






Mineralogical Components of the Upper Jurassic Naokelekan Formation from Northeastern Iraq

Sumaya Asghar Samad^{1,2*} , Hasan A. Ali Al-Jumaily² , Ali Ismail Al-Juboury³ , Harry Rowe⁴ , Giovanni Zanoni⁵ , Rebwar H. Rasool⁶ , Arkan O. Sharazwri⁷ 

¹Northern Oil Company, Kirkuk, Iraq.

^{2,6}Department of Applied Geology, College of Science, University of Kirkuk, Iraq.

³Petroleum Engineering Department, College of Engineering, University of Al-Kitab, Kirkuk, Iraq.

⁴Premier Corex Laboratories, Houston, TX 77041, USA.

⁵Rohm Tek, Houston, TX 77024, USA.

⁷Department of Petroleum Geosciences, Faculty of Sciences, Soran University, Soran- Erbil, Iraq.

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Correspondence:

Name: Sumaya Asghar Samad

Email:

scgm23003@uokirkuk.edu.iq

ABSTRACT

The upper Jurassic Naokelekan Formation has been investigated using X-ray diffraction and scanning electron microscopy in two outcrop sections from Halabja and Barzinja areas in northeastern Iraq to determine mineralogical composition. The study shows that the abundance of calcite, rare dolomite, quartz, feldspars, and some other minerals, such as pyrite, goethite, natrolite, and apatite, form the main non-clay composition. Illite (mica) and mixed layers of illite/smectite are the clay minerals. Calcite is the dominant mineral in all the studied samples, while the presence of the framework silicates of quartz and feldspars (plagioclase and K-feldspar) shows a variation across the studied sections, where they increase in the argillaceous limestone and calcareous shale samples. Various forms of calcite reflect contributions from depositional environment, diagenesis, inorganic mineralization, and biogenic contribution, while detrital contribution due to weathering is responsible for the quartz and some feldspar content. Hot arid paleoclimatic conditions are indicated by the common presence of illite (mica).

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المكونات المعدنية لتكوين ناوكليكان (الجوراسي المتأخر) من شمال شرقي العراق

سمية اصغر صمد^{1,2*} ID، حسن احمد علي الجميلي² ID، علي اسماعيل الجبوري³ ID، هاري روي⁴ ID، جيوفاني زانوني⁵ ID، ريبوار رسول⁶ ID، ارکان شارازويري⁷ ID

¹ شركة نفط الشمال-كركوك-العراق.

^{2,6} قسم الجيولوجيا التطبيقية-كلية العلوم-جامعة كركوك.

³ قسم هندسة النفط-كلية الهندسة-جامعة الكتاب-كركوك-العراق.

⁴ مختبرات بريمر كوريس-هيوستن-الولايات المتحدة الاميركية.

⁵ شركة روم تيك-هيوستن-الولايات المتحدة الاميركية.

⁷ قسم جيولوجيا النفط-جامعة سوران-اريل-العراق.

الملخص

تمت دراسة تكوين ناوكليكان (الجوراسي الاعلى) باستخدام حيود الاشعة السينية والمجهر الماسح الالكتروني في مقطعين سطحين هما حلجة وبرنجة شمال شرقي العراق لتبيان المكونات المعدنية. أظهرت الدراسة بأن وفرة الكالساييت وندرة من الدولومايت، الكوارتز والفلسبار مع معادن اخرى مثل البايرايت والجوثايت والناترولوايت والاباتايت تشكل المعادن غير الطينية. الالاييت (المايكا) و الصفائح المختلطة من الالاييت-سمكتايت تشكل المعادن الطينية. يعد الكالساييت المعدن الاكثر شيوعا في جميع نماذج الدراسة في حين ان السليكات الهيكلية من الكوارتز والفلسبار (البلاجيوكليس والفلسبار البوتاسي) تظهر تغيرا عبر المقطعين قيد الدراسة اذ تزداد في الصخور الجيرية الطينية. الاشكال المختلفة للكالسايت تعكس اضافات وتأثير البيئة الترسيبية والعمليات التحويرية والتمعدن غير العضوي او التأثير العضوي. بينما يعود وجود الكوارتز والفلسبار الى التأثير الفتاتي نتيجة عمليات التجوية. كما عكس شيوع الالاييت تأثير الظروف المناخية القديمه الحارة والجافة.

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المراسلة:

الاسم: سمية اصغر صمد

Email: scgm23003@uokirkuk.edu.iq

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Introduction

The late Jurassic Naokelekan Formation is composed of black, bituminous limestones that are thin to medium-bedded and dolomitic limestone intercalated with thin, black, and calcareous shale in both the upper and lower portions of the formation (Bellen et al., 1959).

In most parts of northern Iraq, the tripartite separation of the formation is identifiable based on field measurements and petrographic study. This sequence from bottom to top is composed of (1) dolomitic limestone, (2) kerogenous limestone, and (3) laminated shaly kerogenous limestone. Mature organic matter can be found in the Naokelekan Formation source rock, which is part of the Zagros Mountain belt and is produced during the oil generation window (Abdula, 2023). Shallow marine subtidal to supratidal and euxinic (anoxic) depositional environments have been confirmed by the presence of algal mats rich in organic matter (Buday, 1980; Abdula, 2016). Additionally, the shallow marine, brackish lagoons and

subtidal (lagoon) have also been considered by several authors, such as Salae (2001), Balaky (2014), Omar et al. (2023).

Little is known about the mineralogy of various lithologies of the formation, distribution, and origin. Therefore, the mineralogical composition of the lithological units of the formation using X-ray diffraction (XRD) supported by scanning electron microscopy (SEM) has been conducted on two outcrop sections (Barzinja and Halabja) from northeastern Iraq to determine the main mineralogical phases and conclude variation in their content along the studied sections.

Study Area

The two studied sections lie in Halabja and Barzinja cities, respectively. The Halabja section is in the Shnrwe anticline at the coordinates (35° 07' 36.53" N- 46 ° 03' 57.10" E), while the Barzinja section lies in the Galal valley at coordinates (35° 32' 56.00" N- 45° 44' 56.87"E), as shown in Figure 1. Argillaceous limestone and thin shale beds, with common fractures and gastropods, form the main lithology in the Barzinja section (Fig. 2). In the Halabja section, the lithology is composed of limestone, argillaceous limestone, calcareous, organic-rich shale, and black chert. The limestones are thin to medium-bedded, fractured, and stylolitic (Fig.3). Tectonically, the Barzinja sections lie in the High Folded Zone, while the Halabja sections in the Imbricated Zone according to the tectonic divisions of Iraq (Fouad, 2015) (Fig.1).

