

Mineralogy and Geochemistry of Qulqula Limestone in Shenrui, Halabja Governorate, Northeastern Iraq

Nian Wali Samin
College of Basic Education
University of Halabja

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ABSTRACT

This study focuses on the mineralogy and geochemistry of the limestone unit of the Qulqula Formation (Early Cretaceous). The age and stratigraphic position of this formation are controversial because of insufficient studies and the complex structure of the occurrence area. Twenty samples were selected and analyzed which represent six sections: Sarkan, Awer, Griana1, Griana2, Hawar, and Hawara Kon at the Mount of Shenrui in Halabja area located in Kurdistan Region, northeastern Iraq near the Iraqi-Iranian borders. The mineralogical study by the X-ray diffraction technique shows that the samples are composed of 70.14 % calcite, and 28.07% quartz. The geochemical analysis shows that CaO is abundant in most of the studied samples, the increases of (SiO₂, Al₂O₃, Fe₂O₃, TiO₂, and K₂O), and the decrease in (CaO and MgO) leads to the change of limestone to siliceous type. The limited dolomitization can be confirmed by the low concentration. The component Sr has a negative correlation for both major and trace elements. On the other hand, (Cr, Co, Ni, V, and Zr) have a positive correlation with (MgO, Fe₂O₃, SiO₂, Al₂O₃, K₂O, and TiO₂), this may indicate the effect of the basic rocks in surrounding areas enriching limestone with these elements.

Keywords: Mineralogy, Geochemistry, limestone, Qulqula, North Iraq.

معدنية وجيوكيميائية الحجر الجيري لقلقلة في شنروي حلبجة، شمال شرقي العراق

نيان والي سمين
كلية التربية الاساسية
جامعة حلبجة

المخلص

تركز هذه الدراسة على معدنية وجيوكيميائية الحجر الجيري في تكوين قلقللة. يعتبر العمر والموقع الطباقى لهذا التكوين مثيراً للجدل بسبب عدم كفاية الدراسات والتركيب المعقد لمنطقة وجوده. تم تحليل عشرين نموذجاً تمثل ستة مواقع شملت مناطق سركان، وناويرا، وجريانا1، وجريانا2، وهاوار وهاوارا كون في منطقة جبل شنروي بمحافظة حلبجة الواقعة شمال شرقي إقليم كردستان العراق، بالقرب من الحدود العراقية الإيرانية. أظهرت الدراسة المعدنية بتقنية حيود الأشعة السينية أن العينات تتكون من 70.14% من الكالسيت و28.07% من الكوارتز. أظهر التحليل الجيوكيميائي أن CaO وفير في معظم نماذج الدراسة، مما أدى إلى زيادة SiO₂ و Al₂O₃ و Fe₂O₃ و TiO₂ و K₂O، ويؤدي الانخفاض في CaO و MgO إلى تغيير صخور الحجر الجيري جزئياً إلى الحجر الجيري السليكي. اثبتت الدراسة ضعفا ملحوظا في عملية الدلمتة في المنطقة من خلال التركيز المنخفض لعنصر السنترونيوم Sr والعلاقة العكسية بين تراكيز العناصر النزرة وتراكيز العناصر الرئيسية الموجودة في منطقة الدراسة، والزيادة الطردية بين كل من Cr, Co, Ni, V, Zr. من ناحية أخرى، فإن Cr و Co و Ni و V و Zr

لها علاقة إيجابية مع MgO و Fe₂O₃ و SiO₂ و Al₂O₃ و K₂O و TiO₂ مما يشير الى مساهمة للصخور القاعدية في المناطق المجاورة في اغناء صخور الحجر الجيري بهذه العناصر.
الكلمات الدالة: معدنية، جيوكيمياء، حجر جيري، تكوين قفلة، شمالي العراق.

INTRODUCTION

Qulqula Formation was first mentioned by Bolton (1955). The formation is a part of the Qulqula group, which consists of Qulqula radiolarian and Qulqula conglomerate Formations. Qulqula group forms of thick successions of bedded chert, shale, and siliceous limestone (Buday, 1980; Jassim and Goff, 2006). The studied area is characterized by obscured anticlines and synclines, which have been stacked together as very thick and tight packages of layers and overturned towards the southwest or even overthrusts (Raza, 2009).

