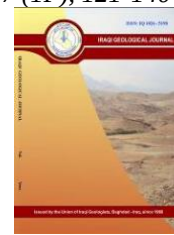




Iraqi Geological Journal

Journal homepage: <https://www.igi-iraq.org>



Provenance Analysis and Diagenesis Using Integrated SEM-Cathodoluminescence and Optical Microscopy for the Sandstones of the Pirispiki Formation, Kurdistan Region, Northern Iraq

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Abstract

Received:
16 February 2024

Accepted:
14 April 2024

Published:
30 June 2024

The paragenetic history and provenance of framework grains in marginal marine and deltaic sandstones of the Pirispiki Formation in two sections, (Ora and Kaista), northern Iraq were examined. This was achieved using an integrated approach employing scanning electron microscopy-cathodoluminescence (SEM-CL), and optical microscopy. The Pirispiki Formation is lithologically characterized by alternating layers of thin- and thick-bedded sandstones. These were intercalated with thin laminae of grey-black shale. Petrographic analysis revealed that the dominant detrital grains in the mature quartz arenite sandstone were monocrystalline quartz grains with undulose extinction. The diagenetic analysis of the sandstones documented several processes that occur in three distinct stages. The SEM-CL technique enabled the identification of two distinct quartz overgrowth phases. Quartz cementations are of two types, the earliest phase and the most significant by volume is the Q1 cement characterized by dark non-luminescence which led to the destruction of most of the primary pores and the healing of mechanically induced cracks. Q2 cement is formed after major compaction and is characterized by bright luminescence with sector zoning, revealing an intricate SEM-CL zonation. CL revealed five special textures within detrital quartz grains including healed fractures and zoning. The provenance of the detrital quartz grains indicated the dominance of plutonic and metamorphic sources, with trace amounts originating from volcanic sources. These were mostly derived from the Bitlis Massif Zone in Turkey.

Keywords: Provenance; Cathodoluminescence; Quartz cement; Authigenic chlorite; Pirispiki Formation; Northern Iraq

1. Introduction

The petrographic analysis of sandstone frameworks depends on the provenance parameters such as the source rock lithology, climate, weathering, transport, and geotectonic settings (Dickinson, 1985; Omer, 2015; Kettanah et al., 2020; Nkongho et al., 2022; Ghasemlooytakantapeh, 2023). The scanning electron microscopy-cathodoluminescence (SEM-CL) technology has become an essential tool for sedimentologists because of its ability to reveal new features in siliciclastic sedimentary rocks (e.g. diagenesis, provenance), distinctive features and fabrics such as different types of quartz cementation (Milliken, 1994; Seyedolali et al., 1997; Richter et al., 2003; Friis et al., 2010; Omer and Friis, 2014). Quartz cements are the most common types observed in quartz arenites and are thought to be the principal cause of sandstones' decreased primary porosity (McBride, 1989; Haddad et al., 2006; Omer and Friis, 2018). They are crucial authigenic components in diagenesis and have little impact on the

DOI: [10.46717/igi.57.1F.11ms-2024-6-20](https://doi.org/10.46717/igi.57.1F.11ms-2024-6-20)

maturity and quality of reservoir rocks. Nonetheless, the sedimentary basin's tectonic settings frequently have a significant impact on other variables (Pettijhon et al., 1987; Armstrong-Altrin and Verma, 2005; Guojun et al., 2011; Ghaznavi et al., 2018; Radwan, 2022).

The Pirispiki Formation, in northern Iraq (L. Devonian-E. Carboniferous), is selected in this research. It is characterized by alternating thin and thick-bedded sandstone layers. The quartz is the most stable mineral in the sandstones. Previous sedimentological studies are mainly based on facies analysis of the sandstones of the Pirispiki Formation. Al-Hadidy (2001), identified two facies for the Pirispiki Formation red sandstone- mudstone facies and grey-to-red sandstone facies intercalated with shale, representing floodplain deposits and an interchannel environment. The depositional environments of the Pirispiki Formation are suggested to be continental, marginal marine to deltaic, and shore-face.

Accordingly, it is vital to focus on the provenance of quartz cement that led to the elimination particularly the primary porosity and diagenesis analysis within the sandstones of Pirispiki Formation.

This study aims to enhance understanding of the provenance and tectonic setting of sandstones of the Pirispiki Formation by using SEM- CL and Optical Microscopy (OM) techniques to investigate and determine the origin of detrital quartz grains and the diagenetic processes of the sandstones of Pirispiki Formation, especially the quartz cement phases and the paragenetic sequence.

1.2. Geological Setting

The distribution of Paleozoic rocks in northern Iraq is complex and often appears as isolated patches and limbs of anticlines in the Northern Thrust and Imbricated Zones of Iraq (Fig. 1). The boundaries of these formations are somewhat unclear and are typically characterized by a relatively steep-angle reverse or thrust fault (Buday and Jassim, 1987). The siliciclastic successions of the Pirispiki Formation are located near the Iraqi-Turkish border within the Northern Thrust Fault Zone (Fig. 1). Pirispiki Formation was first introduced by Wetzel (1950). Furthermore, Bellen et al. (1959) suggested that the Pirispiki and Chalki formations should be considered together because the latter forms an integral part of the Pirispiki Formation. Petrographic studies of the Chalki rocks revealed a low-grade metamorphism (Ali et al., 2016). The original thickness of the formation, as described by Bellen et al. (1959), was 83 m. It comprises thin- to medium-bedded yellowish-brown well-bedded sandstones intercalated with thin-laminated gray and- red shale.

Tectonically, Iraq occupies the northeastern part of the Arabian Microplate and collides with the Iranian Microplate. Fouad (2010) divided tectonic of the outer platform of Iraq into four tectonic zones: the Imbricate Zone, High-Fold Zone, Low-Fold Zone, and Mesopotamia Foredeep (Fouad, 2010). According to Fouad (2010), the Pirispiki Formation is located within the Imbricate Zone.

2. Materials and Methods

The siliciclastic deposits from the Pirispiki Formation in northern Iraq were studied on two distinct outcrops (Figs. 1 and 2). Vertical sections were logged regularly across both successions, and the data were documented in the field. The Pirispiki Formation is situated between the Khabour Formation (Late Ordovician) and the Ora Formation (Devonian- Late Carboniferous). Both boundaries exhibited an unconformable nature (Figs. 3a, b, c). A total of 27 samples (unweathered) were collected from the succession sandstones of the Ora section (longitude 43° 22' 50" E, latitude 37° 16' 49" N), where Paleozoic formations are successively well exposed (Fig.2-B). An additional 15 fresh samples were gathered from the second section, Kaista (longitude 43° 10' 30" E, latitude 37° 17' 49" N) (Fig. 2-A). Subsequently, 42 polished thin sections were created from the samples collected from the Ora and Kaista sections and studied under a standard Nikon Eclipse LV 100 Pol petrographic microscope at Warsaw University, Poland. Modal analysis was achieved by measuring 500- 600 grains in each polished thin section using the method established by Ingersoll and Suczek (1979) (Tables 1 and 2). The SEM

analyses are accomplished at Aarhus University, Denmark, using a Cam-Scan® MaXim SLi with a LaB6 filament equipped with an energy-dispersive spectrometer micro-analyzer (EDAX) and a CL detector (SEM- CL). Twenty sandstone samples from the Ora and ten from the Kaista sections were prepared for SEM-SE by coating rock chips with gold. The SEM-CL technique was applied to investigate and determine the origin of detrital quartz grains and quartz cement phases (Sikorska and Paczeńska, 1997; Milliken, 2000; Boggs and Krinsley, 2006).