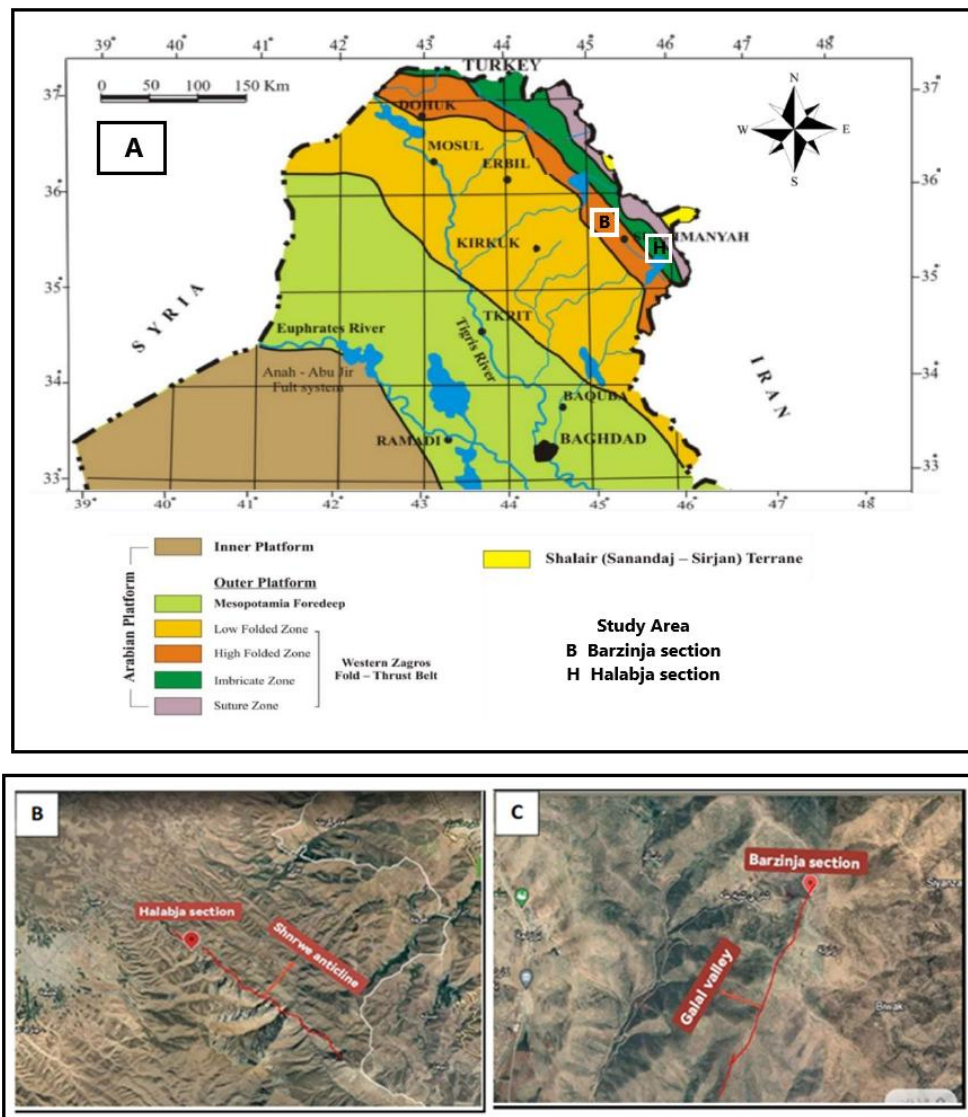


Fig. 1. (A) Tectonic division map of Iraq from Fouad (2015), showing the location of the studied sections, (B) and (C), Aerial photos of the Halabja and Barzinja sections, respectively

Geological History

The Mesozoic sequence in Iraq was deposited along the Arabian craton's eastern passive boundary (Numan, 1997). This boundary underwent suture in the Late Cretaceous and Early Tertiary, after evolving from a rift situation in the Permian and Triassic to drift in the Jurassic and Early Cretaceous (Burchette, 1993). The exposed Jurassic formations in the Zagros/Foreland basin of Iraq crop out in isolated locations within the eroded cores and limbs of anticlines in the structural zones of the Zagros Basin, i.e., High Folded, Imbricated, and Thrust Zones of Northern Iraq (Numan, 2000).

A foreland basin that accommodated the deposition of the Naokelekan and Barsarin formations was formed near the craton's edge during the Late Jurassic (Murriss, 1980; Numan, 2000; Kameran et al., 2023).

There are significant differences in the thickness of the Naokelekan Formation throughout the area of northern Iraq (Bellen et al., 1959). At every outcrop location, the Barsarin Formation overlies the Naokelekan Formation, while the Sargelu Formation underlines it. The lithological composition and the composition of all known occurrences of the formation are similar (Buday, 1980).