The studied area is defined as the Crushed Zone, because of its high deformation. The contact of the formations is difficult to be determined and marked because of the intensive folding, faulting, and thrusting (Stocklin, 1968). Qulqula radiolarian series was divided by Jassim *et al.* (1984) into two formations namely Qulqula Formation (Aptian–Albian) and Qulqula conglomerate Formation (Cenomanian), Qulqula conglomerates are composed of thickly conglomerate limestone and cherty grit, interbedded with grey marly shales, chert, and detrital Limestone. Qulqula conglomerate was found at the base of the Qulqula Formation. The conglomerate crops out between Chuarta and Said Sadiq (Karim, 2003). Moreover, Sissakian (2005) mentioned that the contact between Qulqula Formation and Qulqula conglomerate Formation is gradational. In the meantime, Raza, (2009) referred that the lower member of the Qulqula Formation consists of an alternation of more than four detrital limestone successions, each one is more than 25 m thick. The current study aims to identify the mineralogical composition of limestone facies of the Qulqula Formation, as well as to study the geochemistry of major and trace elements and their distribution in limestone to detect the origin and the depositional environment of the formation.

GEOLOGICAL SETTING

The study area is located within the western part of the Zagros Fold-Thrust Belt, which is developed from the basin fill of the Neo-Tethys and deformations that accompanied colliding of the Iranian and Arabian plates (Law *et al.*, 2013). Dunnington, (1958) believed that the thrust zone has developed during the Alpine Orogeny which started from Upper Cretaceous time and continued through the Tertiary, where it is accentuated during the Pliocene time. Stocklin (1968) called the studied area “crushed zone” because it is highly deformed on a large and small scale. According to Karim (2003), the deformation and development of the studied area mainly began from Campanian. (Baziany, 2006) mentioned that the continental part of the Iranian and Arabian plates was collided with Iranian and a foreland basin is formed. Karim (1975) inferred the age of Albian to Cenomanian depending on the study of foraminifera. Structurally, the study area is located within the Imbricated and Thrust Zones (Buday and Jassim, 1987, Jassim and Goff, 2006) which consists of a very long belt in the Thrust Zone.

LOCATION OF THE STUDY AREA

The studied area is located in northeastern Iraq, in the southeastern part of Halabja governorate in the Iraqi Kurdistan Region, where it extends between (35°.07'.47"N - 35°.11'.32" N) and (46° .03'.96"- 46° .08'.57" E (Fig. 1). Six locations are selected for the detailed study of the lower part of the Qulqula limestone Formation (Table-1). The sampling is located at the Mount of Shenrui. The Mount of Shenrui takes a rectangular shape in a southeastern-northwestern direction with a length of 20.2 km, and the average width of the study area from east to west about 10 km covers an area of about 126.03 Km² where its northern edges are adjacent to the Sharzur plain. As for its southern margins, they extend with the Sirwan River for a length of 11.8 km, as it forms a part of the Iraq and Iran border.

Table 1: Location of the Studied Sections

Section	No. of Samples	Location	Thickness(m)
Sarkan	3	46° .03'.96"E 35° .11'.32"N	3
Awera	4	46° .06'.06"E 35° .10'.12"N	8
Griana1	4	46° .08'.57"E 35° .07'.47"N	30
Griana2	3	46° .07'.91"E 35° .07'.95"N	10
Hawar	3	46° .07'.29"E 35° .09'.23"N	3.5
HawaraKon	3	46° .06'.66"E 35° .08'.48"N	5