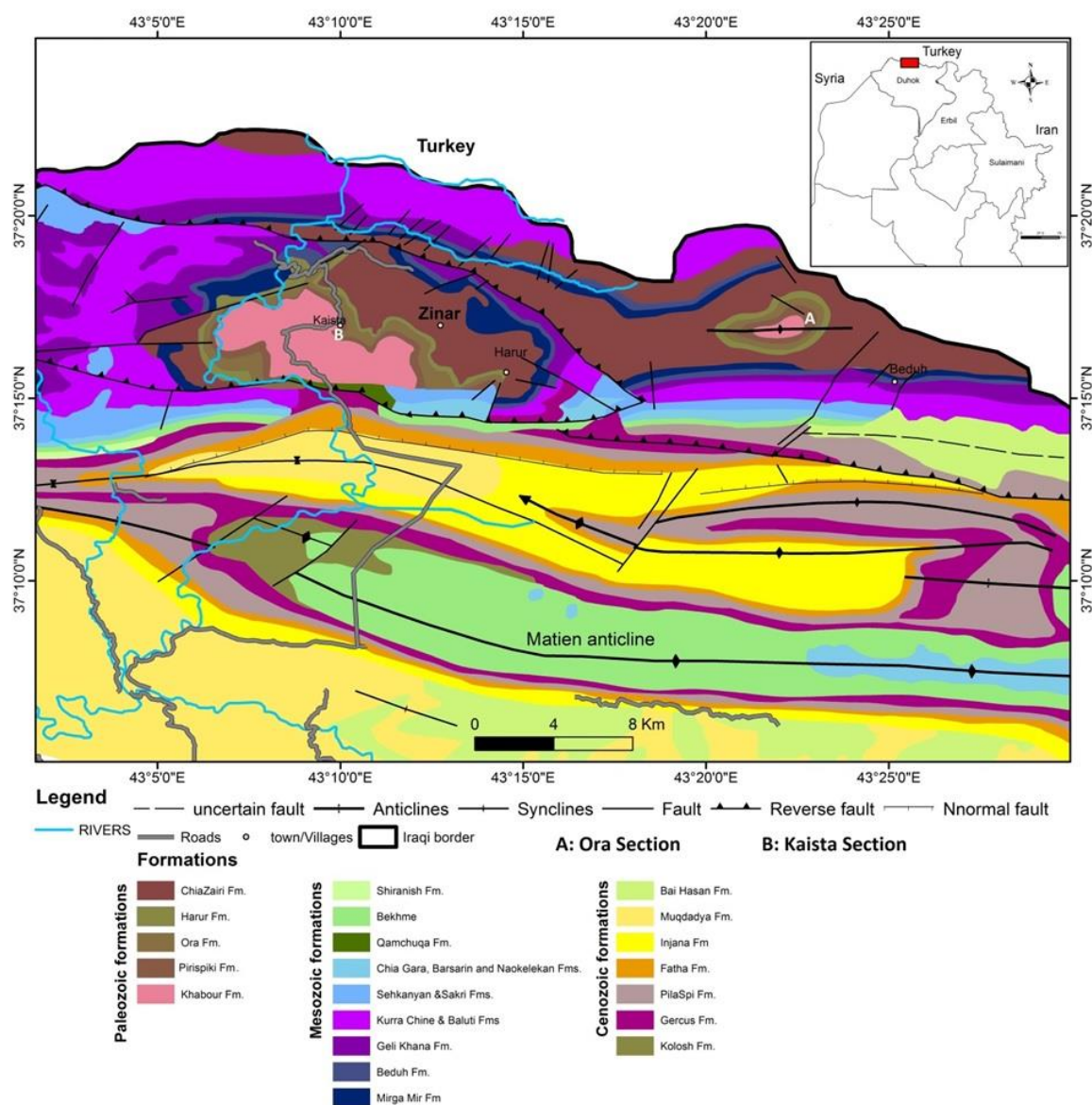


Fig.1. Location and geological map showing Pirispiki Formation and other Paleozoic rocks (modified after Sissakian 2000). A and B Columnar sections of the Pirispiki Formation in two outcrops, (A: Ora and B: Kaista sections).

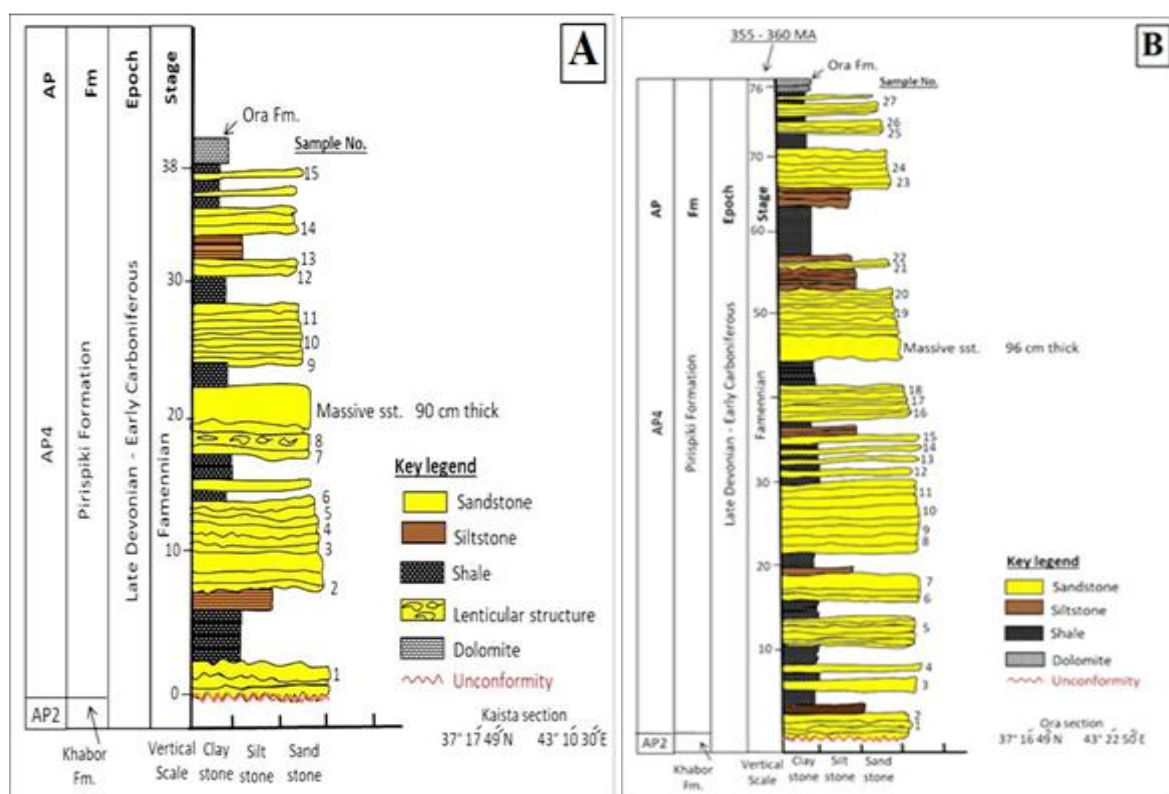


Fig.2. Locations of collected samples of the Pirispiki Formation in two outcrops, A: Kaista and B: Ora Columnar sections.



Fig.3. Field photograph showing lower and upper contacts of the Pirispiki Formation: (a) major unconformity contact (dash line) and undulated surface between Khabour and Pirispiki formations in northern Iraq. (b) close-up view of (a). (c) the upper contact of the Pirispiki Formation with unconformable Ora shale Formation which is represented by fissile of the black to grey shale and current ripples (red arrow) in the thin sandstones of the Ora Formation. a-b: Kaista section; c: Ora section.

Table 1. Modal analysis of the sandstones of the Pirispiki Formation (Ora section), northern Iraq.

