicating the formation of Na-type smectite. The Mg-saturated samples show the basal reflection appear at (15) Å, expanded by glycolation to (16.9) Å and collapsed by heating to (10) Å (Fig.4). The XRD pattern shows the basal reflection characterized by non-integral case for the (001) reflection, which indicate that the studied smectite is smectite-rich illite – smectite mixed layer, therefore, it is not a pure smectite which is characterized by integral d-space (Srodon, 1984).

The IR bands (Fig.5) are characterized by a broad band of (OH) in 3622 cm, which also indicates the presence of smectite-rich random illite – smectite mixed layer in addition to discrete smectite (Fig.6). Li -200 test and the (060) reflection, which appear at (1.51 – 1.50) Å, indicate that the smectite is dioctahedral montmorillonite-rich montmorillonite-beidellite lattice type, because it has tetrahedral and octahedral substitutions. This is supported by the IR testing which indictes that the smectite is montmorillonite, whose absorption spectrogram is similar to that of the Cheto-type (Russel, 1987). Biscaye’s index displays medium crystallinity with a trend in crystallinity increase with seaward and transgressive succession and decrease shoreward and regressive succession associated with increase in the palygorskite content. The crystallinity of the studied smectite samples range between 0.28 in the Ga'ara area to 0.85 in the Traifawi area. In the Traifawi area, the clay samples have about (60 – 80) % smectite and very low palygorskite content (less than 10%). The XRD and IR analyses confirm the presence of kaolinite in some samples and reach up to 21% (mean = 5%).

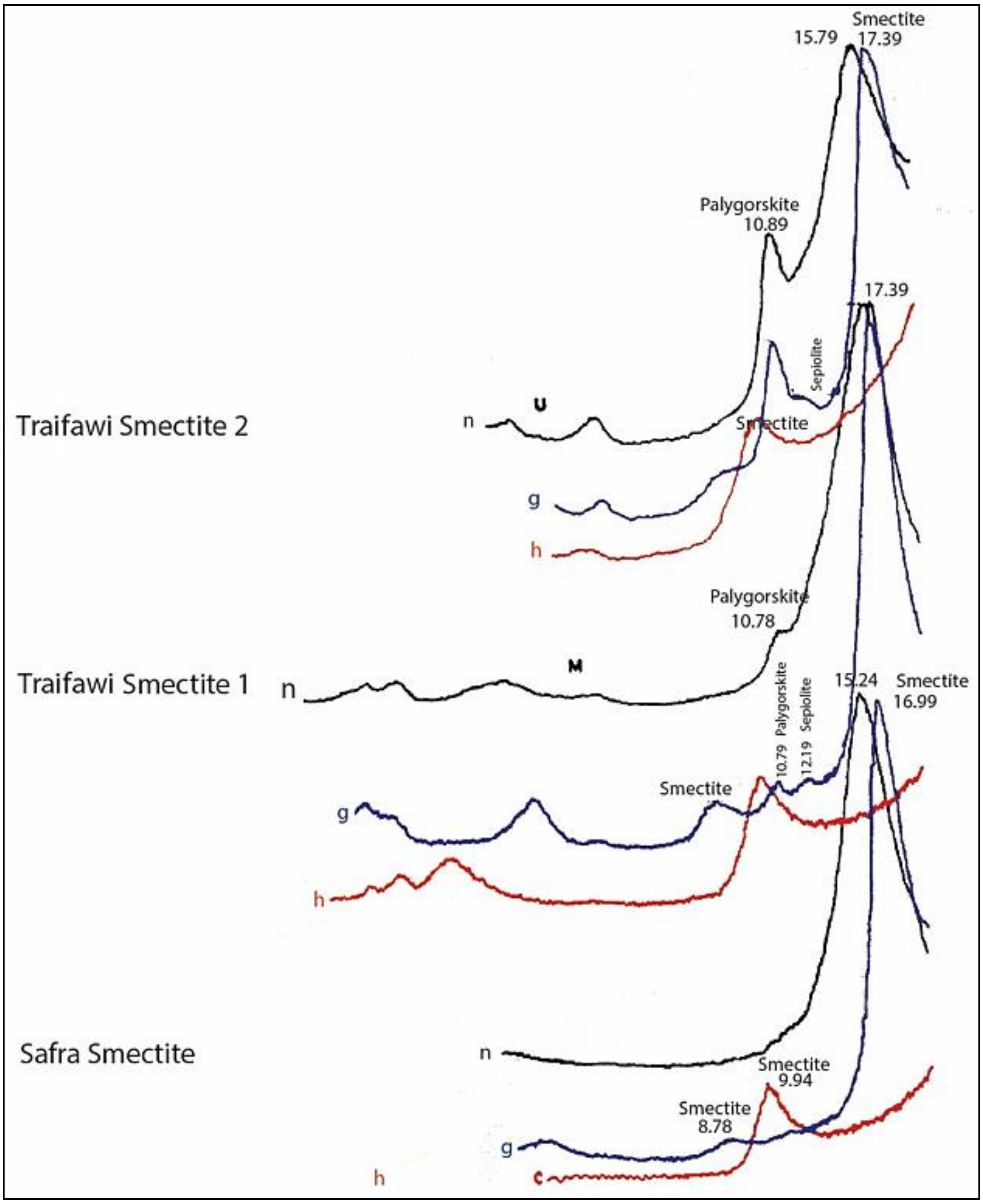

Fig.4: X-ray diffractograms of the <2 micron fraction oriented smears of the Safra and Traifawi smectite samples.
n: untreated, g: glycolated and h: heated at 550 °C