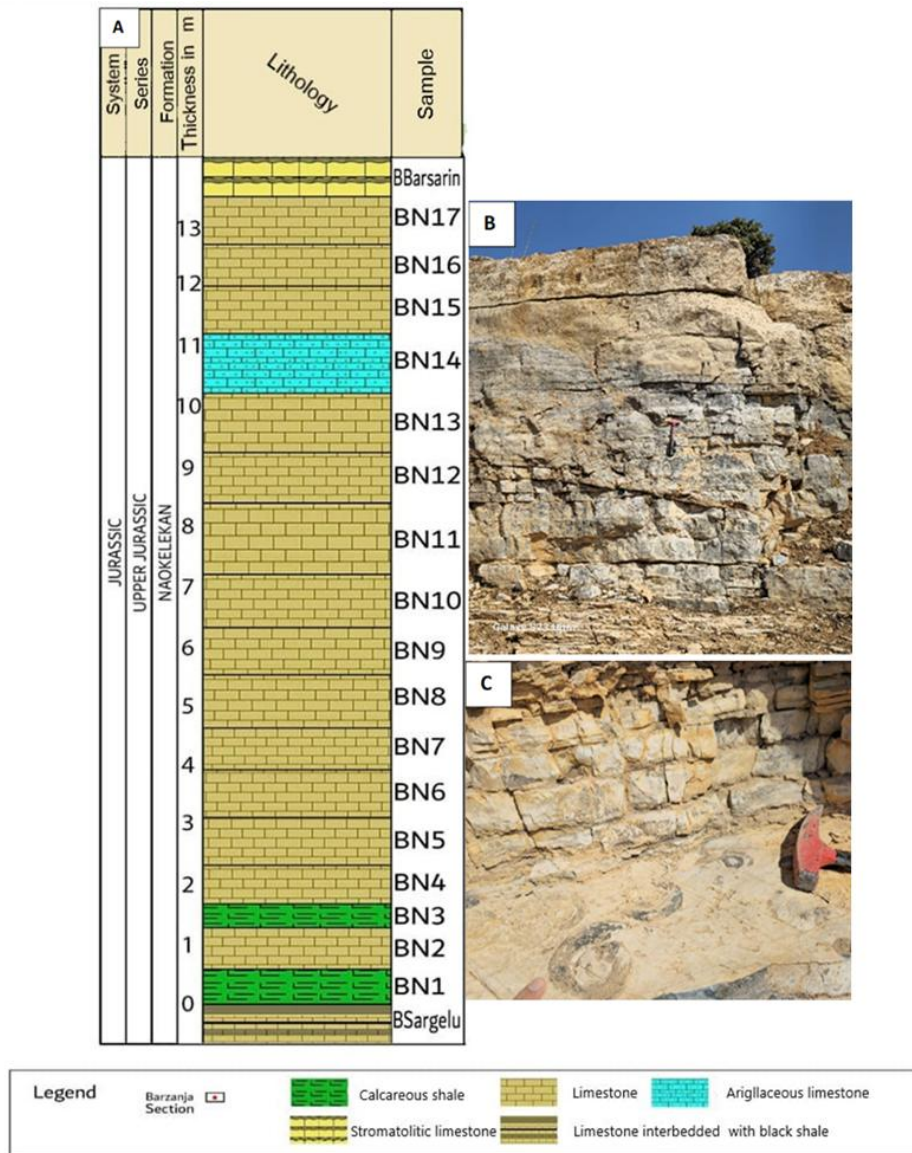


Fig. 2. (A) Lithological section of the Naokelekan Formation at Barzinja section with samples location showing the common limestone with shale intercalations; (B) Thin to medium bedded fractured limestone ; (C) Gastropods, commonly observed in large sizes in limestone units.

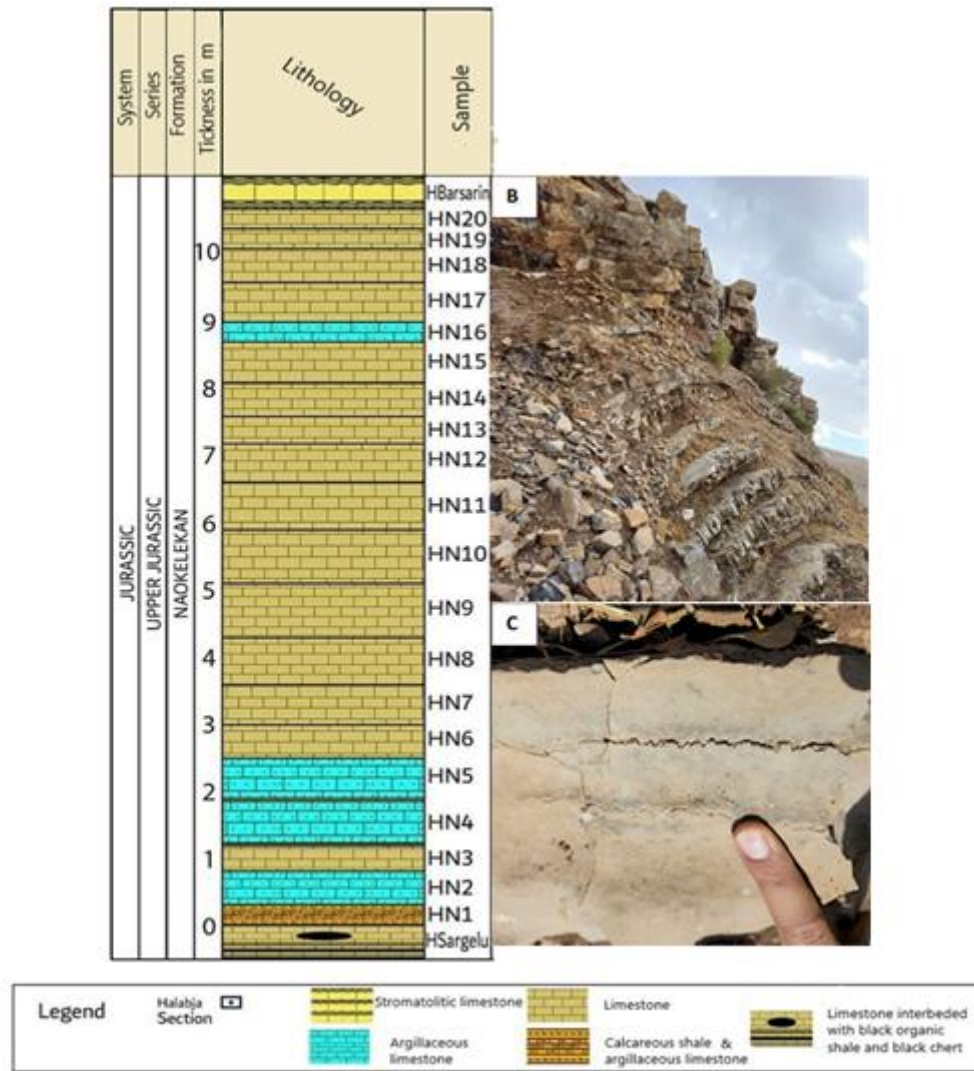


Fig. 3. (A) Lithological section of the Naokelekan Formation at Halabja section with sample locations showing the common limestone, some are argillaceous alternated with calcareous shale; (B) Thinly bedded limestone alternated with shale that characterizes the studied succession; (C) Common stylolite in the limestone units.

Materials and Methods

Samples of this study were selected from the limestone, argillaceous limestone, and calcareous shale samples of the two studied sections (Barzinja and Halabja) of the Naokelekan Formation (Figures 2 and 3) and then prepared for the mineralogical analyses.

At Premier Corex Laboratories in Houston, Texas, X-Ray diffraction (XRD) examination is carried out on a selection of bulk samples. A Bruker D8 Advance XRD equipment, fitted with a Lynxeye XE-T detector and a theta-theta goniometer with a 250 cm radius, is used for this purpose. CuK radiation is used for all measurements, and 40 kV and 30 mA of voltage and current, respectively, were applied. The TOPAS software suite is used to quantify the mineral phases in the bulk diffraction pattern. Scanning electron microscopy (SEM) analyses are performed on a limited number of samples at the same laboratories using FEI Quanta FEG 650 FE-SEM instruments. These instruments are furnished with an FEI R580 Everhart-Thornley (ETD) electron detector and two Bruker EDS XFlash 5030 energy dispersive X-ray spectroscopy (EDS) detectors.

Results and Discussion

X-Ray diffraction analysis (Table 1) has revealed that the main mineralogical components according to their groups, are as follows: framework silicates (quartz and feldspars), carbonate

(calcite and dolomite), others (pyrite, goethite, natrolite, and apatite), in addition to clay minerals including illite (mica) and mixed layers illite/smectite.