Ora Samp.	Quartz			P	R.F.	Mat	Mica			Cements				Porosity		
	Qm	Qp	K				Mu	Ch	Bio	Sil	Cl	Ca	Fer	HM	Pyr	TPC
1	59.8	5.4	4.4	0.7	1.8	1.2	3.4	1.7	2.0	10.5	2.9	0.6	0.4	1.9	1.5	501
2	63.5	3.5	0.9	1.9	0.6	0.9	1.0	0.5	0.0	16.9	2.0	0.0	0.2	2.5	0.6	517
3	53.4	3.9	1.0	0.7	0.4	0.8	2.1	4.7	0.2	14.0	6.5	0.6	3.0	3.0	0.4	528
4	56.5	5.0	1.0	0.7	0.2	2.0	2.6	1.3	0.4	15.9	6.0	0.0	0.5	4.7	0.0	589
5	53.2	4.8	1.7	5.2	1.2	1.2	2.4	1.6	1.9	9.2	6.3	0.0	1.1	1.6	2.3	543
6	55.7	7.9	2.0	1.8	1.3	1.6	2.9	1.0	2.2	10.2	5.0	0.6	2.0	1.5	1.1	541
7	47.1	5.4	0.5	0.3	6.8	1.6	1.2	6.1	0.4	10.4	11.0	0.1	0.4	2.2	3.1	570
8	51.6	6.0	1.8	1.0	5.6	1.6	4.7	2.2	2.1	9.9	5.1	0.9	1.6	1.2	1.1	522
9	53.0	5.4	2.3	1.9	6.0	1.3	2.2	1.9	1.5	10.2	6.1	0.0	1.9	1.4	1.6	590
10	52.8	5.7	2.3	1.0	5.8	1.6	0.9	3.7	1.8	8.6	9.8	0.4	0.0	1.2	1.5	600
11	55.1	5.8	5.1	1.7	1.3	1.0	1.9	1.6	2.3	10.8	5.2	0.0	1.4	1.7	1.0	597
12	58.0	6.4	0.4	1.0	1.4	1.8	1.0	3.0	1.4	10.0	8.1	0.0	1.2	1.7	0.0	593
13	50.4	3.5	2.0	1.6	6.5	2.0	1.7	3.3	0.9	7.7	9.4	0.0	1.3	1.4	2.8	557
14	54.4	6.0	2.2	0.6	5.9	1.4	3.1	2.1	1.0	11.0	6.8	0.1	1.6	1.0	0.7	510
15	55.2	5.3	2.1	1.1	5.7	1.7	0.8	2.4	1.2	7.0	9.2	1.1	1.1	1.7	1.3	593
16	54.4	5.5	1.2	2.2	6.4	2.2	2.0	1.0	1.4	9.3	8.1	0.0	0.0	1.2	1.6	538
17	60.3	6.4	1.2	0.1	0.1	1.5	2.5	1.0	1.8	9.4	5.7	1.1	2.6	1.9	1.2	508
18	56.8	6.3	6.3	2.5	1.3	1.2	1.2	0.4	0.9	9.2	4.4	1.8	1.5	2.1	1.0	520
19	55.4	5.1	4.9	2.5	2.5	1.1	2.4	0.3	0.9	10.5	4.1	1.0	1.2	2.3	0.8	559
20	58.9	5.4	0.9	0.8	1.0	0.9	1.7	1.0	0.9	16.1	3.2	0.6	1.0	2.8	0.9	555
21	57.9	5.7	0.9	0.7	0.3	0.8	1.5	1.1	1.3	13.9	4.3	0.0	1.7	2.9	1.4	545
22	58.5	4.4	1.5	0.3	0.5	0.7	1.1	0.7	1.2	15.7	5.0	0.8	1.4	3.0	1.4	581
23	59.2	6.0	1.8	0.2	0.7	0.4	1.2	0.4	0.2	16.3	5.1	0.2	1.8	2.6	0.5	500
24	58.5	5.9	1.2	0.2	1.1	0.8	1.8	0.5	1.2	13.8	6.0	0.6	2.2	1.7	0.0	575
25	58.9	5.5	1.0	0.6	1.2	0.5	1.6	0.7	0.5	15.1	5.2	0.9	1.6	1.9	0.0	596
26	60.0	5.3	1.1	0.2	1.0	1.1	1.7	1.0	0.9	13.2	6.6	0.3	1.0	1.8	0.9	600
27	62.9	4.8	1.5	0.6	1.1	0.9	2.2	1.0	1.3	12.5	4.9	0.0	0.8	1.4	1.0	551
Range	47.1- 63.5	3.5- 7.9	0.4- 6.3	0.1- 5.2	0.1- 6.8	0.4- 2.2	0.8- 4.7	0.3- 6.1	0.0- 2.3	7.0- 16.9	2.0- 11.0	0.0- 1.8	0.0- 3.0	1.0- 4.7	0.0- 3.1	1.2- 4.0
Mean	56.3	5.4	1.9	1.2	2.5	1.2	2.0	1.7	1.1	11.7	6.0	0.4	1.2	2.0	1.1	2.2

Table 2. Modal analysis of the sandstones of the Pirispiki Formation (Kaista section), northern Iraq.

Kai. Samp.	Quartz		K	P	R.F.	Mat	Mica			Cements				Porosity				
	Qm	Qp					Mu	Ch	Bio	Sil	Cl	Ca	Fer	HM	Pyr	PP	SP	TPC
1	64.1	4.3	1.0	0.6	0.7	1.0	3.2	2.0	1.2	10.5	4.2	0.2	0.7	1.5	1.3	0.9	2.0	565
2	60.1	4.4	0.9	1.2	0.7	1.0	2.2	1.9	0.7	12.6	5.1	0.9	0.9	2.9	1.2	0.5	1.9	523
3	63.4	5.5	1.0	0.3	1.0	0.9	1.7	2.4	1.0	9.7	4.1	0.6	0.8	2.4	0.9	1.4	2.4	580
4	53.7	5.8	5.2	2.9	1.0	1.3	2.2	1.5	1.4	9.3	5.0	0.0	1.1	1.6	2.3	1.7	3.2	563
5	52.5	3.4	2.0	1.3	6.9	2.1	1.6	3.0	1.6	10.0	6.3	0.0	1.9	1.3	1.3	0.9	2.0	572
6	53.1	6.0	4.1	1.5	1.0	1.8	1.9	2.0	1.5	11.7	5.4	0.6	1.9	1.3	1.5	0.6	2.4	595
7	52.8	5.3	2.3	1.4	5.9	1.0	2.7	2.4	1.7	9.9	6.2	0.0	2.4	1.5	1.0	0.6	1.9	600
8	53.5	6.0	1.9	1.3	5.4	1.7	1.8	2.9	1.6	9.6	8.8	0.0	1.0	1.2	0.4	0.8	2.1	592
9	62.0	5.9	1.0	0.2	0.5	0.4	1.1	0.5	0.2	14.4	5.1	0.2	1.5	2.6	0.5	1.4	2.0	567
10	59.7	5.9	1.2	0.3	0.6	0.5	1.9	1.7	0.4	13.1	6.2	0.6	2.1	1.7	0.0	1.5	1.7	525
11	60.5	5.3	1.3	0.4	1.2	0.5	1.4	0.7	0.4	14.8	5.0	0.9	1.6	2.1	0.0	1.6	2.3	569
12	63.3	4.9	1.0	0.5	0.3	0.2	1.7	1.3	0.9	12.6	4.8	0.5	1.1	2.7	0.0	1.3	2.0	581
13	62.7	5.4	1.3	0.2	0.4	0.7	1.9	1.6	1.0	13.4	4.2	0.3	0.0	2.0	0.5	1.1	2.7	533
14	61.9	5.7	1.1	0.6	0.8	1.0	2.0	1.3	1.2	14.7	4.0	0.2	0.0	1.7	0.2	1.0	1.8	516
15	63.4	5.3	1.2	0.7	0.3	0.8	1.8	1.4	1.5	13.4	4.4	0.0	0.2	1.9	0.1	1.6	2.9	590
Range	52.5-	3.4-	0.9-	0.2-	0.3-	0.2-	1.1-	0.5-	0.2-	9.3-	4.0-	0.0-	0.0-	1.2-	0.0-	0.5-	1.7-	
	64.1	6.0	5.2	2.9	6.9	2.1	3.2	3.0	1.7	14.8	8.8	0.9	2.4	2.9	2.3	1.7	3.2	
Mean	59.1	5.3	1.7	0.9	1.8	1.0	1.9	1.7	1.1	11.9	5.2	0.3	1.1	1.9	0.7	1.1	2.2	

Ora: Ora section; Kai: Kaista section; Samp.: Sample number; Qm: Monocrystalline quartz; Qp: Polycrystalline quartz; K: Potash feldspar;

P: Plagioclase feldspar; R.F: Rock fragment; Mat: Matrix; Mu: Muscovite; Ch: Chlorite; Sil: Silica cement; Cl: Clay cement; Ca: Calcite cement; Fe: Ferruginous;

HM: Heavy minerals; Pyr: Pyrite; PP: Primary porosity; SP: Secondary porosity; TPC: Total point counts.

3. Results

3.1. Sandstone Petrography

The results indicated that the Quartz grains are the predominant detrital components of the Pirispiki Formation sandstones, with a considerable proportion of monocrystalline (Tables 1 and 2). Monocrystalline quartz appears in three forms: non-undulose, slightly undulose ($< 5^\circ$), and undulose ($> 5^\circ$); this follows the terminology established by Scholle (1979), Basu (1985), and Tortosa et al. (1991). The proportion of monocrystalline quartz (Qm) ranges from 47.1% to 63.5% and 52.5% to 64.1% in the Ora and Kaista sections, respectively (Tables 1 and 2). Common grain contacts include long concave-convex, and suture types, with point contacts being rare (Figs. 4-a, b, and d) Although minor in quantity, polycrystalline quartz grains are also present (Fig. 4-c).