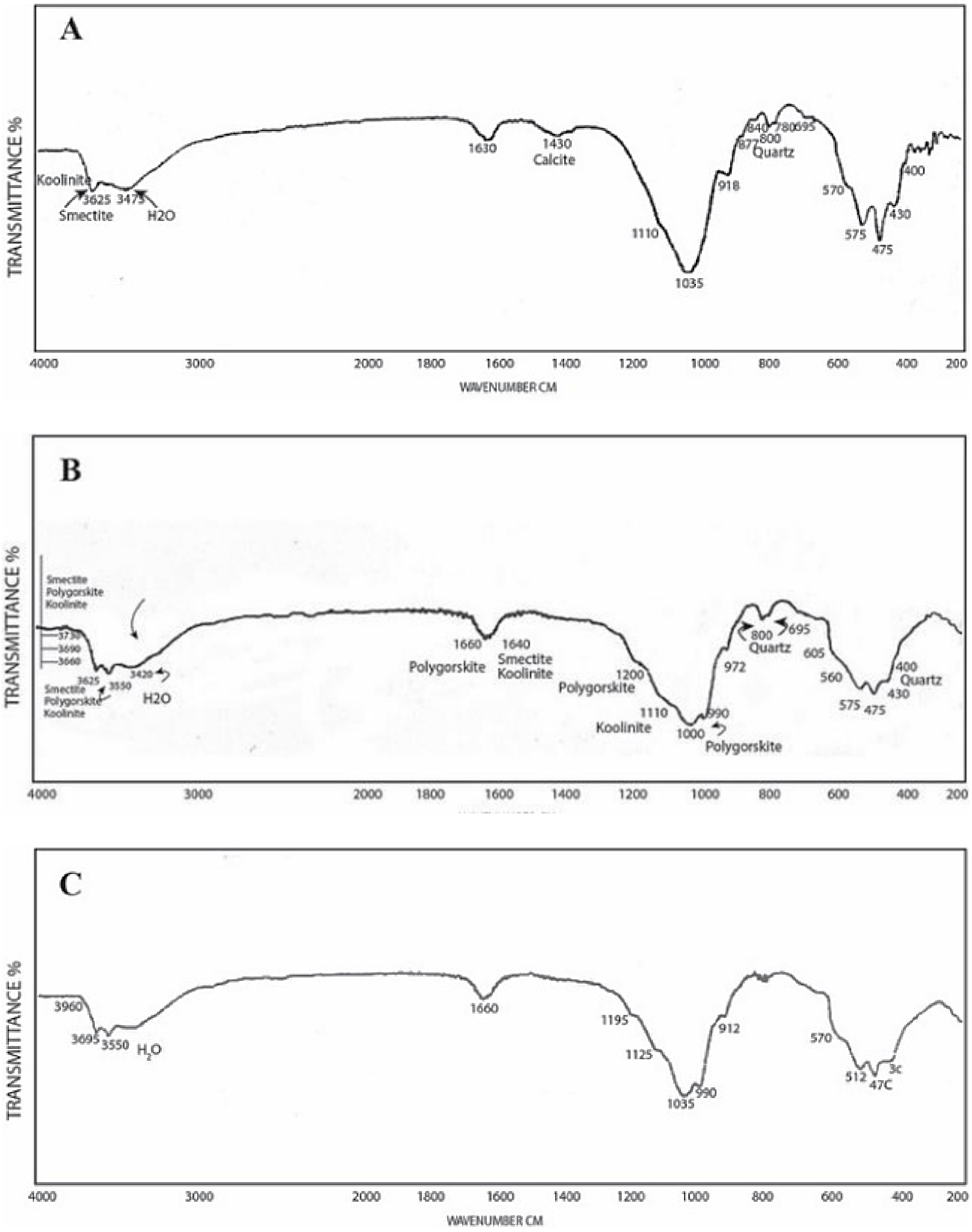

Fig.5: IR Spectra of <2 micron fraction of smectitic from the studied shale. **A)** Whole smectite-rich shale of the Safra Member (south of H3 area); **B)** Traifawi Member (south of H3 area); and **C)** Safra Beds (north of H3 area)

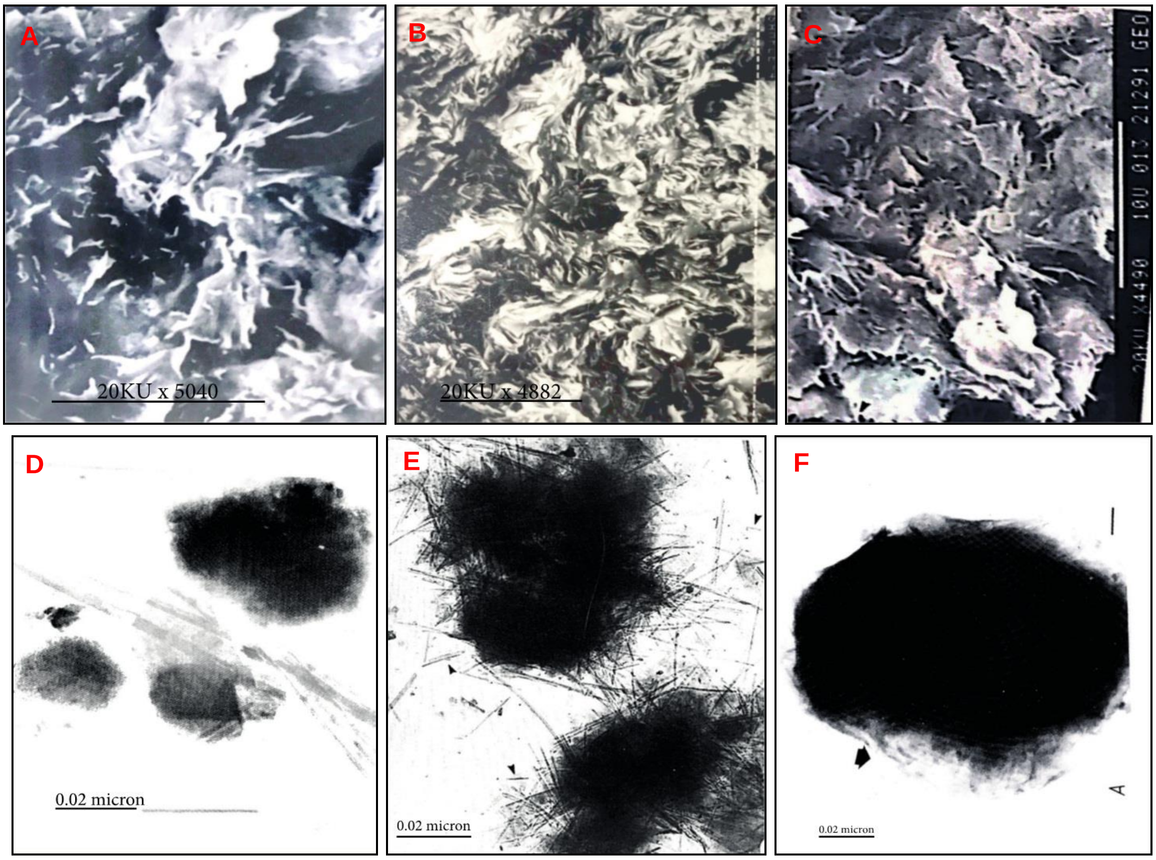

Fig.6: SEM and TEM images of the studied smectites: A, B and C are SEM images show foliated undefined outline flake of smectite and fibrous palygorskite and sepiolite. D, E and F are TEM images show smectite plates transformed to palygorskite, where the short needle-like fibres in the perepheries sprout out from the smectite which is still unaltered at the core

– **High Magnification Petrography:** Some of the smectite-rich samples were examined by SEM and TEM. The SEM images (Figs.6A – C) reveal a three dimensional topography of the smectite and associated palygorskite clays. The smectite grains are present in the form of flakes or plates with very thin, irregular with curled edges or wings structure. The texture of the smectite grains in these images shows the characteristic features of the detrital smectite transported from continental soil (Chamly, 1989). While the palygorskites show their fibrous shape, developed on the outer boundary of the smectite flakes (Fig.6F). The fibrous palygorskite texture indicates typical authigenic origin (Singer, 1981; Chamly, 1989 and Knidiri *et al*., 2014). In the earlier stage, palygorskite, identified as short acicular fibrous, spread on smectite outer rim and the parent smectite stays in the core (Fig.6E).

The TEM micromorphology (Figs.6E – F) shows typical film of dark smectite color, because of electron-opaque crystal aggregates. Smectite grains are anhedral rounded and with unclear outer boundary. Again TEM images indicate the smectite grain micromorphology give the characteristic features of the detrital origin (Chamly, 1989). Through the same image the bundles of the palygorskite fibres can be clearly noticed. They are euhedral and have straight edges that indicate the authigenic formation.