The x-ray diffractograms (Figures.4 and 5) illustrate the mineralogical constituents of the studied Naoklekan samples in the two studied sections.

The presence of the framework silicates quartz and feldspars (plagioclase and K-feldspar) varies across the studied sections, where they increase in the argillaceous limestone and calcareous shale samples. Quartz ranges from 2.4 – 4 with an average of 2.94 in the studied samples of the Naoklekan Formation at Halabja section, whereas it increases in Barzinja section and ranges from 2 – 7.5 with an average of 4 (Table 1). Plagioclase feldspar appears only in the limestone sample, with a very small amount not exceeding 0.4, while K-feldspars show a larger amount than plagioclase and range from 0.3 to 4.0 in the Halabja section, with an average of 1.43, while in the Barzinja section, they appear in one sample of calcareous shale with 5.1%.

The carbonates dominate the mineralogical constituents of the studied samples since most of the lithological components are limestone. Calcite is the common mineral with a range from 74.4 – 96.0 (average 89.38%) and 71.6-94.8 (average 87.75%) in the Halabja and Barzinja sections, respectively. While dolomite and Fe-dolomite appear in argillaceous limestone samples HN1 and HN4, from the Halabja section, which is confirmed by common dolomitization in these samples (Fig. 5 and Table 1).

The other components include pyrite and apatite (0.4% and 2.2%) only in the calcareous shale sample from the Barzinja sample, respectively, goethite (averaged 0.4 and 0.1%) in the Halabja and Barzinja sections, respectively, and natrolite (averaged 0.1 and 0.9%) in the Halabja and Barzinja sections, respectively (Table 1).

The clay minerals include two minerals: the illite (mica) with a range of 0.3 to 4.3, average 2.18%, and a range of 1.8 to 8.4, average 4.7% in the Halabja and Barzinja sections, respectively. The mixed layers illite/smectite appear in one sample only, with an amount of 2% (Table 1).

In the SEM images, various types of calcites are recognized in the current study; these include: blocky calcite that fills coccolith nannofossil shells (Fig. 6A), scattered calcite grains (Figs.6A, B, C and 7C), star-shaped Mg-calcite microcrystals (Figs. 6B, 7A, B and 8A), columnar and euhedral calcite (Figs. 6B and 7B), disc-shaped calcite (Fig.7A), and bladed and platy calcite (Figs.8A and 9A). Calcite also observed in microspar and blocky cement (Fig.9B) and in radial and fibrous calcite forms (Figs. 7C and 9C).

These different shapes of calcite reflect the depositional conditions where the rocks of the Naoklekan Formation were deposited, which is, in general, shallow marine to lagoonal, as also mentioned in many previous works such as (Salae, 2001; Balaky, 2014; Omar et al., 2023). Formation of the star-shaped calcite crystals relates mostly to Mg²⁺ rich inorganic mineralization without organic contribution (Zhu et al., 2006), while the disc-shaped crystals were formed as a biogenic-specific morphology of carbonate minerals (Yang et al., 2023). Most of the other forms were formed diagenetically due to the effect of recrystallization or cementation processes (Dickson, 2022; Mahmood et al., 2025; Abdullah et al., 2025).

Table 1: Mineralogical constituents of the Naokelekan Formation in the studied sections.

Sample no./ Lithology	Non-clay Minerals										Clay Minerals		Total %
	Framework Silicates				Carbonates			Others			ML Illite/Smectite %	Illite (Mica) %	
	Quartz %	Plagioclase %	K-feldspar %	Calcite %	Dolomite %	Fe-Dolomite %	Pyrite %	Goethite %	Natrolite %	Apatite %			
Halabja section													
HN20 Limestone	2.6	0.2	Trace	94.8	0	0	0	0	0	0	Trace	2.4	100
HN16 Argillaceous Limestone	2.7	0	0.3	90	0.	0	0	0.7	0	0	2	4.3	100
HN11 Limestone	3	0	0.4	96	0	0	0	0	0	0	0	0.6	100
HN4 Argillaceous Limestone	2.4	0	1	91.7	4.3	0.3	0	0	0	0	0	0.3	100
HN1 Argillaceous Limestone	4.0	0	4.0	74.4	12.6	0	0	1.2	0.5	0	0	3.3	100
Range	2.4 – 4	0 – 0.2	0.3 – 4.0	74.4 – 96.0	0 – 12.6	0 – 0.3	0 – 0	0 – 1.2	0 – 0.5	0-0	0 – 2.0	0.3 – 4.3	
Average	2.94	0.04	1.43	89.38	3.38	0.06	0	0.38	0.1	0	1	2.18	
Barzinja section													
BN15 Limestone	2	0.2	0	90.3	0	0	0	0.8	0.3	0	0	6.4	100
BN12 Limestone	3	0.4	0	94.8	0	0	0	0	Trace	0	0	1.8	100
BN7 Limestone	3.5	Trace	0	94.3	0	0	0	0	Trace	0	0	2.2	100
BN3 Calcareous Shale	7.5	0	5.1	71.6	0	0	0.4	3.3	1.5	2.2	0	8.4	100
Range	2 – 7.5	0 – 0.4	0 – 5.1	71.6-94.8	0 – 0	0 – 0	0 – 0.4	0 – 3.3	0.3 – 1.5	0-2.2	0 – 0	1.8 – 8.4	
Average	4	0.2	1.28	87.75	0	0	0.1	0.1	0.9	0.55	0	4.7	

The framework silicates that are also recorded are quartz in rhombohedral form with secondary overgrowth (Fig. 6C) and feldspar (Fig. 7C), while illite is the main clay mineral that is also recorded in the SEM images in fibrous and platy forms (Figs. 6A, 8B, C)

Mineralogical composition of the late Jurassic Naokelekan Formation at Halabja and Barzinja sections reveals little variation in mineralogical components between the studied areas. In general, the calcite content belongs to the type of the studied rocks, which are commonly limestone, sometimes argillaceous, which in turn includes less calcite than in pure limestone (Table 1).

The silicate framework of quartz and K-feldspar content increases in the calcareous shale and argillaceous limestone (Table 1), reflecting their detrital origin as influx due to increased weathering. This conclusion was recorded by other authors. According to Tobia et al. (2019), Mena and Abdulla (2023), and Rasool et al. (2024), the main source rocks of the Sargelu and Naokelekan formations are felsic and felsic-intermediate igneous rocks supplied from the Mid Oceanic Ridge and deposited in an active setting. This may be related to a gradual increase in weathering from older sources, which has been confirmed in the middle to upper Jurassic formations from northern Iraq.