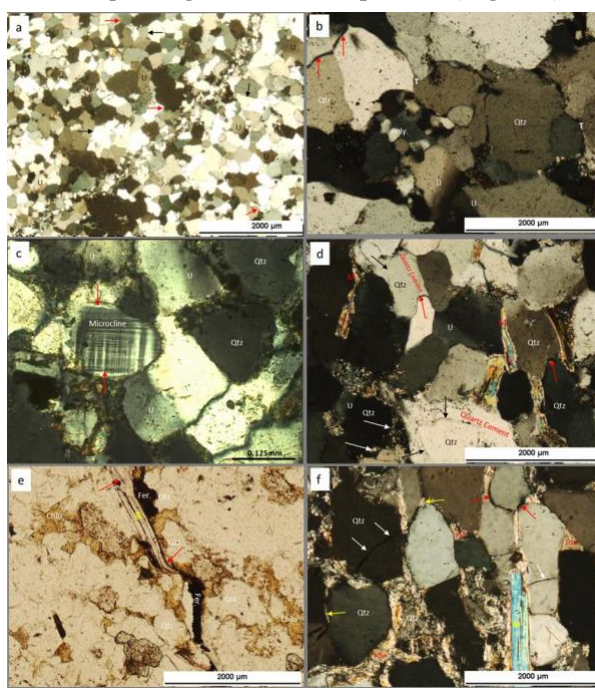


Fig.4. Photomicrographs of framework grains in the sandstones of the Pirispiki Formation under cross-polarized light (XPL). (a) quartz arenite sandstones, where quartz grains are packed and rounded, moderately sorted, and well cemented. Common contacts between quartz grains are long (black arrow) with undulatory extinction and concave-convex (red arrow) (Ora section, sample 7). Pressure solution observed by thick-dark lines (red arrow). (b) Polycrystalline quartz grains showing slightly undulose extinction with curved intercrystal boundaries (Poly.) (Ora section, sample 2). (c) Microcline grain with tartan twinning observed on the lower and upper margin of grain (red arrow) (Ora section, sample1). (d) heavily packed and subrounded quartz arenite sandstone showing thin rims of iron oxide surrounding detrital quartz grain representing authigenic mineral of quartz overgrowths reduced pores (black arrow). Matrix at boundaries of quartz grains (white arrow), pervasive concave-convex contact between monocrystalline quartz grains (red arrow). Mica flakes muscovite are mostly oriented parallel with detrital quartz grains, with little bending of mica an indication of moderate mechanical compaction (Sample, 9 Kaista section). (e) Ferruginous cement fills pores and occurs, after both chlorite and quartz cement. Thus most of the primary porosity, between detrital grains is reduced by chlorite cement, a little bending of muscovite grain (red arrow) (Sample, 6 Ora section, PPL), (f) Medium grained quartz arenite sandstones; minor fractures (white arrow) and pressure solution (red arrow) as a result of heavy compaction of the sandstones. Chlorite coatings around detrital quartz grains (yellow arrow), and chlorite cement occluded primary porosity and most of the intergranular pores, minor quartz overgrowth (Sample, 3 Ora section). Qtz: quartz grain; Poly: polycrystalline quartz grain; M: Muscovite grain; T: triple junctions; U: undulose extinction; Fer: Ferruginous cement, IGP: Intergranular pores.

Feldspar (mainly Orthoclase and Microcline) is the second most dominant mineral, with mostly subangular to sub-rounded outlines (Figs. 4-a, b, and c). These sandstones have a remarkably low matrix content compared to cement (Tables 1 and 2). In the Ora and Kaista sections, the average matrix contents of the sandstones are 1.2% and 1%, respectively. The matrix was primarily composed of very fine-grained quartz (< 0.02 mm) and detrital clay minerals (Fig. 4d). Lithic grains, predominantly silt-sized chert, and crushed muscovite, were more abundant than fragments of metamorphic rocks.

Mica, comprising 0.0 – 6.1% of framework grains, is a minor framework grain in two sections. Generally appears as elongated muscovite flakes, chlorite, and occasionally as biotite, often buckling and bending around the quartz grains (Figs. 4-d, e, and f). Chlorite is more common in the thick successions of sandstones than in the thin sandstones of the Pirispiki Formation. Heavy minerals, constituting less than 2.5% of the total, were a minor component in the two studied sections, these minerals are dominated by well-rounded zircon grains, along with tourmaline and rutile grains, which are characterized as ultrastable heavy minerals.

According to Folk et al. (1970) classification, the sandstones of the Pirispiki Formation as mature quartz arenite, subarkose, and immature sublitharenites (Fig. 5a). The QtFR tectonic indicator in the ternary diagram based on Dickinson et al., (1983), shows that most sandstones of the Pirispiki Formation fall within the “Craton Interior” field. However, a few samples were plotted at the boundary area between the “Craton Interior” and the Recycled Orogen” fields (Fig. 5b). This argues that most of the samples originated from stable zones of a craton, with a few samples from recycled orogens (Dickinson et al., 1983).

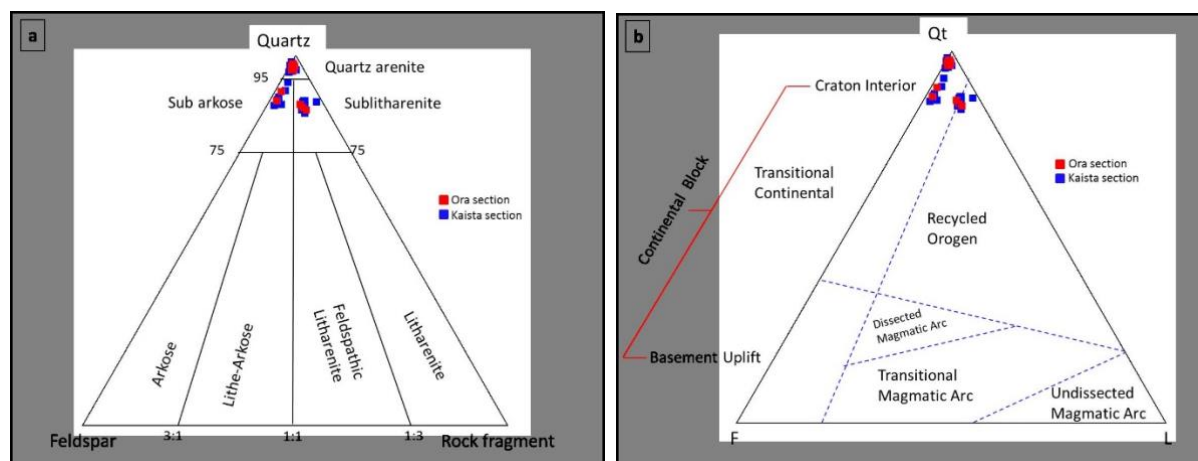


Fig.5. (a) classification of the sandstones of Pirispiki Formation based on Folk et al., (1970). (b) Qt F L tectonic discrimination diagram by Dickinson et al., (1983) applied on the detrital components of the Pirispiki sandstones (Qt is the total quartz, F is the total feldspar, L is the total rock fragments; the dotted lines mark the boundaries between the major fields of tectonic provenance).

3.2. Diagenetic Processes

Diagenetic processes occur in three stages: early eogenesis, middle mesogenesis, and late-stage telogenesis, which are mostly close to fractures and uplifts. The most important diagenetic processes of the sandstones of the Pirispiki Formation are compaction, dissolution, Pyritization, and cementation (Fig. 6). The paragenetic sequence of the sandstones was inferred concerning time depended on optical microscopy (OM) and SEM- CL analyses (Fig. 6).