GEOCHIMSTRY

The chemical analysis of the bulk- rock samples (Tables 3 and 4) of the Safra and Traifawi smectitic-rich shales show the domainant composition of aluminosilicates, carbonates and phosphates expressed by the oxides (SiO_2, Al_2O_3, Fe_2O_3, MgO, TiO_2, CaO and P_2O_5), which match the XRD analysis. Vertical and lateral distribution of the smectite-rich shale in the study area display wide range in Al_2O_3 content, ranging from 11.13 to 17.28 wt.% (mean = 15.50 wt.% for the Safra Beds and 13 wt.%. for the Traifawi Member) (Table 3). Fe_2O_3 contents range from 3.14 wt.% to 5.3 wt.%. (mean = 4.44 wt.%). TiO_2 and P_2O_5 contents have mean values of 0.65 wt.% and 1.14 wt.% respectively, whereas, the CaO contents show a large variations among the studied samples, ranging from 2.8 wt.% to 13.86 wt.% (mean = 6.56 wt.%) and MgO, like CaO, has a wide range from 2.94 wt.% to 8.6 wt.% (mean = 8.6 wt.%). The variation in Ca and Mg oxides is attributed to the high smectite proportions in the lower parts of the shale beds and the increase of calcite and dolomite upwards. K_2O and Na_2O are generally low in most samples with average of 0.53 wt.% and 0.5 wt.% respectively.

Table 3: Chemical composition of major elements (in wt.%) and trace elements (in ppm) in the Safra Beds shale (Maastrichtian) and Traifawi Member shale (Danian)

	Maastrichtian Shale			Danian Shale			Average Composition	PAAS	UCC
Oxides	**Min**	**Max**	**Mean**	**Min**	**Max**	**Mean**			
SiO_2	50.89	62.19	56.46	46.2	60.86	52.9	54.68	62.8	64.92
Al_2O_3	13.92	17.28	15.50	11.13	16.14	13.4	14.45	18.9	14.63
Fe_2O_3	4.42	5.33	4.87	3.21	4.91	4.0	4.44	6.5	4.42
CaO	2.80	10.37	6.22	3.38	13.86	6.9	6.56	1.3	4.12
MgO	2.94	4.12	3.30	3.2	8.6	5.2	4.25	2.2	2.24
Na_2O	0.40	0.65	0.55	0.19	0.81	0.62	0.59	1.2	3.46
K_2O	0.51	0.68	0.58	0.19	0.70	0.48	0.53	3.7	3.45
P_2O_5	0.49	0.71	0.57	0.54	2.2	1.7	1.14	0.16	0.15
TiO_2	0.68	0.88	0.78	0.39	0.64	0.52	0.65	1	0.52
Trace-elements									
Mn	39	140	81	46	80	59	70	850	237
Zr	83	185	129	41	263	114	122	160	35
Cr	152	606	296	307	1271	741	519	90	26
Nb	10	26	19	10	22	16	18	18	20.7
Y	7	21	13	10	28	18	16	26	18.6
Ni	31	135	69	56	165	100	85	86	52
Zn	79	640	238	171	664	405	322	95	53
V	111	413	211	50	440	209	210	130	12
Co	4	20	10	24	8.43	58	34	19	14
Cu	11	15	13	22	51	36	25	45	316
Sr	161	200	177	301	413	314	246	300	2.5
U	20	37	26	101	10.2	5	16	3.70	17
Cd	ND	ND	ND	40	107	78	78	0.3	---
Pb	ND	ND	ND	5	29	12	12	20	1.4
Mo	ND	ND	ND	7	70	32	32	2.6	---

ND: Not detected

Table 4: Previously reported chemical analyses of major elements (wt.%) and smectite content (%) of the Safra and Traifawi shales

Oxides	Safra-1	Traifawi-2	Traifawi-3	Safra-4	Traifawi-5	Traifawi-6
Al_2O_3	15.5	13.4	15.1	15.67	15.7	17.94
SiO_2	56.46	52.9	54.43	56.77	56.8	60.17
Fe_2O_3	4.87	4	4.42	5.12	5.1	6.02
CaO	6.22	6.9	5.4	4.48	7.5	0.66
MgO	3.3	5.2	4.62	3.42	3.5	4.97
Na_2O	0.55	0.62	0.593	1.11	1.1	0.04
K_2O	0.58	0.48	0.67	0.6	0.6	0.42
P_2O_5	0.57	1.7	2.05	0.65	0.7	0.14
TiO_2	0.78	0.52	0.54	ND	ND	0.33
SO_3	ND	ND	ND	ND	ND	ND
L.O.I.	ND	ND	10.02	9.49	9.5	8.45
Smectit %	80	70	79.7	79	78.8	95

ND: Not detected; **1)** Safra Shale (Mohammed, 1988, unpublished data); **2)** Traifawi Shale (Mohammed, 1988); **3)** Traifawi industrial beds (Mohammed, 1993); **4)** Safra industrial beds (Al-Bassam and Majid, 1990); **5)** Traifawi high grade shale (Al-Bassam *et al*., 1989); **6)** Carbonate free Traifawi shale, washed with 10% acetic acid (Mohammed, 1993).

The average contents of the major elements (Table 3) and oxide ratios (Table 5) are compared with Post-Archen Australian Shale (PAAS) of Turekian and Wedepohl (1961) and Upper Continental Crust (UCC) of Wedepohl (1995). This comparison shows higher concentration of CaO, MgO and P_2O_5, lower concentration of Na_2O and K_2O and close concentrations of SiO_2, Al_2O_3, Fe_2O_3 and TiO_2 relative to the PAAS and UCC. Based on the geochemical classification diagram of Herron (1988), the studied shales are classified as Fe-Shale type. In this diagram most samples fall near the boundary of the shale and Fe-sand (Fig.7).

The trace elements shown in Table (3) show their concentrations are decreasing in the order Cr > Zn > Sr > Zr > V > Cd > Ni > Mn > Co > Cu > Mo > Nb > Y > U > Pb. The Trace elements were normalized in comparison to Average Shale (Turkian and Wedepohl, 1961; Taylor and McLennan, 1985 and 1991) (Fig.8), and UCC and PAAS (Table 3). The results reveal higher concentration of the chalcophile trace elements Cd, Mo, Cr, Zn, V, U and Ni in the studied shales, which are known as elements enriched in black shales, whereas, the rest of the trace elements are lower in concentrations, except Zr which is higher in the UCC and lower than its concentration in the PAAS.

Table 5: Significant oxides ratios in the studied shales compared to world average shale

Ratio	Safra	Traifawi	PAAS	UCC
Al_2O_3 / SiO_2	0.275	0.264	0.300	0.225
Al_2O_3 / Fe_2O_3	3.183	3.255	2.908	3.310
Al_2O_3 / Na_2O	28.18	24.491	15.75	4.22
Al_2O_3 / K_2O	26.72	27.264	5.108	4.24
Al_2O_3 / TiO_2	19.87	22.23	18.9	28.13