Sediments that have experienced diagenesis in an alkaline reducing environment often contain pyrite (Hanjo et al., 1965). This may confirm the deposition of the grey to black pyrite-bearing calcareous shale of the Naokelekan Formation in a reducing environment. Pyritization, which produces different crystal forms of pyrite, had taken place in reducing circumstances and is probably facilitated by the sulfate solution produced by reducing bacteria or by the

breakdown of organic molecules, which is then triggered by anaerobic bacteria (Hudson, 1982; Al-Auqadi *et al.*, 2024).

Natrolite is a common zeolite that can be found in altered basaltic rocks in veins and holes, but it can also exist in sedimentary rocks as a byproduct of diagenetic alteration. In some of the examined samples, the presence of natrolite conjugated with high amounts of dolomite and feldspars, particularly K-feldspar, this may suggest that the carbonate rocks were subjected to intense diagenetic processes (Deer *et al.*, 2004).

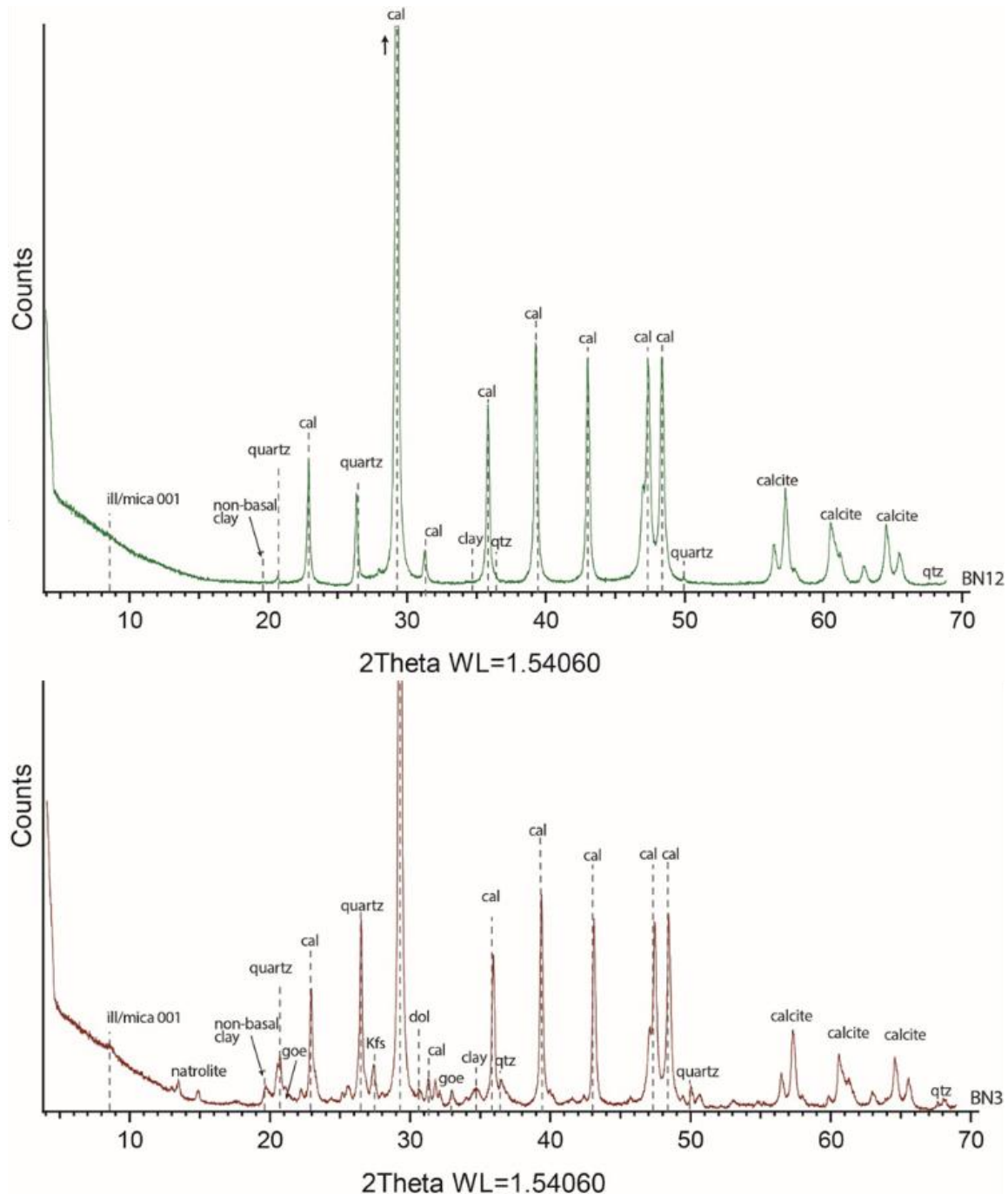


Fig. 4. X-ray diffractograms of selected B3 and B12 samples from Barzinja section illustrating the presence of illite (mica), quartz, calcite (Cal), natrolite, K-feldspar (Kfs), dolomite (dol) and goethite (goe) mineral phases.