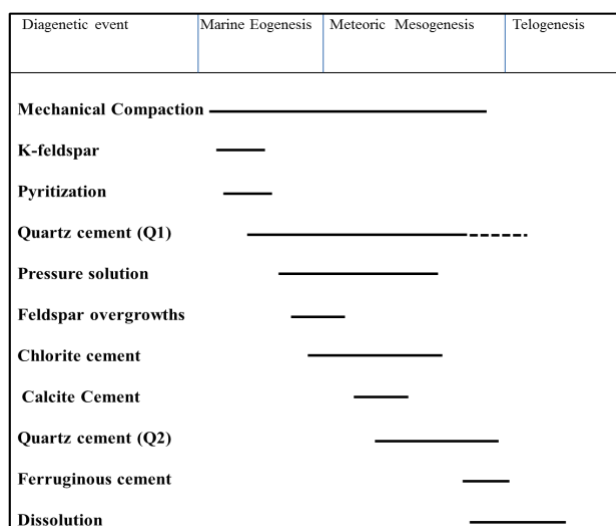


Fig.6. Paragenetic sequence of the sandstones of Pirispiki Formation, northern Iraq.

3.2.1. Compaction

Mechanical compaction is evident in the Pirispiki Formation, as indicated by kink bands, rupturing of flexible mica grains (Figs. 4, e, and f), disaggregated rock fragments (e.g., siltstone), and squeezing of grains. Furthermore, chemical compaction was obvious from the mutual stylolite pressure solutions between the detrital grains (Figs. 4-b, and f).

3.2.2. Grain dissolution

The dissolution process is essentially the opposite of those required for cementation (Boggs, 2006). In the studied sandstones, the intensive dissolution of carbonate grains, particularly dolomite, was observed, leading to an increase in the secondary porosity of the sandstone (Fig. 7d).

3.2.3. Pyritization

Pyritization normally occurs during the early stages of eogenesis under reducing conditions in marine environments. As a result, authigenic pyrite was formed and created as cement (Table 2), which appeared in small proportions as framboidal pyrite (Fig. 7). The reducing environments necessary for pyritization are available during the early burial stages when anaerobic bacteria become active (Schieber, 1989; Boucot et al., 2013).

3.2.4. Cementation

Quartz cement is considered as a main diagenesis in the sandstone of the Pirispiki Formation, appearing as syntaxial quartz cement overgrowths around quartz grains in all samples (Figs. 4b, d, and 7a). The average quartz cement ranges from 7.0% to 16.9%, with a mean of 11.7% in the Ora section, and from 9.3% to 14.8%, with a mean of 11.9% in the Kaista Section (Table 2). Quartz cement overgrowth can close the integral porosity (Fig. 4d). Therefore, quartz cementation was the main factor contributing to the destruct primary porosity in the Pirispiki Formation, which is consistent with observations from other Paleozoic rocks (Møller and Friis, 1999; Chima et al., 2018). Based on OM, the thickness of quartz overgrowths ranges from 15 μm to 285 μm (Figs. 4d, and 7a), and the size of the authigenic quartz crystals is generally <25 μm (Fig. 7b). Euhedral crystal faces, or straight links between neighboring grains, are another characteristic that sets quartz cement apart, and occasionally, inclusion-rich detrital quartz grains are next to inclusion-free quartz cement. The evolution and characteristics of

successive quartz cement phases in quartz arenite were studied using SEM-CL results (Table 3). The SEM-CL patterns and intensities of the quartz overgrowth revealed two different phases.

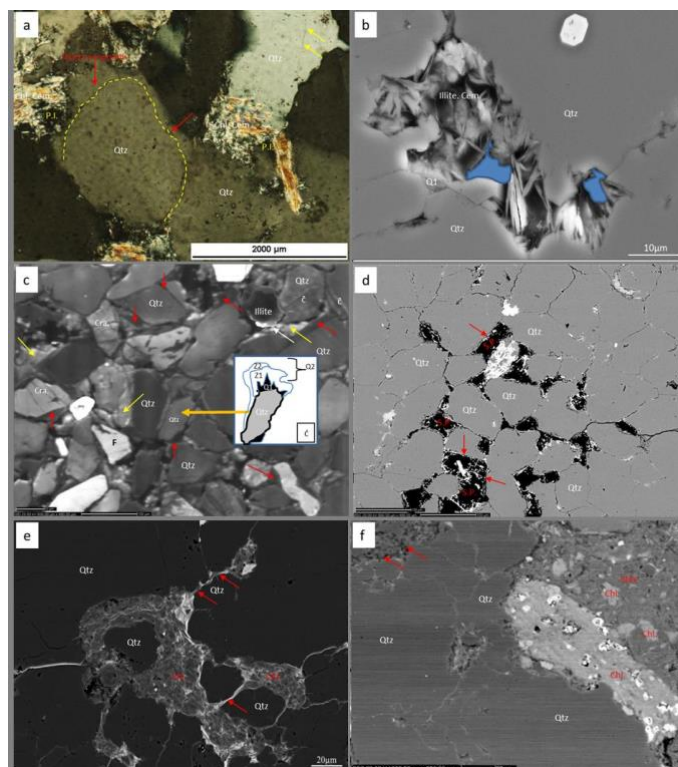


Fig.7. SEM-CL Images and photomicrographs of diagenetic processes in the sandstones of Pirispiki Formation. (a) quartz cement and overgrowths, the primary intergranular pores (primary porosity) remain not filled by chlorite cement. Fluid inclusions trails (yellow arrow) (Sample, 2 Kaista section, XPL). (b) SEM image of quartzarenite sandstone, showing the fibrous habit of authigenic illite and closing pores to prevent quartz cement from growing. Primary intergranular pores (blue color), thin rims of (Q1) cement predated authigenic chlorite (Sample, 13 Kaista section). (c) SEM-CL image of moderately compacted quartz arenite sandstone, showing a large Q1 cement of dark grey non-luminesces developed around detrital quartz grains (red arrow), reduced most of the primary pores and followed by Q2 cement of bright luminescence (yellow arrow). Q2 cement is filling secondary porosity after dissolved feldspar (F), then engulfing authigenic illite and occluding the volume of authigenic illite (Sample 16, Ora section). (d) Sector and growth zoning in Q2 cement including Z1 and Z2, hatching indicates syntaxial relation of cement to detrital quartz grain. Fracture detrital grain (e). (f) BSE image of feldspar dissolution (red arrow) forming secondary porosity (S.P.) (Sample 15, Kaista section). (e) SEM images of medium-grained sandstone are coated by authigenic chlorite (red arrow). Intergranular pores are filled by chlorite cement around quartz grain inhibiting the growth of quartz cement (Sample 1, Ora section). (f) BSE image of immature sublitharenite sandstones, showing a sharp contact between authigenic chlorite and illite. Spot of chlorite cement is spread within illite cement and proportion of authigenic pyrite (red arrow) (Sample 10, Ora section). P.I.: Primary intergranular pores, Chl. cem.: Chlorite cement, Cra.: Cracks healing, F.: Feldspar, S.P.: Secondary porosity, Q1: quartz cement, Z1 and Z2 zonation of Q2 cement.

3.2.4.1. Quartz phase (Zone Q1)

In the sandstones of the Pirispiki Formation, this initially developed as authigenic quartz cement (zone Q1) and is characterized by its dark, non-luminescent nature under CL. This phase starkly contrasts with the subsequent Q2 phase, which emits a bright luminescence. In the Ora section, Q1 percentages range from 2.65 to 4.91 while in the Kaista section, they ranged from 3.78% to 5.82% (Table 3). The Q1 cement precipitated in the primary pores and healed the mechanically induced cracks (Figs.

7b, and c). Despite its darker luminescence, the Q1 phase can be challenging to detect in low-luminescence grains. Typically, the Q1 cement measures between 8 and 25 μm , occasionally developing overgrowths of up to 60 μm in thickness. The thin quartz cement has a uniformly dark appearance. However, the Q1 phase was more volumetrically important in the Kaista section than in the Ora section (Table 3). They commonly have patchy appearances on detrital surfaces. Numerous researchers have reported similar occurrences (Milliken, 2000; Ren et al., 2021). The Q1 cement is the most important crack-healing authigenic quartz phase and fills up to 20 μm wide within mechanically induced cracks in the detrital quartz grains.

Table 3. Percentage point counting of quartz cement for sandstones of Pirispiki Formation based on (CL) study.