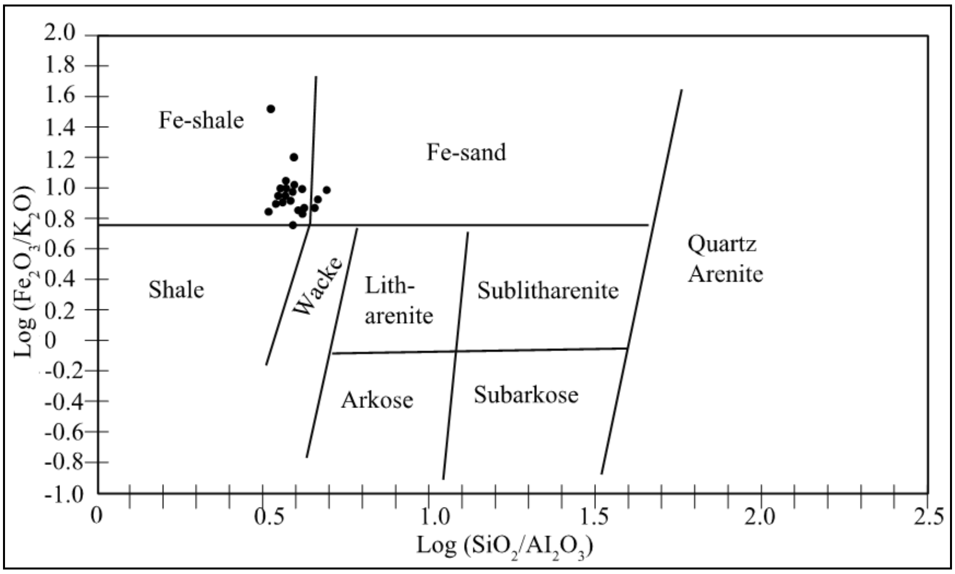

Fig.7: Geochemical classification of the studied Late Maastrichtian – Danian shale, using log (SiO_2/ Al_2O_3 vs. log (Fe_2O_3/ K_2O) diagram of Herron (1988)

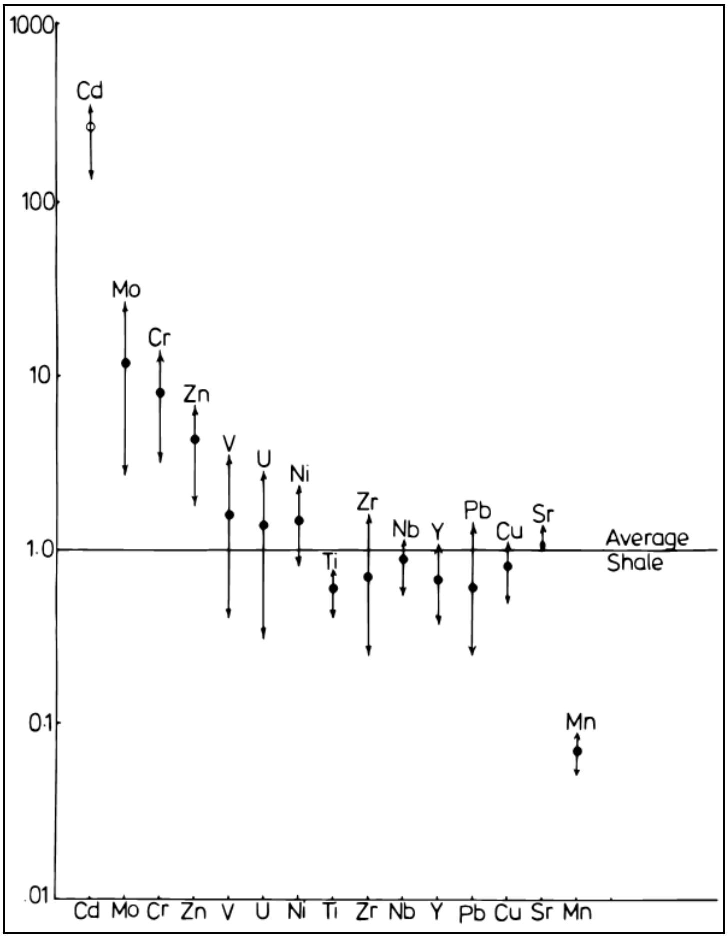

Fig.8: Distribution pattern of trace elements in the Maastrichtian and Danian shales normalized to Average Shale (Turkian and Wedepohl, 1961)

- **Weathering and Paleoclimate**

The changes and modifications in the chemical composition of the mobile and immobile elements that occur in the source rocks reflect the degree of the intensity of weathering, the type of prevailing climate and to a lesser extent the type of source rock. Chemical weathering intensity has a strong impact on the chemistry of the siliciclastic products (Floyed *et al.*, 1991 and McLennan, 1993) and crystallization of the clay mineral assemblages (Nesbitt and Young, 1984, 1989 and 2004). These geochemical parameters (Table 6) convey a message to the sedimentary succession including the degree of weathering and paleoclimatic conditions at the source area.

The Chemical Index of Alteration (CIA; Nesbitt and Young, 1982), applied to estimate the degree of source rock weathering, is based on the molecular proportions of Al_2O_3, Na_2O, K_2O and CaO. In this equation CaO* represents the CaO fraction in source rock detritus, and because the studied shales contain a large amount of total carbonate, mainly from marine calcite and apatite and locally dolomite, gypsum and anhydrite, therefore, the quantity of CaO* will be equal to the amount of Na_2O as an approximation (McLennan, 1993). As for Na_2O, all samples containing chlorine ratio of sodium equivalent to precipitation of halite were neglected to avoid any error. Furthermore, to complement the results, other indices are used, including the Plagioclase Index of Alteration (PIA), the Chemical Proxy of Alteration (CPA) and A-CN-K triangular diagram of Nesbitt and Young (1984 and 1989). The CIA was calculated: $CIA = [Al_2O_3/ (Al_2O_3 + CaO^* + Na_2O + K_2O)] \times 100$ in molar concentration (Nebsitt and Young, 1982). The CIA values (Table 6) vary between (83 – 89) % (mean = 86%) in the Safra shale and between (81 – 91) % (mean = 86%) in the Traifawi shale.

The CIA values were compared with the A-CN-K diagram (Fig.9), calculated as molar proportions of $Al_2O_3 – (CaO^* + Na_2O) – K_2O$. Most samples fall in the boundary between the intermediate and strong weathering field and some are showing intermediate and strong weathering. They plot mostly over the smectite – illite line and towards the Al_2O_3 apex near A-CN side and parallel to the ideal weathering trend (IWT), suggesting steady state weathering conditions (Fathy *et al.*, 2017; Nesbitt and Young, 1984) (Fig.9). The average of the CIA and A-CN-K plot locations of the PAAS Shale (Nesbitt and Young, 1984) are lower than those in the studied shale samples. The PIA value was calculated as: $PIA = [(Al_2O_3 – K_2O)/ Al_2O_3 – K_2O + CaO^* + Na_2O)] \times 100$ (Fedo *et al.*, 1995) and $CPA = (Al_2O_3/ Fe_2O_3) \times 100$ (Buggle *et al.*, 2011).

The PIA values in the Safra shale range between (87 – 92) %, (mean = 89%) and in the Traifawi shale range between (88 – 98) %, (mean = 95%), whereas, the CPA range between (93 – 96) % (mean = 95%) in the Safra shale and (91 – 99) % (mean = 95%) in the Traifawi shale. These values suggest strong weathering in the hinterland rocks according to Fedo *et al.* (1995) who classified the weathering intensity, based on the proxies CIA, PIA and CIW (Chemical Index of Weathering; Harnois, 1995), as: low weathering (~ 60%), moderate weathering (~ 60 – 80 %) and intensive weathering (>80%). On the other hand K_2O/ Al_2O_3 ratio (Cox *et al.*, 1995) of the studied shale is less than 0.046, whereas in the alkali feldspar it ranges between (0.4 – 1), in illite about 0.3, and <0.3 in clay minerals, which indicate the dominance of K-poor clay minerals.