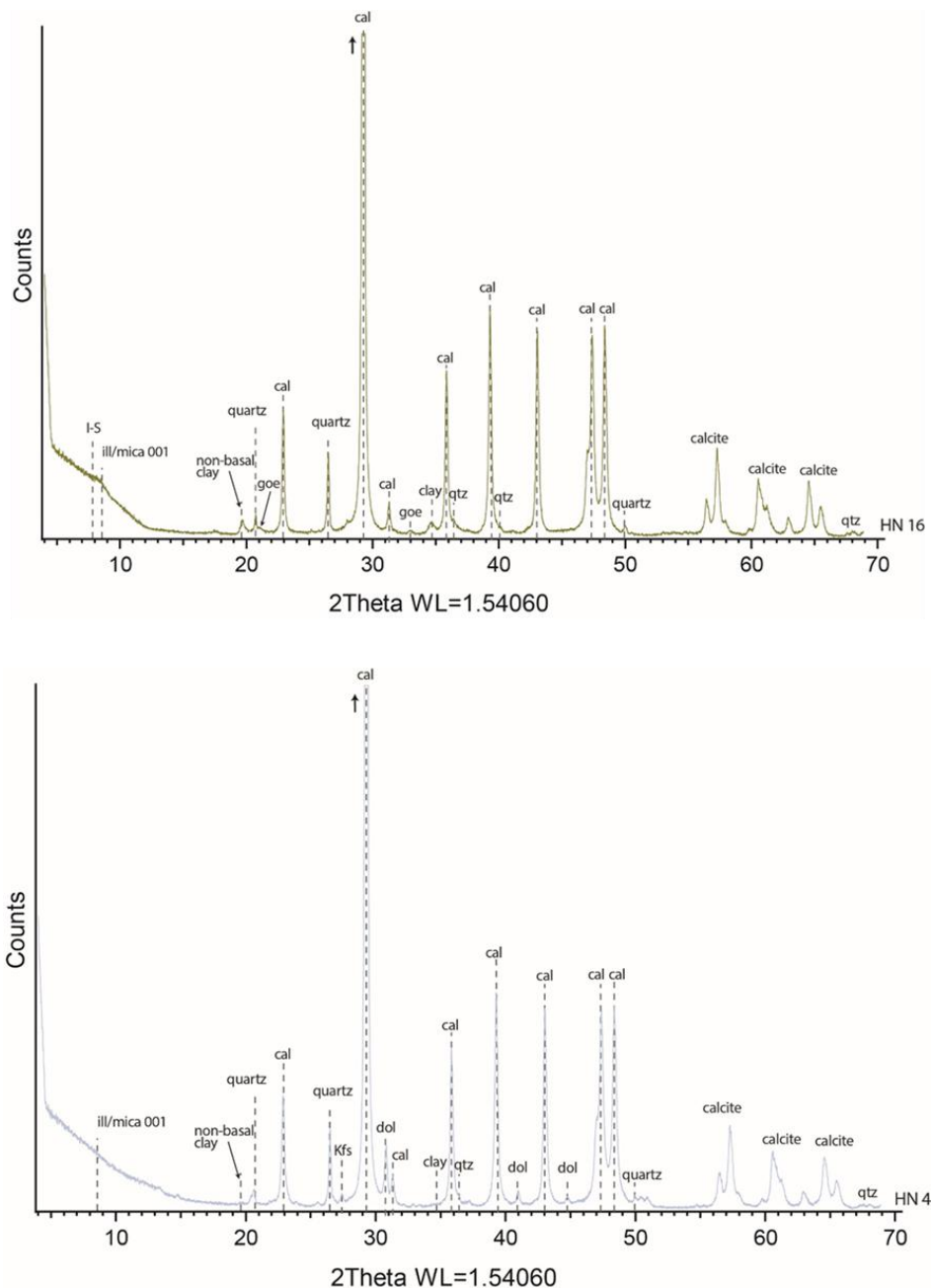


Fig. 5. X-ray diffractograms of selected HN4 and HN16 samples from Halabja section illustrating the presence of illite (mica), quartz, calcite (Cal), K-feldspar (Kfs), and dolomite (dol) mineral phases.

The sedimentary rocks that were formed in marine and lacustrine environments include some apatite, bones, teeth, scales, and feces, which were among the phosphatic organic waste that had accumulated and calcified there during diagenesis (Ptáček, 2016).

Clay mineral types provide information on the weathering conditions in the sedimentary basin and the dominant climate at the time of sedimentation (Chamley, 1989). Climate, tectonic events, pressure-temperature modulatory processes, and geochemical interactions are all linked to fractionation in clay minerals (Weaver, 1989).

Dominance of illite-mica reflects the dry and hot conditions prevailing during deposition of the Naokelekan Formation (Chamley, 1989). The dry condition in the Naokelekan Formation is promoted by relatively high amounts of dolomite in some of the studied samples. Dolomite is a common mineral that formed in hot and dry conditions (Warren, 2000; Al-Tae et al., 2024).

The limited and evaporative environmental deposition is reflected in the common calcite. In addition to weathering from older sedimentary rocks, the presence of both quartz and

feldspars with conjugated traces of natrolite indicates the contribution of felsic igneous rocks. This could be related to a provenance dominated by felsic rocks from the Arabian Shield and/or the Zagros ophiolites and related igneous rocks.

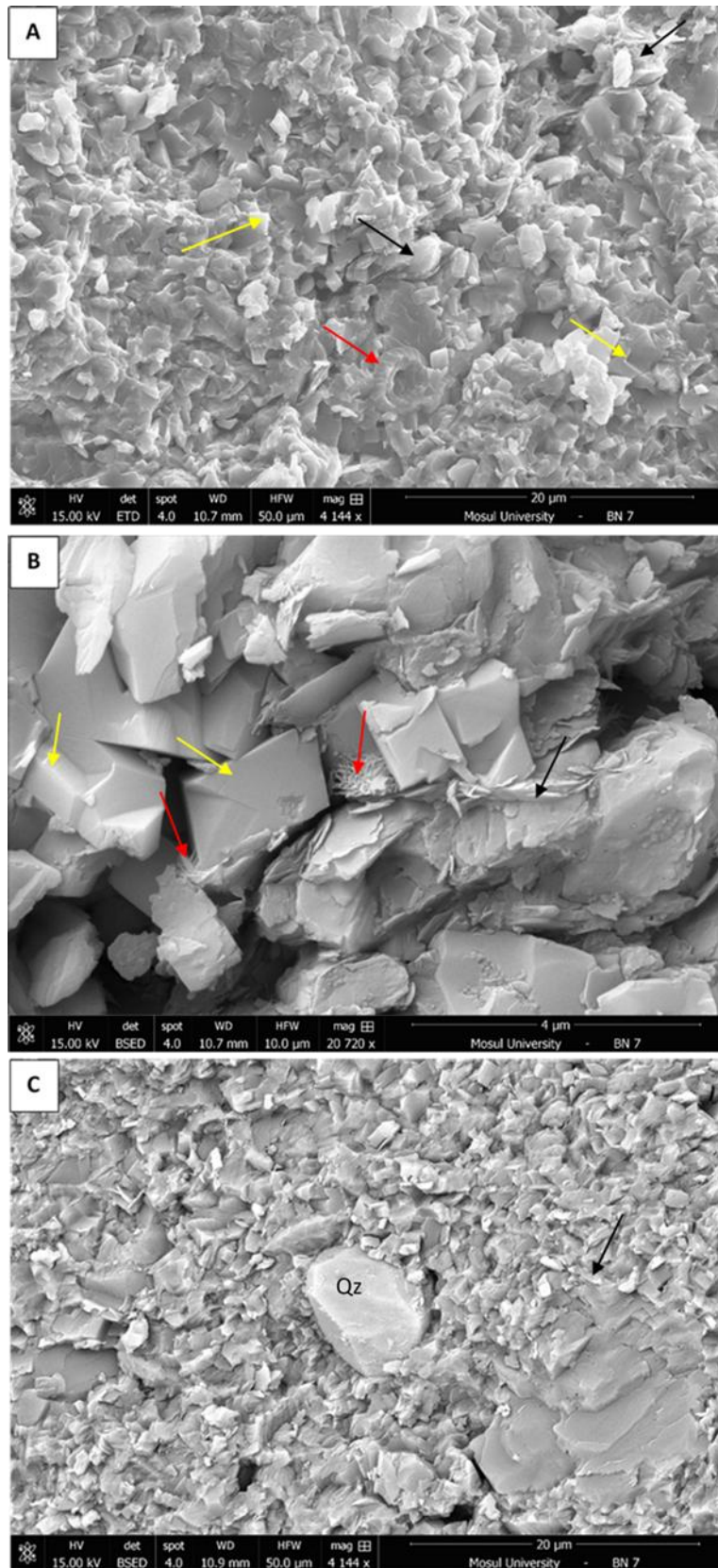


Fig. 6. Scanning electron microscopic images showing: A- calcite filling shell of coccolith (red arrow), scattered calcite grains (yellow arrows) and fibrous illite (black arrow). B- columnar and euhedral calcite (yellow arrows) and star-shaped calcite (red arrows). C- rhombohedral quartz (Qz) and grains of calcite (black arrow). Naokelekan Formation, sample BN7 of Barzinja section.