Sample No.	Detrital. quartz	Q1%	Q2%	Total counts	Total grid
Or.2	85.71	4.91	8.89	251	300
Or.4	87.87	3.66	7.12	211	250
Or.12	85.28	3.15	9.75	268	300
Ka.2	83.41	5.66	8.14	210	300
Ka.5	88.17	4.11	6.27	233	300
Ka.7	83.40	5.82	7.77	281	300
Or.16	86.22	4.77	6.45	260	300
Ka.9	87.74	4.51	6.78	257	300
Or.17	86.60	3.10	8.80	206	300
Or.20	84.10	4.00	7.90	252	300
Or.21	86.24	2.65	8.99	275	300
Or.25	86.73	3.49	7.79	290	300
Ka.15	86.37	3.78	6.50	216	300
Or.27	86.15	2.90	7.65	287	300

CL: Cathodoluminescence; Or.:Ora section; Ka.:Kaista section

3.2.4.2. Quartz phase (Zone Q2)

The Q2 quartz cement was more easily distinguishable owing to its bright grey variable luminescence under CL (Fig. 7c). This phase is widely spread and typically develops overgrowths that are 12–80 μm thick. In the Ora section, Q2 percentages ranged from 6.45% to 9.75%, whereas in the Kaista section, they ranged between 6.27% and 8.14% (Table 3). The Q2 phase displays sector zoning and growth banding under SEM-CL (Figs. 7c, é). Quartz cement zonation reveals distinctive patterns, sometimes forming lamellar structures that cross the crystallographic boundaries. Sometimes, these patterns merge into a crisscross. Despite its intricate structure, generally, an internal cement stratigraphy that is separated into the zones Z1 and Z2 can be established (Fig. 7b). This complex zonation pattern was also observed by Omer and Friis (2014). This elaborate zonation pattern was visible within the pore-filling cement and was confined to the primary pores. Thin zones are often formed owing to the early precipitation of Q1 quartz cement. With more available pore space, the cement can grow larger and develop into euhedral crystals within the secondary pores. Two types of porosity (primary and secondary) were enclosed (particularly after dissolved feldspar) and were also found in intergranular fractures in the detrital quartz grains (Fig. 7c).

3.2.4.3. Clay cement

These Authigenic minerals (clay cement) formed a second common bond within the Pirispiki Formation sandstones (Tables 1 and 2), with chlorite being the most widespread authigenic clay mineral.

This distribution is typically confined to thick-bedded sandstones. Illite, smectite, and kaolinite are less common and appear mainly in the lower and uppermost sections of the formation. Chlorite is present in two distinct forms: coating and pore-filling substances. The chlorite coatings were mostly observed along the edges of the grain surfaces. These coatings are 7- 10 μm thick and sometimes cover detrital grains completely (Fig. 7e). As a diagenetic feature, chlorite coatings typically form owing to the alteration of detrital clay coats in the primary depositional environment. (Wooldridge et al., 2019). Pore-filling chlorite was observed to grow towards the pore centers between detrital quartz grains, gradually filling the pores with chlorite cement (Figs. 4f, 7e, f). This is a common type of authigenic chlorite.

3.2.4.4. Ferruginous cement

In the sandstones of the Pirispiki Formation, ferruginous cement ranks third among frequent cement (Tables 1 and 2). This cement was frequently seen coating cavities and around detrital quartz grains (Fig. 4e). Iron-rich rock weathering produces ferruginous cement.

3.3. Provenance Analysis of Detrital Quartz Grains

Previous studies on the provenance of quartz grains were primarily based on the CL color, as observed using a luminescope. This helps to distinguish quartz from various source rocks (Zinkernagel, 1978; Marshal, 1988; Augustsson and Bahlburg, 2003). In the Pirispiki Formation sandstones, notable variations were observed in single detrital quartz grains based on their luminescence. These grains showed various kinds of textural fabrics, including patches, dark CL streaks, zoning, and healed fractures (Fig. 8).

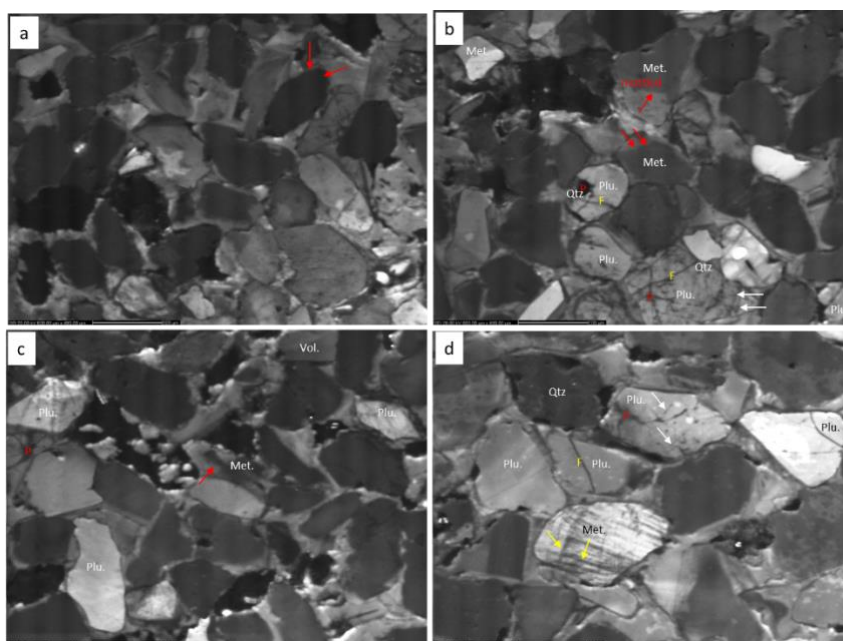


Fig.8. SEM-CL Images of different types of (Plutonic, metamorphic, and volcanic) sandstones of Pirispiki Formation. (a) luminescent pore-filling cement with complex zonation (Sample 13, Kaista section). (b) CL Image of plutonic quartz shows filled fracture (F), dark streaks (white arrow), and patches (P). (c) CL characteristics range from indistinct mottled to nearly uniform (non-differential) metamorphic quartz (red arrow) (Sample 1, Ora section). (c) Heavily cemented quartzarenite sandstone shows three types of quartz (plutonic, metamorphic, and volcanic). Volcanic quartz with oscillatory zone (Sample 4, Ora section). (d) Shocked quartz shows at least two sets of planar fractures an indication of metamorphic quartz (yellow arrow) (Sample 14, Kaista section).

Unfortunately neither an optical microscope nor a BSE microscope could see these occurrences (Seyedolali et al., 1997; Bernet and Bassett, 2005; Hooker and Laubach, 2007). These features help distinguish plutonic, metamorphic, and volcanic quartz. The characteristics of the plutonic rock include healed fractures, dark streaks and patches (Figs. 8b, c, and d), fluid inclusion trails, and slightly undulose extinction (Figure 7a). Thin ($< 8 \mu\text{m}$), clearly visible black fracture lines with weak (CL) or undetectable fractures are seen in many quartz grains. Indistinct mottled patterns of irregular CL intensity across grains (Figs. 9b, c, and d) and shocked quartz with two sets of planar features (Figure 9d) suggest a metamorphic origin (Hooker and Laubach, 2007). However, the contact types of the mineral grains were linear, concave-convex, and punctual (Wang et al., 2021) (Figs. 7c, and d). These microstructures were similar to those found in plutonic and metamorphic quartz by Seyedolali et al. (1997). Volcanic quartz was less common in this study and typically exhibited zoning features or homogeneous CL. In the sandstones derived from volcanic sources, suture contacts comprise the majority of the mineral grain contact types (Fig. 7c). Quartz crystal zoning could be caused by chemical variations in the melt or changes in the rate at which the crystal grows (Watt et al., 1997). The SEM-CL was used to investigate the origins and sources of 200 individual quartz grains from 18 quartz arenite samples from sandstones of the Pirispiki Formation Table 4 and Fig. 9. Three distinct forms of quartz grains are identified by the data, which explain 38% of the origins of metamorphism, 60% of plutonic sources, and 2% of the trace of volcanic provenance.

Table 4. Point-count results of individual quartz grain based on SEM-CL study.