Table 6: Major elements analysis and geochemical indices of the Maastrichtian and Danian smectite-rich shale (industrial beds)

A. Maastrichtian smectite rich-shale samples (Traifawi and Wadi Al-Jandali areas)

Samples **Oxides**	**M.A1**	**M.A2**	**M.A3**	**M.A4**	**M.A5**	**M.B1**	**M.B2**	**M.B3**	**M.B4**	**M.B5**	**Avag**
SiO_2	50.82	62.19	62.12	59.21	55.61	53.69	51.23	59.89	53.18	56.62	56.46
Al_2O_3	13.92	15.96	15.95	16.52	15.58	15.29	14.35	17.28	14.82	15.39	15.50
Fe_2O_3	4.61	4.68	4.98	5.08	4.88	4.72	4.42	5.32	5.31	4.72	4.87
CaO	10.37	3.56	3.70	4.2	7.01	8.41	10.37	2.8	6.86	4.90	6.22
MgO	3.01	3.18	3.17	3.11	2.94	3.07	3.73	3.23	3.48	4.12	3.30
Na_2O	0.53	0.56	0.64	0.40	0.62	0.40	0.60	0.41	0.61	0.63	0.55
K_2O	0.65	0.53	0.51	0.57	0.53	0.57	0.51	0.65	0.68	0.62	0.57
P_2O_5	0.62	0.60	0.49	0.57	0.50	0.50	0.59	0.52	0.71	0.62	0.57
TiO_2	0.70	0.81	0.75	0.83	0.81	0.69	0.68	0.88	0.87	0.80	0.78
CIA*	83	85	86	89	86	89	85	89	84	85	86
PIA*	88	87	88	92	88	92	87	92	87	94	89
CPA*	94	94	94	96	94	96	93	96	94	95	95
A(Al_2O_3)	84	85	86	90	86	89	85	89	84	87	87
CN(CaO+Na_2O)	11	12	12	7	11	8	12	7	12	9	10
K (K_2O)	5	3	2	3	3	3	3	4	4	4	3
Al_2O_3/TiO_2	20	20	21	20	19	22	21	20	17	20	20

B. Danian smectite-rich shale samples (Traifawi area)

Samples **Oxides**	**D.A1**	**D.A2**	**D.A3**	**D.A4**	**D.A5**	**D.A6**	**D.A7**	**D.A8**	**D.A9**	**D.A10**	**D.A11**	**D.A12**	**Avag**
SiO_2	54.98	59.92	60.86	52.82	44.56	58.64	46.2	46.26	56.44	50.53	43.84	59.83	52.4
Al_2O_3	15.52	13.42	13.32	14.34	10.85	13.4	11.52	11.13	16.14	12.43	12.57	16.1	13.4
Fe_2O_3	3.97	4.67	3.89	14.52	3.21	4.39	3.6	3.14	---	4.69	3.33	4.90	4.0
CaO	4.98	3.66	3.38	7.29	11.22	4.20	7.03	8.73	5.33	9.33	13.86	4.62	6.9
MgO	4.66	3.61	3.9	3.2	8.6	3.88	4.45	4.6	3.22	4.89	3.52	3.10	5.2
Na_2O	0.56	0.39	0.41	0.63	0.49	0.64	0.19	0.31	0.72	0.81	0.40	0.65	0.62
K_2O	0.70	0.64	0.41	0.41	0.19	0.41	0.48	0.51	0.58	0.39	0.46	0.54	0.48
P_2O_5	2	1.6	1.4	2	2	1.8	1.7	2.2	0.65	2.04	2.04	0.54	1.7
TiO_2	0.52	0.64	0.54	0.54	0.39	0.60	0.50	0.49	X	0.52	0.48	x	0.52
CIA*	85	87	88	91	85	84	84	87	84	81	87	85	86
PIA*	94	97	97	96	96	93	98	97	92	88	94	92	95
CPA*	93	99	97	94	93	93	x	99	95	91	97	96	95
A(Al_2O_3)	85	87	88	86	85	92	85	87	83	80	87	84	86
CN(CaO+Na_2O)	11	8	9	11	12	5	13	9	13	18	9	12	11
K (K_2O)	4	5	3	3	3	3	2	4	4	2	4	4	3
Al_2O_3/TiO_2	30	21	23	27	28	22	23	23	X	31	26	x	25

* CIA (Chemical index of alteration) expressed as the molar volumes of (Al_2O_3/ Al_2O_3 + CaO + Na_2O + K_2O) X100 (Nesbitt and Young, 1982); PIA (Chemical proxy of alteration = 100 × Al_2O_3 {Al_2O_3 + Na_2O} (Buggle *et al.*, 2011); PIA = {(Al_2O_3 - K_2O)/(Al_2O_3 - K_2O + CaO* + Na_2O)} × 100 (Fedo *et al.*, 1995). CaO* represented the CaO present only in the silicate minerals (Nesbitt and Young, 1982, 1984 and 1989). Since the studied shale samples contain considerable proportions of calcite, the CaO* content is assumed equivalent to Na_2O content according to McLennan (1993). For more accuracy only halite-free samples are selected.