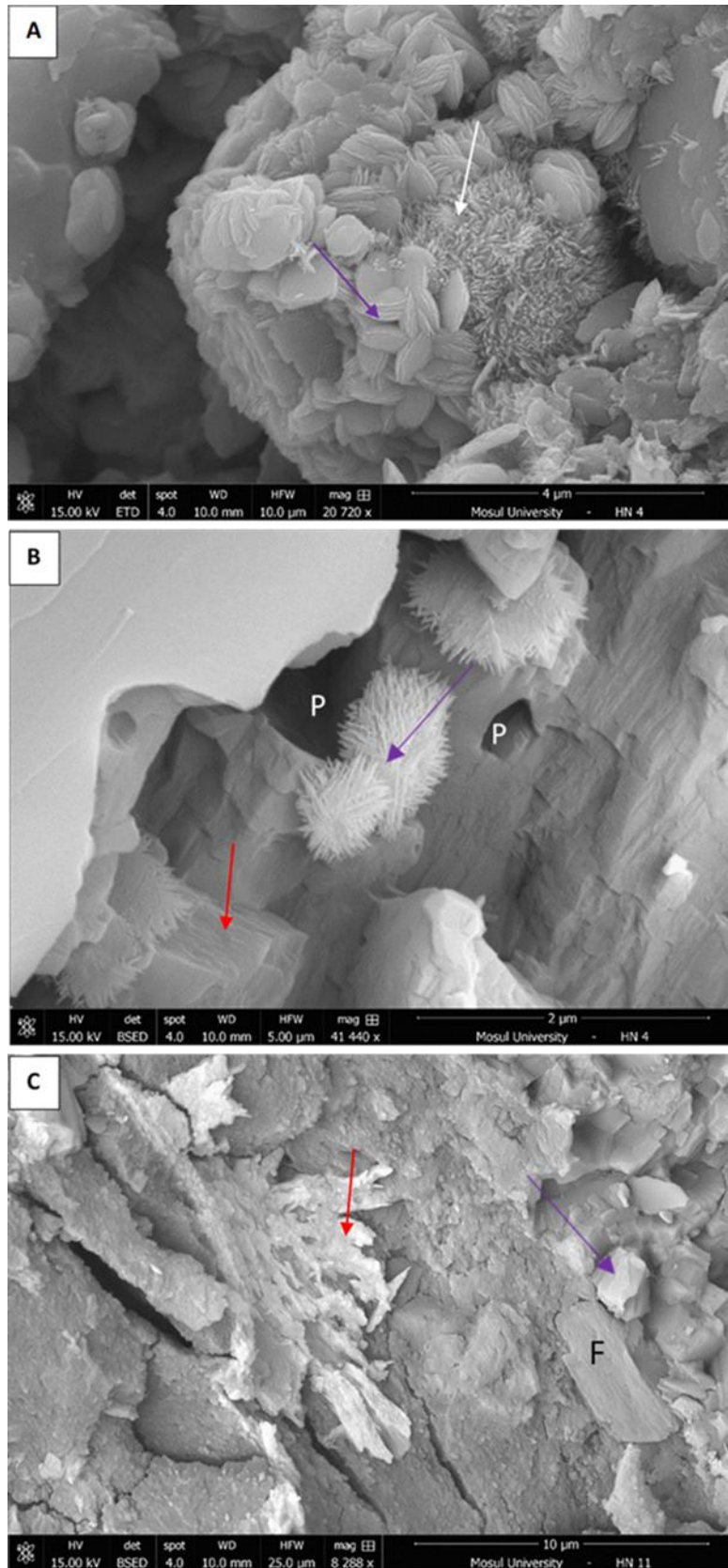


Fig. 7. Scanning electron microscopic images showing: A- Disc- shaped calcite (violet arrow), and star-shaped calcite (white arrow). B- columnar calcite (red arrow), star-shaped (violet arrow) and pores (P). C- radial calcite (red arrow), scattered calcite grains and feldspar (F), samples HN4 and 11, from the Naokelekan Formation at Halabja section.