No.	Pl. Qtz %	Vol. Qtz%	Meta. Qtz%	T.P.C.
Or.1	69.7	3.9	26.2	200
Or.2	58.3	3.7	37.9	200
Or.3	59.8	2.1	37.8	190
Or.4	68.0	1.0	30.7	187
Or.6	38.2	0.5	61.3	177
Or.12	60.6	1.2	38.0	191
Or.17	44.0	1.6	54.4	200
Or.21	34.3	2.8	62.5	194
Or.24	70.5	1.0	28.5	179
Or.25	69.5	0.7	29.8	186
Or.27	71.6	1.1	27.3	200
Ka.1	66.9	1.4	31.7	200
Ka.2	69.3	0.9	29.8	195
Ka.3	71.2	0.5	28.0	200
Ka.9	59.7	1.5	38.8	180
Ka.13	44.0	2.2	53.8	192
Ka.14	59.0	1.0	39.5	183
Ka.15	67.2	1.9	30.9	200
Average	60%	2%	38%	

CL: Cathodoluminescence; Or.: Ora section, Ka.: Kaista section; Pl.: Plutonic;

Vol.: Volcanic; Meta.: Metamorphic; Qtz.: Quartz; T.P.C.: Total point counted.

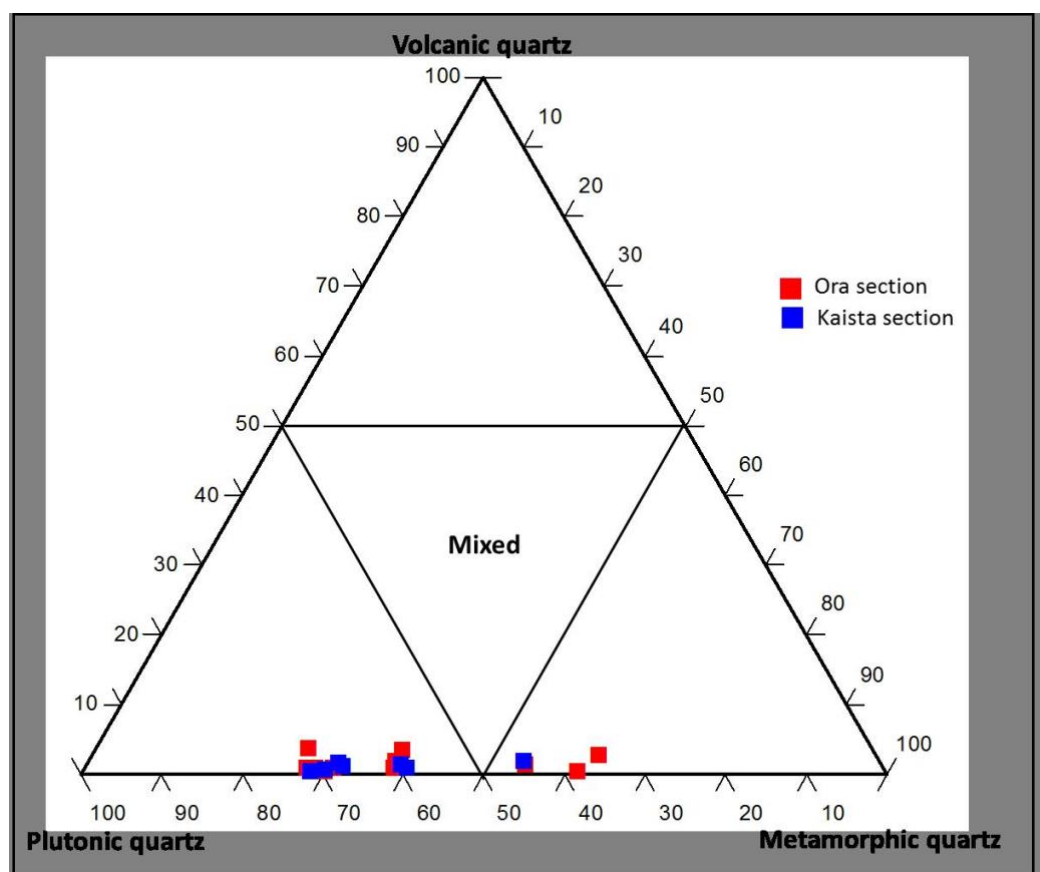


Fig.9. Ternary provenance discrimination diagram of Bernet and Bassett (2005) is applied to the sandstones of the Pirispiki Formation based on SEM-CL study analysis of ~ 200 single grains per sample.

4. Discussion

Petrographic studies are crucial for understanding and interpreting the tectonic settings, sediment provenance, and sediment composition influenced by hydrodynamic control and diagenesis. Quartz cement is the most essential diagenetic mineral affecting the porosity of deep-buried sandstones. It plays an important role in determining hydrocarbon reservoir quality (Worden and Morad, 2000; Molenaar et al., 2008). In general, the authigenic quartz significantly reduced and enclosed the intergranular porosity of the sandstones (Wang et al., 2019) (Figs. 4b, and d). The sandstones of the Pirispiki Formation are mainly thin- to medium-bedded fine- to medium-grained quartzarenites. In the mature to supermature sandstones, single quartz grains (monocrystalline) predominated in the framework grains, with trace amounts of feldspar, rock fragments, and sporadically mica grains (Tables 1 and 2). The diagenesis mechanisms in the sandstones of the Pirispiki Formation were primarily affected by the pore geometry, grain size, and depositional environment. The sandstone samples undergone eogenesis, mesogenesis, and telogenesis, three intense and complicated stages of diagenesis (Fig. 6). The pores are filled with two different phases of quartz cement (Figs. 7b, and c). The detrital quartz grains' surfaces as well as the fractures that healed within them showed the initial phase of quartz cement (Figs. 7b, and c). SEM-CL analysis distinguished this Q1 cement as dark and non-luminescent without zonation, indicating that it is an early stage of quartz cement. This cement is the most essential in terms of volume, with a maximum thickness of up to 80 μm , particularly in the Kaista section (Table 3). The sandstones were subjected to mechanical and chemical compaction during progressive burial. Evidence for this includes kink-folds, flexible mica grains, and pressure solutions (Figs. 4d, and f). The first episode of mechanical compaction formed a concave-convex grain contact, cemented by Q1. Microcracks healed by Q1 cement

indicate quartz cementation post-dated incipient dissolution and extensive mechanical crushing (Figs. 7c, and d) (Milliken, 2000; Ren et al., 2021). This study revealed that most of the primary pores were occluded by mechanical compaction and subsequent quartz cementation (Figs. 4b and 7). Whenever tight quartz cementation occurs at elevated temperatures $>90^{\circ}\text{C}$, the first 2 km of burial are usually the most crucial to compaction (Worden and Morad, 2000; Bernet et al., 2007). Intensive grain fracturing primarily resulted from the strong compaction and pressure solution of the detrital quartz grains before a considerable amount of Q2 cement was added (Figs. 8a, and d). Petrographic and SEM observations indicated that chemical compaction activation mainly occurred immediately after Q1 cement and before any remarkable development of Q2 cement. In this order, the silica required for the synthesis of quartz cement Q2 can be supplied by a pressure solution. Evidence of the elongated detrital quartz grains that frequently form microstylolites and fractures give a pressure solution at the grain interface (Figs. 4b, and f). The sedimentary succession of the Pirispiki Formation primarily comprises thin-, medium-, and thick-bedded sandstones and siltstones intercalated with black to gray shale. Beyond all of the physical and chemical circumstances, quartz cement phases are related to the depositional facies. Thin shale laminations intercalated with sandstone could potentially be a source of silica for quartz cementation and serve as impermeable layers within sandstone bodies. Another possible source of quartz cement is the thin black-to-gray shale laminae found in the Pirispiki Formation, which are interbedded with thinly bedded sandstones. Čyžienė et al. (2006) noted an instance similar to this in the Cambrian Deimena Group sandstones of Lithuania. In this study, thin shale acted as an open system, directing silica toward the intercalated sandstones (Bloch and Hutcheon, 1992). The quartz overgrowth in the related sandstones would be explained by the silica liberated during shale diagenesis. The second phase of quartz cement (Q2) often forms after major compaction and is characterized by its bright grey luminescence with sector zoning under the CL. The Q2 cement follows two pathways. First, it fills both primary and secondary pores following feldspar dissolution. Second, it engulfs authigenic illite and occludes a volume of illite (Fig. 7c). The absence of Q2 cement's zoning and distinguishably strong luminescence in Q1 cement suggests that particular circumstances existed during its precipitation. This is particularly true for the thickly-bedded sandstones of the Pirispiki Formation, which display two styles of chlorite: coating and pore-filling (Figs. 4f, 7e, and f). Secondary porosity occurs because of the dissolution of dolomite cement, volcanic rock fragments, and feldspar grains. The chlorite coatings successfully prevented dissolution in the chlorite-cemented sandstones (Fig. 7e). Chlorite cement serves to fill the dissolved pores and total intergranular pores (IGP) (Fig. 4f). Little dissolution occurs when the detrital grains have been coated with chlorite, according to the SEM and petrographic investigations (Figs. 4d and 7e). This is because the chlorite coating effectively inhibited feldspar grain dissolution. On the other hand, the uncoated detrital grains could have entirely dissolved or partially dissolved, forming a large number of secondary pores (Fig. 7d). Despite the minor provenances and major diagenetic variations, the porosity evolution in the sandstones in this study was mainly determined by the depositional environment (Ärlebrand et al., 2021). The chlorite coating is beneficial for reservoir quality because it inhibits quartz cement formation (Fig. 4f). Guojun et al. (2011) also concluded that chlorite coatings considerably affect the quality of sandstone reservoirs by preventing quartz overgrowth and pressure dissolving to improve pore-throat structures. The thickly-bedded sandstones of the Pirispiki Formation may have received chlorite cement from various sources. During the late eogenesis and early mesogenesis stages, clay minerals such as smectite and kaolinite transform into chlorite as the burial depth increases and higher temperature conditions are reached (Grigsby, 2001). The depositional environments of the Pirispiki Formation included marginal marine and shore faces, Fe-rich chlorite coats, commonly found in marginal marine sandstones, are a possible source (Worden et al., 2020). During the mesogenesis stage, chlorite may have originated from the alteration of feldspar and minor lithic rock fragments. Ferruginous cement filled the pores during mesogenesis in minor amounts; thus, two types of quartz cement and chlorite were dated (Fig. 4e). Feldspar overgrowths, although less