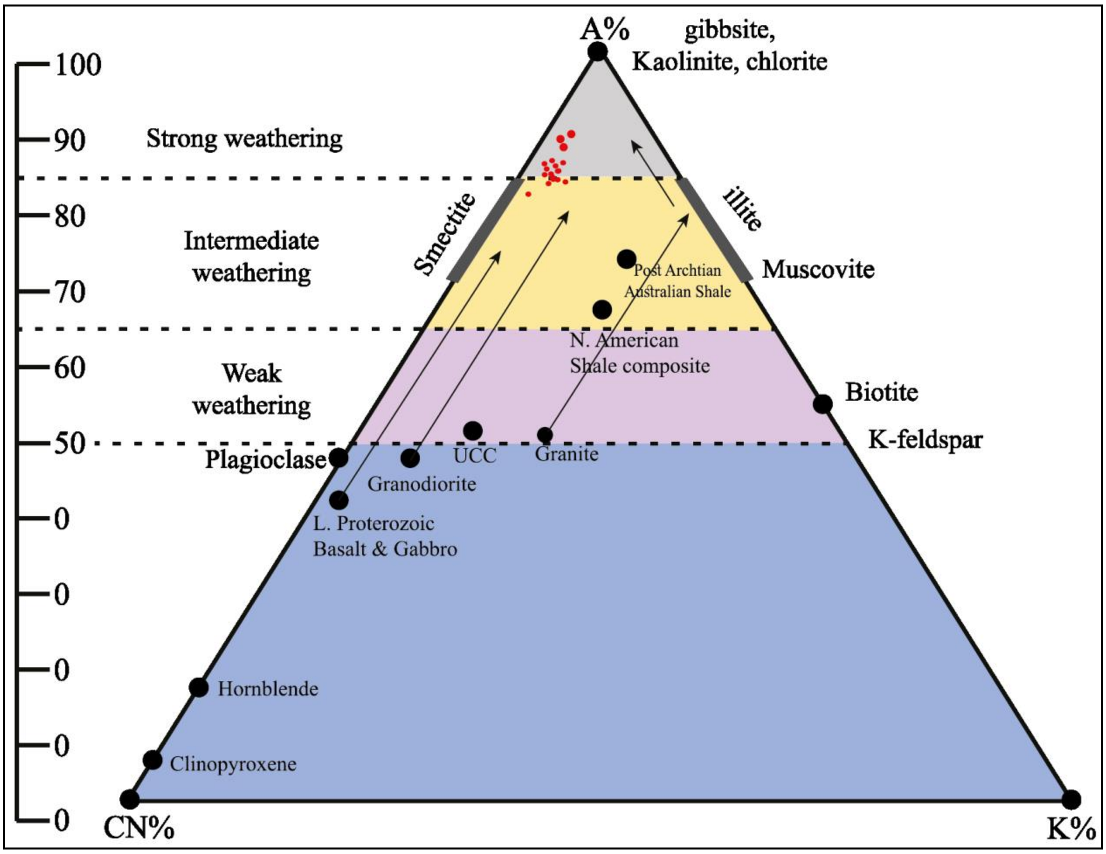

Fig.9: Ternary diagram of molecular proportions A-CK-K (Nesbitt and Young, 1984) for the studied shale samples. Average granite, granodiorite and upper continental crust (Taylor and Mclennan, 1985; Nesbitt and Young, 1984 and Wedepohl, 1995). Post Archaean Australian Shale from (Taylar and McLennan, 1985), North American Shale Composite (Grommet *et al*., 1984), Global mean of basalt composition and gabbro (Condie 1993) and idealized mineral compositions (McLennan, 1993) CaO represent the CaO associated with the silicate fractions. Arrows indicate the weathering trend

The above results suggest that the studied shales have high and uniform weathering index values, which indicate intense chemical weathering condition at the source rock in the distal Arabian Shield. The alteration was enhanced by river(s) transportation, which led to the disappearance of the plagioclase, K-feldspar, muscovite, biotite, other ferromagnesian minerals and mostly washout the Ca, Na and K, coupled with the concentration of Al, Fe, Ti, Si and Mg. The degree of the weathering apparently did reach the extreme state of transfering the smectite into kaolinite, but these conditions led to the formation of smectite – kaolinite paragenesis. The Si, Al, Fe, Mg and Ti are further concentrated in the soil product, with no or trace amount of illite/ smectite mixed layers (Nesbitt and Young, 1982). This scenario is confirmed by the XRD results and the badly crystalline kaolinite indicated by IR analysis. These proxies, especially the CIA and A-CN-K diagram, also help to predict the composition of the source rock and the paleoclimate conditions (McLennan, 1993). The Ideal Weathering Trend (IWT) and A-CN-K plot close to the A-CN axis which may indicate intense weathering of the plagioclase and other feldspars, and suggest that the weathered rocks were a mixture of basalt/gabbro and granodiorite/granite igneous rocks, weathered under warm and humid climate.

The binary diagram of SiO_2 vs. total Al_2O_3 + K_2O + Na_2O (Fig.10) (after Sutter and Dutta, 1986) was applied to estimate the maturity of the studied smectitic-rich shale which shows the paleoclimate of the source rocks. The studied shale samples plot in the field of semi-humid climate, which probably suggest that the paleoclimate of the Arabian Shield in the latest Maastrichtian – Danian interval was warm and semihumid. The high proportion of rounded to subrounded quartz, the abundance of well-sorted mature sandstone rich in mono-crystalline quartz grains, the absence of detrital feldspar as well as the dominant ZTR-Staurolite among the heavy minerals in the studied siliciclasts indicate distal source rocks. On the other hand, when the smectites were deposited, there are important signs of climate becoming drier and may be changed from semihumid in the older times of the Cretaceous and Jurassic to semiarid and warmer in the Late Cretaceous and early Paleogene, indicated by the smectite transformation to palygorskie (Singer, 1981; Chamly, 1989), gypsum-anhydrite, halite and probably barite precipitation and the chert nodules and geodes which were originated by silica replacement of the original anhydrite nodules (Petrank *et al.*, 1983 and Mohammed, 1993).

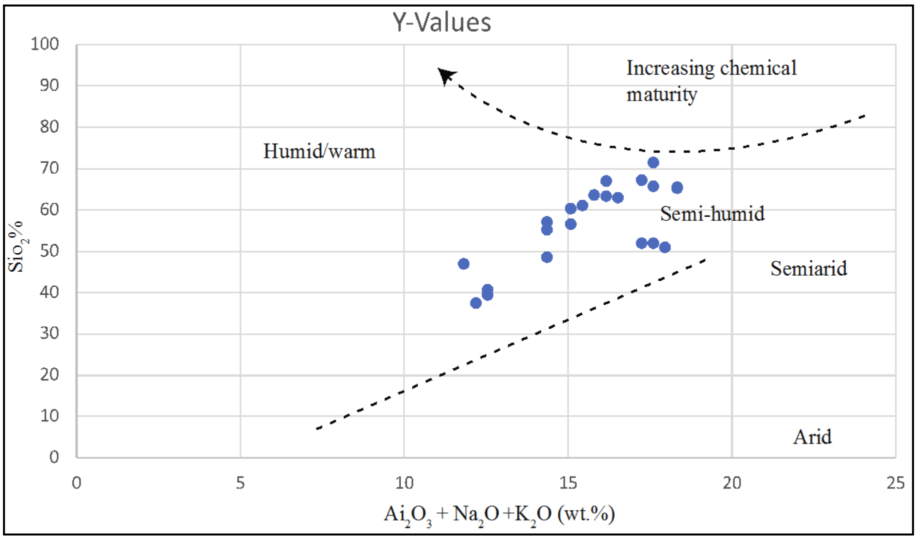

Fig.10: Bivariant plot for SiO_2 vs (Al2O3 + Na2O + K2O)
(after Sutter and Dutta, 1986)

- **Provenance and Tectonic Setting**

Immobile major and trace elements present in the siliciclastic sediments carry important messages about the source rocks from which these sediments were formed, because they remain relatively constant and unchanged during chemical weathering, long fluvial transport, early and late digenesis and metamorphism, like Al_2O_3, Fe_2O_3, TiO_2, Zr, Y, Nb and other trace elements, and their ratios are good parameters to predict the composition of the parent rocks (Floyed and Winchester, 1978; Bhatia and Crook, 1986; Condie, 1993; Cullers, 1995; Sugitani *et al.*, 1996; Hayashi *et al.*, 1997; Nesbitt and Young, 2004; Zhou *et al.*, 2015; Moradi *et al.*, 2016; Fathy *et al.*, 2017; Hassan, 2017; Fathy *et al.*, 2018).

According to the binary diagrams of TiO_2 vs. Al_2O_3 and TiO_2 Vs. Zr (Hayashi *et al.*, 1997), the studied samples of both Safra and Traifawi shales fall mainly in the field of Granite + Basalt (Fig.11A) and most samples fall within the Intermediate field and a few within the felsic igneous rocks (Fig.11B). In the Y-Ti and Nb-Ti diagram used by Hassan (2017), the studied shale samples fall in the area restricted between granite – granodiorite and Wadi

Natash Basalt (Figs.11C, D), which reveal an intermediate or mix felsic and mafic hinterland. Floyed and Winchester (1978) used the ratio of Zr/TiO_2 vs. Nb/Y diagram for the source of signatures of volcanic or equivalent plutonic rocks. The majority of the studied shale samples fall in the Trachyandesite – Andesite and Alkali-Basalt (Fig.11E), which indicate intermediate igneous and metamorphic source rocks.