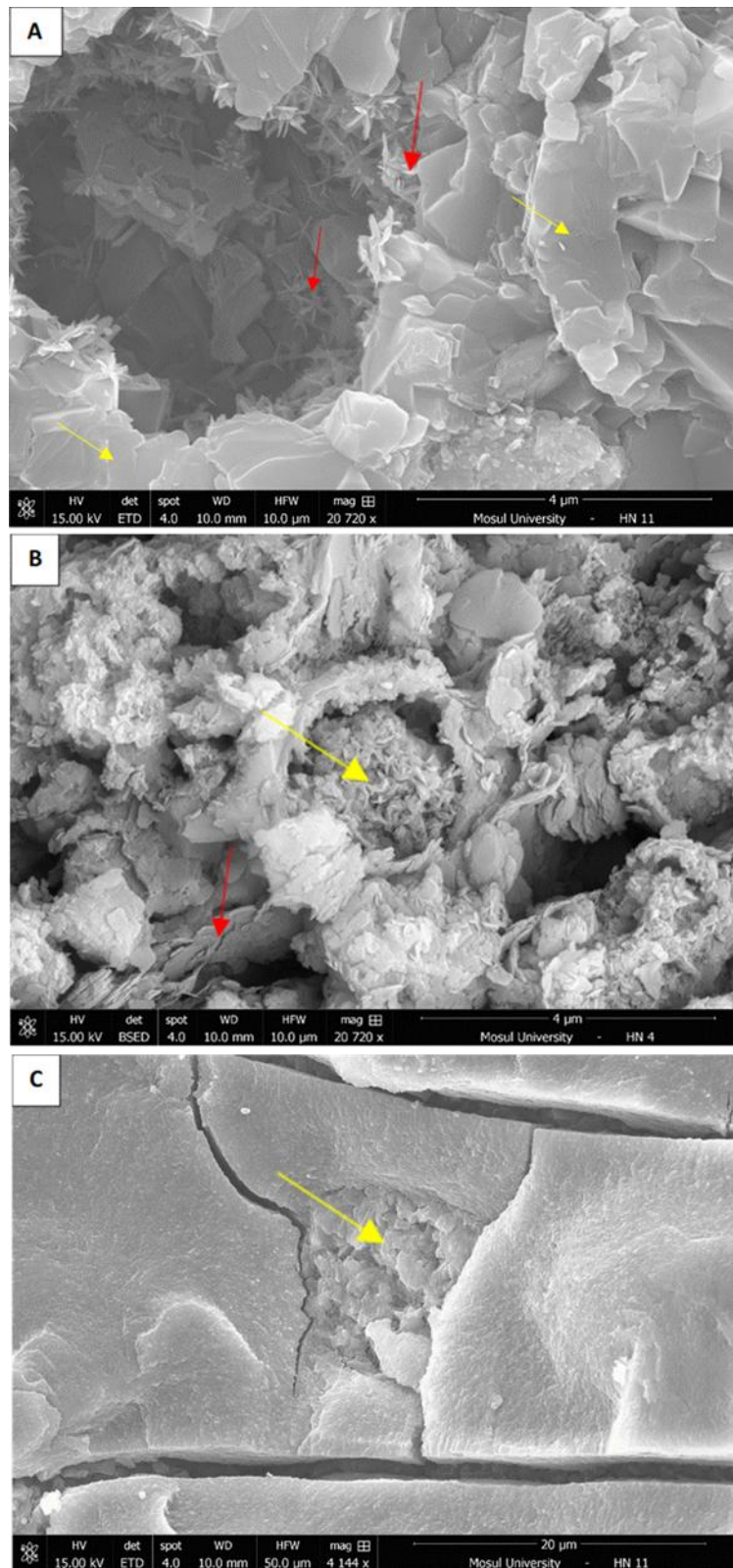


Fig. 8. Scanning electron microscopic images showing: A- star-shaped calcite (red arrows), and bladed calcite (yellow arrows). B- Illite in fibrous (yellow arrow) and platy (red arrow) forms. C- platy illite (yellow arrow) filling pores in fractured limestone, samples HN4 and 11, from Naokelekan Formation at Halabja section.

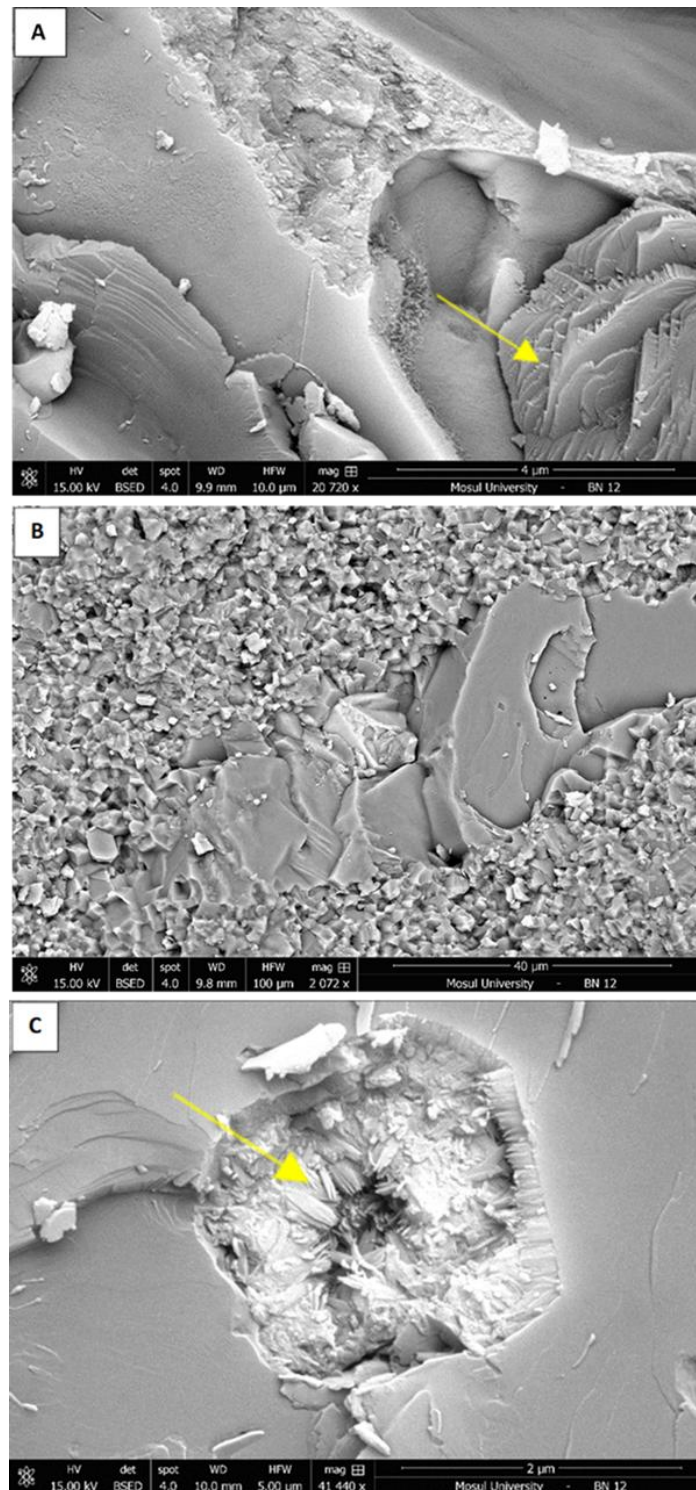


Fig. 9. Scanning electron microscopic images showing: A- bladed platy calcite (yellow arrow). B- microspar and blocky calcite cement. C- fibrous and radial calcite filling pore (yellow arrow). Sample BN12, from the Naokelekan Formation at the Barzinja section.

Conclusion

X-ray diffraction analysis supported by scanning electron microscopy of the limestone and alternated calcareous shale of the Naokelekan Formation has revealed a predominance of calcite, while dolomite is rare. The framework silicates are represented by quartz and feldspars. Some other minerals, such as pyrite, goethite, natrolite, and apatite, are recorded as well. On the other hand, the clay minerals are dominated by illite (mica) and mixed layers of illite-smectite. Predominance of illite (mica) reflects the hot arid paleoclimatic conditions. The presence of calcite in various forms, such as star-shaped, columnar, and euhedral, disc-shaped,

and fibrous calcite, reflects the shallow marine to lagoonal depositional environment, in addition to diagenetic, inorganic mineralization, and biogenic contribution. Framework silicates of quartz and feldspars reflect the detrital contribution due to weathering from acidic igneous sources.

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