common than quartz cement, are associated with quartz cement in the early to middle stages (Fig. 4c). Under reduced marine environmental conditions, authigenic pyrite formed during the early eogenesis (Fig. 7f).

The sandstones of the Pirispiki Formation are characterized by late diagenetic processes, such as the dissolution of carbonate and feldspar rocks (Fig. 7d). These events typically occur after the main compaction, pressure solution, and cementation when there is a significant kaolinite interstitial fluid, or when the pore fluids are undersaturated relative to carbonate. Consequently, most of the silica overgrowths and some of the detrital quartz grains were dissolved. Thick illite overgrowth coatings inhibited the formation of quartz overgrowths. These unusual mixed-layer clays occur after quartz overgrowth and before calcite cementation (Q1), suggesting that they were formed during the mesogenesis stage (Fig. 7b) (Umar et al., 2011; Zaid, 2013).

The sandstones of the Pirispiki Formation exhibit a mix of five different detrital quartz types: healed fractures, dark streaks and patches, indistinct mottling, shocked quartz, and zoning features (Fig. 8). Most of the quartz particles in the study exhibit healed fractures and dark streaks or patches indicative of a typical plutonic origin, with some metamorphic quartz (Boggs and Krinsley, 2006). These features are caused by tectonism-induced lattice dislocations, which may be displacive crystal development, or the filling of micro-dissolution seams (Seyedolali et al., 1997) (Figs. 8b, c, and d). Plutonic quartz is regarded as the primary source of the sandstone in Pirispiki Formation comprising approximately 60% of the formation. However, the sandstones also feature an indistinct pattern of nondifferential CL, which gives the grains a textured and shocked appearance (Figs. 8c and d). A unique characteristic that suggests that quartz grains with these properties originate from low- and medium-grade metamorphic sources is their irregular mottled textures, which are very common in metamorphic quartz (Seyedolali et al., 1997; Bernet and Bassett, 2005). However, the contact style of the mineral grains in the studied quartz grains indicates linear and punctual contacts, which is evident in the metamorphic-sourced sandstones. In contrast, the volcanic-sourced sandstones primarily exhibit linear and sutured contacts (Figs. 8c and d) (Wang et al., 2021). The observations of detrital quartz grains and CL features suggest that the sandstones of the Pirispiki Formation originated from multiple lithologies or source regions. The predominant sources are plutonic and metamorphic, but there is also a notable, albeit scarce, quantity of detritus that originated from volcanic rocks, as shown by the zonation features (Fig. 8c; Table 4). Furthermore, petrography identified two types of quartz grains: one composed of fewer than four subgrains with straight to undulose extinction and curved inter crystal boundaries (Fig. 4b), indicating plutonic rock origins (Folk, 1970; Blatt et al., 1980), and the other comprising polycrystalline quartz grains composed of more than four subgrains. The latter, featuring a polygonal form and sutured intercrystal boundaries, originates from metamorphic source rocks (Asiedu et al., 2000).

The high percentage of detrital quartz grains in the Pirispiki Formation aligns with characteristics of a “Craton Interior and Recycled orogeny” (Fig. 5b). A plutonic origin is suggested by the comparatively unstrained monocrystalline quartz grains that are present in all of the samples and contain similar inclusions (Hindrix, 2000). According to Al-Harbi and Khan (2008), the low percentages of feldspar and rock fragments—or their complete absence—in numerous samples from the Kaista and Ora sections support a cratonic source with a developed transport regime and moderate chemical weathering under warm, dry climate conditions.

According to Yimaz et al. (1993), the Bitlis Massif in Turkey, an allochthonous unit on a regional scale with a lower-grade cover sequence and a high-grade metamorphic basement, records the closing of the southern Neotethyan Ocean. It was deposited in the Early Miocene along the Arabian continental margin and the foreland basin (Yilmaz et al., 1993; Robertson et al., 2007). The juvenile continent crust of the Bitlis Massif, located roughly 1000 km north of the Arabian-Nubian Shield, is composed of a complex of igneous and metamorphic rocks, including ophiolite units. It is estimated to be between 0.55

and 0.83 Ga in age (Ustaömer et al., 2012). Heavy minerals including rutile, zircon, tourmaline, apatite, biotite, chlorite, and opaques are plentiful in it (Ustaömer et al., 2012).

The petrographic examination of the Pirispiki Formation sandstones also identified various accessory minerals. Given the granitoids of the Bitlis Massif and their heavy mineral concentrations, it is plausible that the provenance of these minerals could be the Bitlis Massif Zone located approximately 150 km northwest of the studied Pirispiki Formation.

5 . Conclusions

- The Pirispiki Formation's sandstones are categorized as immature sublitharenite and mature quartzarenite with subordinate subarkose. The majority of the detrital grains were monocrystalline quartz grains with slight undulose extinction.
- The Pirispiki Formation's sandstones went through several complicated and thorough paragenetic processes, such as eogenesis, mesogenesis, telogenesis, pressure solution, mechanical compaction, quartz cementation (Q1 and Q2), chlorite cement, feldspar overgrowths, ferruginous cementation, and dissolution.
- Two distinct quartz cementation phases were identified. The first phase, Q1, is a dark non-luminescent CL without zonation found both on the surface and inside the cracks of the detrital quartz grains. Q1 cement is considered to be an early-stage quartz that considerably reduces primary porosity. The second phase, Q2, forms after major compaction and is characterized by a bright gray luminescence appearance with sector zoning under the CL, filling the primary and secondary pores after feldspar dissolution.
- Authigenic chlorite cement primarily fills the intergranular pores in the thickly- bedded sandstones of the Pirispiki Formation and occludes the pores during the mesogenetic stages.
- A high proportion of detrital quartz grains and a low proportion of feldspar and rock fragments (unstable grains) are characteristics of mature sediments. These properties point to a recycled orogenic cratonic interior.
- Five CL features were observed in the detrital quartz grains: healed fractures, dark streaks, patches of plutonic origin, indistinct mottled texture, shocked quartz of metamorphic origin, and zoning typical of volcanic origin.
- The quartz CL characteristics of the Pirispiki Formation indicate multiple sources of quartz grains, with major contributions from plutonic and metamorphic sources and notable contributions from volcanic sources. The sediments were probably deposited in a passive margin setting, such as the Bitlis Massif in southeast Turkey, where material from earlier strata was feeding the margin.

Acknowledgements

The present study is part of a broader research program surveying the Paleozoic Formation in northern Iraq. The authors thank the Ministry of Higher Education and Scientific Research, Baghdad for funding this project. I am grateful to Professor Henrik Friis of Aarhus University, Denmark, whose constructive criticism and review considerably improved the quality of this manuscript.

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