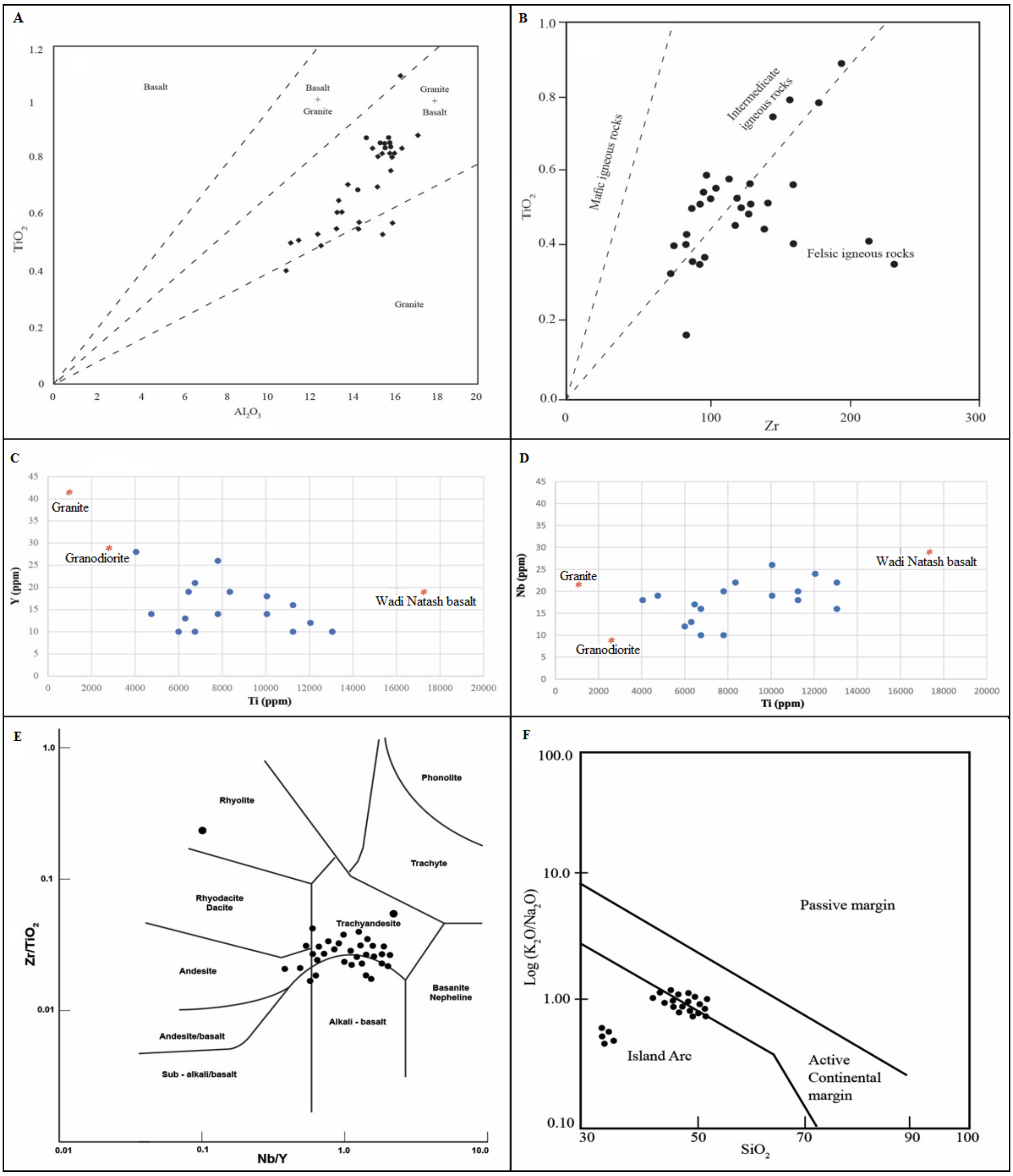

Fig.11: Provenance discrimination diagrams for the studied shales: **A)** TiO_2 vs. Al_2O_3;. **B)** TiO_2 vs. Zr (after Hayashi *et al.*, 1997); **C)** and **D)** Y vs. Ti and Nb vs. Ti (after Hassan, 2017); **E)** Zr/TiO_2 vs. Nb/Y (after Winchester, 1978) and **F)** Log (K_2O+Na_2O) vs SiO_2 (after Roser and Korsch, 1988). Average granite and granodiorite (after Condie, 1993) and Wadi Natash Basalt (after Hubbard *et al.*, 1987)

The major and trace elements proxies reveals that the smecte-rich shale of the studied Maastrichtian and Danian units was originated from an intermediate source rock provenance with a mixture of mafic, felsic and metamorphic source rocks of the Arabian Shield. For tectonic setting the bivariate discrimination function diagram of SiO_2 vs. K_2O/Na_2O ratio (Roser and Korsch, 1988) was applied and suggest that the studied shale samples fall between the active continental margin and oceanic margin (Fig.11F).

CLAY GENESIS

Field and laboratory investigations conclude that the studied smectite-rich Maastrichtian – Danian succession display two clay mineral associations: smectite- kaolinite characterizes the lower parts of the Safra and Traifawi transgressive sequences and smectite-palygorskite/ sepiolite which characterize the Upper parts of the Safra and Traifawi regressive sequences locally rich with porcelenite (opal-CT) lithofacies. The smectite-kaolinite association was derived from the continental detrital source formed by diagenetic transformation of the precursor siliciclastic sediments and submarine weathering or alteration of volcanic material (Millot, 1970). Detrital smectite and kaolinite probably crystallized by high intensity of chemical weathering of intermediate composition crystalline rocks of the Arabian Shield, including metamorphic, mafic and felsic igneous rocks. The neoformation of the clay minerals (smectite and kaolinite) was likely to have been cooked in warm tropical-semihumid paleoclimate and badly drained low lateritic pedogenic soil profiles, associated with regional Late Campanian – Maastrichtian regional transgression of the Tethys Ocean.

The smectite and kaolinite clay minerals can be redistributed through transportation from the source area to the marine sedimentary basins (Biscay, 1965 and Gibbs, 1977), where the larger size kaolinite is deposied proximal near the shore and the finer size smectite deposited in the distal subtidal deeper environment with minor amount of kaolinite.These two clay minerals (smectite and kaolinite) were deposited, during the Maastrichtian – Danian, in a broad East – West closed seaway basin in an open-subtidal sedimentary environment associated with transgressive sequence and TST of the latest Maastrichtian – Danian depositional cycles. The smectite-bearing sequences were laid down in the lower parts of the Safra Beds and Traifawi Member, at the sediment-water interface under reducing conditions. The reducing conditions were developed by the accumulations of organic matter rich in phosphorus and silicon, both of which could have been the result of the huge bio-productivity generated in the southeastern Tethys Ocean (Parrish, 1987) and induced by the upwelling deep cold water to the coastal region of accumulations around the Cretaceous/Tertiary boundary.

Palygorskite and sepiolite are interpreted as of authigenic origin formed by the in-situ transformation of smectite to palygorskite and probably in advance stages to sepiolite, under a warm-semiarid climate (Singer, 1980 and 1984; Callen, 1985 and Chamley, 1989). These clay minerals and their paragenesis were generated when the paleoclimate was gradually developed from semihumid climate in the source area to the semiarid in the depositional basin. The main quantity of the palygorskite neoformation was recognized at the top of the regressive sequences and HST.

RESOURCES OF MONTMORILLONITE-RICH SMECTITES

The total reserves of the montmorillonitic smectites in the Iraqi Western Desert is about 323 million tons, most of them are located in the Safra Beds of the Digma Formation at Wadi Bashira, south of the Iraq – Jordan highway (Al-Bassam and Saeed, 1989; Al-Bassam *et al.*, 1989b and Al-Bassam *et al*., 1991) (Table 7). The smectite-rich shale approaches 10 m in thickness at this locality. The Danian shale is distributed in several beds of about 1 m thick each and separated by chert horizons at the Traifawi area (Khudair *et al*., 1988; Al-Bassam *et al*., 1989b).

Table 7: Reserves of the smectites-rich shale in the Iraqi Western Desert

Deposit	Formation	Reserve C2 (m ton)	Reserve C1 (m ton)	Reserve B (m ton)	Reserve A (ton)	Reference and report number (GEOSURV)
Wadi Bashira	Safra Beds-Digma Fn.	71.65	201.11	27.67		Al-Bassam and Saeed, 1989 (rep. no. 1922)
Wadi Bashira	Safra Beds-Digma Fn.				1.05*	Al-Bassam *et al.*, 1991 (rep. no. 1981)
Traifawi Area – I	Traifawi Member, Akashat Fn.		5.85	4.27		Khudair *et al.*, 1988 (rep. no. 1573)
Traifawi Area – II	Traifawi Member, Akashat Fn.		11.55	1.32		Khudair *et al.*, 1988 (rep. no. 1573)

(*) About 100 000 tons of which have been mined

MINERAL PROCESSING AND INDUSTRIAL APPLICATIONS

▪ Upgrading

The cation exchange capacity (CEC) of clay minerals is an essential factor in the industrial applications. It can be used to determine the quality (grade) of the bentonite or montomorillonite-rich smectite and to estimate their contents in the claystones (Al-Bassam *et al*., 2011). The calculated CEC of the high grade smectite-rich shale is medium, in average 79 mEq/100g (range between (70 – 85 mEq/100g), which is not far from the value for natural pure montmorillonite of (100 mEq/100g) (Srodon *et al*., 1992). However, most of the industrial smectite reserves of the Wadi Bashira deposit in the Iraqi Western Desert is of low grade due to calcite impurities (>5% CaO) and this presented a serious problem in the Na-activation and industrial applications. Several successful attempts have been made in the Mineral Processing Department of the Iraq Geological Survey to increase the efficiency of the raw material through upgrading the smectite by carrier flotation to remove calcite dilution effect. These experiments succeeded in decreasing the CaO content in the low grade Ca-rich smectites from 14 % CaO to 2.5 % CaO, and increasing the CEC from 60 to 89 mEq/100 g (Al-Ajeel *et al*., 1990 and Al-Ajeel *et al*., 2014). Other experiments have been made to upgrade these smectites by alternative beneficiation techniques, such as dispersion sedimentation (Al-Ajeel *et al*., 2008), acid leaching (Abdulla and Al-Ajeel, 2010). Acid leaching of the calcite impurities by 10 % acid concentration increased the Al_2O_3 content from 14.5% to 18.68 % (Mohammed, 1988 and 1993).

▪ Activation

The Tertiary bentonites of Qara Tappa and Zarloukh are of the Ca-type and the attempts made to activate these montomorillonites to Na-type were unsuccessful. In view of the absence of carbonates and presence of quartz as the main diluent in these bentonites, they were activated by acid and proved efficient after activation, as filter aids. On the other hand, most of the Maastrichtian – Danian smectites contain calcite in appreciable quantities (>5% CaO) and they are composed of Ca-montmorillonite with low cation exchange capacity (CEC). The high carbonate content represented a serious problem in the activation process.

Sodium activation of the high grade montmorillonitic smectite claystone (<5% CaO) was achieved in laboratory experiments by the dry method using Na_2CO_3. The rheological results of the Na-activated clays were compatible to the specifications used by the American Petroleum Institute (API) for drilling fluids (Abdullah and Al-Ajeel, 2010; Mahdi *et al.*, 2013).

- **Industrial Applications**

The bentonites of Qara Tappa and Zarloukh deposits have been used for decades, after acid activation, in the purification of native sulfur. A chemical plant was erected in the Mishraq Sulfur Company, south of Mosul City, for the acid activation of these bentonites and they proved to be efficient filter-aid in the sulfur mining industry.

Following the successful attemptes in the upgrading and Na-activation of the smectite (montmorillonite)-rich deposits of Wadi Bashira, several tons of the high-grade raw materials were excavated (Al-Bassam and Majid, 1990) and transported to the oil fields of southern Iraq. It was used, after crushing and grinding, as a drilling mud in 1991 in the drilling of an oil well in the Zubair Oil Field (Al-Uqaily *et al.*, 1991). The total depth of the well reached 3370 m and the drilling mud specifications conformed to the API specifications throughout the drilling operations which lasted about 7 months. The successful attempt to use these clays in the oil industry encouraged the Iraq Geological Survey to sign a contract with a Chinese company to erect a plant in Falluja (Anbar Governorate) for the production of Na-activated “bentonite” at a capacity of one million tons/year. The product has been used eversince in drilling of oil and water wells.

Attempts have been made to use natural smectite-rich shale, produced from Wadi Bashira (Safra) mine, to improve ion exchangeability of potassium in sandy and gypsiferous soils (Al-Bassam *et al.*, 2009) and as a filter aid in some food industries such as vegetable oil and as absorbent for the removal of oxytetracycline from water (Barbooti *et al*., 2012). It was also tested in other applications such as cosmatics and removal of uranium from polluted soils. The adsorption efficiency for Pb (II) from aqueous solutions was investigated using the smectite from the Traifawi deposit by Awadh and Abdulla (2017). They have reached the thermodynamic result of high capacity for the adsorption of lead ions and they recommended using the smectite as a natural adsorbent of Pb (II) from liquid wastes.

CONCLUSIONS

- Two genetic types of montmorillonite-rich claystones are found in Iraq: **(1)** the bentonite “*senso stricto*” in the Upper Miocene- Pliocene Mukdadiya Formation exposed in the Himreen Mountain Range of the Low Folded Zone, and **(2)** the smectite-rich black shales associated with phosphorites, in the Maastrichtian Digma Formation (Safra Beds) and the Danian Traifawi Member of the Akashat Formation (Paleocene) in the western part of the Iraqi Western Desert, located within the Inner (stable) Platform of the Arabian Shelf.
- The Neogene bentonites formed by the alteration of volcanic ash in fluvial (lakes) system, whereas the Upper Cretaceous and Paleocene smectites formed by the pedogenic alteration of highly weathered igneous, metamorphic and older sedimentary rocks of the Arabian Shield, fluvially transported to the basin and deposited in anoxic marine upwelling regime within a ramp system and a cyclic rythem.
- The total reserve of the Neogene bentonites is about 1.8 m.t. which was mostly consumed, after acid-activation, as a filter-aid in the beneficiation of native sulfur in the Mishraq sulfur mines. The reserves of the montmorillonite-rich smectite shales of the Maastrichtian

and Danian deposits are about 300 m.t in the former and about 23 m.t in the later, and they are being used, after Na-activation, in the preparation of drilling fluids for oil and water wells.

- The enormous reserves of the montmorillonitic smectites in the Maastrichtian Safra Beds (Digma Formation) and the successful industrial production of Na-activated "bentonite" in API standard specifications provide excellent opportunity for Iraq to satisfy the local drilling operations of the oil industry and to have a competitive commodity for export.

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