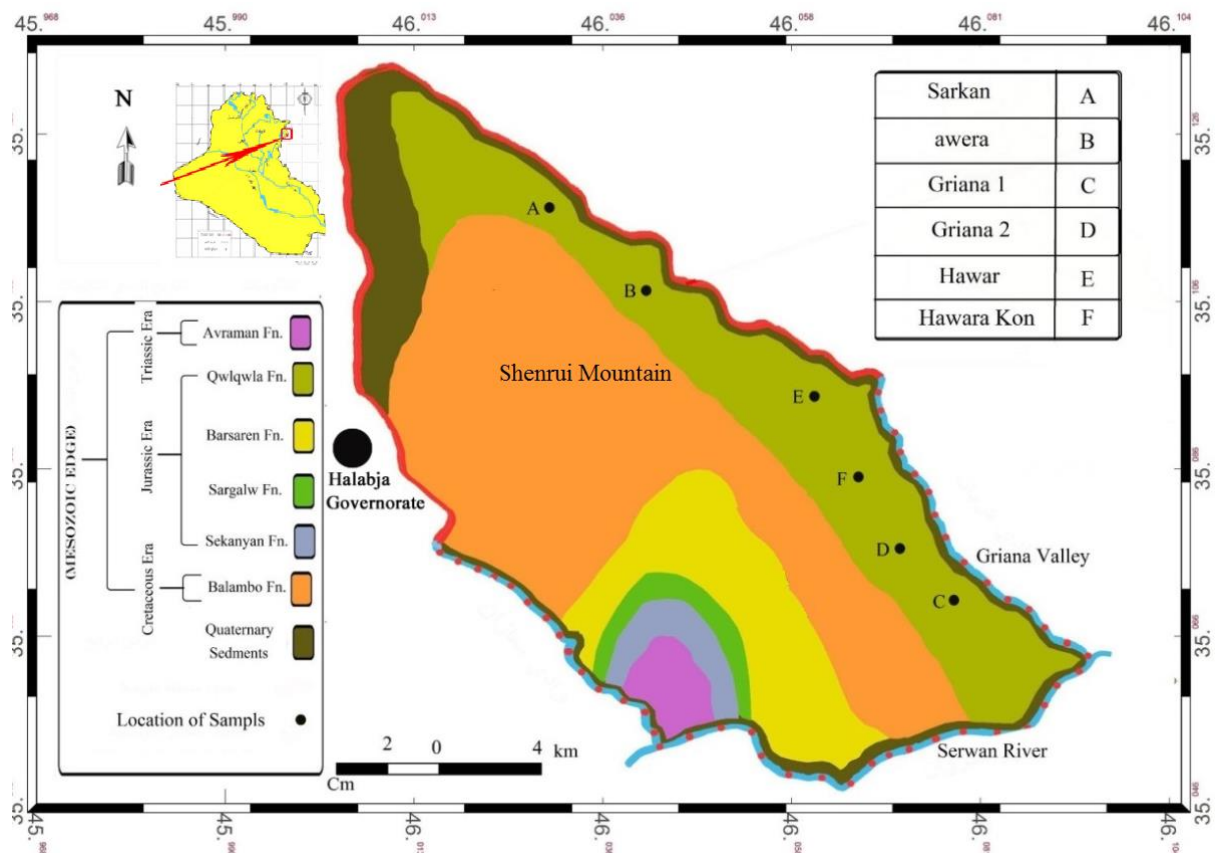


Fig.1: The Geological map of the study area and location of the samples. (Iraq Geological Survey, 2000)

LITHOLOGY OF THE QULQULA FORMATION

The formation consists of different members. The lower member consists predominantly of moderately thickly bedded, oolitic, and detrital limestone with thick beds of white chert. Both limestone and chert layers are inter-bedded with grey marly shale. The middle member is composed of thinly bedded, red and grey, and green shale with intercalation of cherty radiolarian limestone and dark ferruginous shale. Moreover, the upper part is dominated by a thick sequence of dark red ferruginous-siliceous shale and ruby mudstone with occasional beds of oolitic and detrital limestone (Raza, 2009). A conglomeratic bed exists at the base of the Qulqula Formation having a thickness of 0.2–2 m and forming the lower boundary of the formation in the middle part of the studied area. The Formation in the studied area consists of more than four limestone successions; all are situated above the conglomerate bed. A dense interval of alternations of bedded chert and marl separate these successions (Figs.2and3).

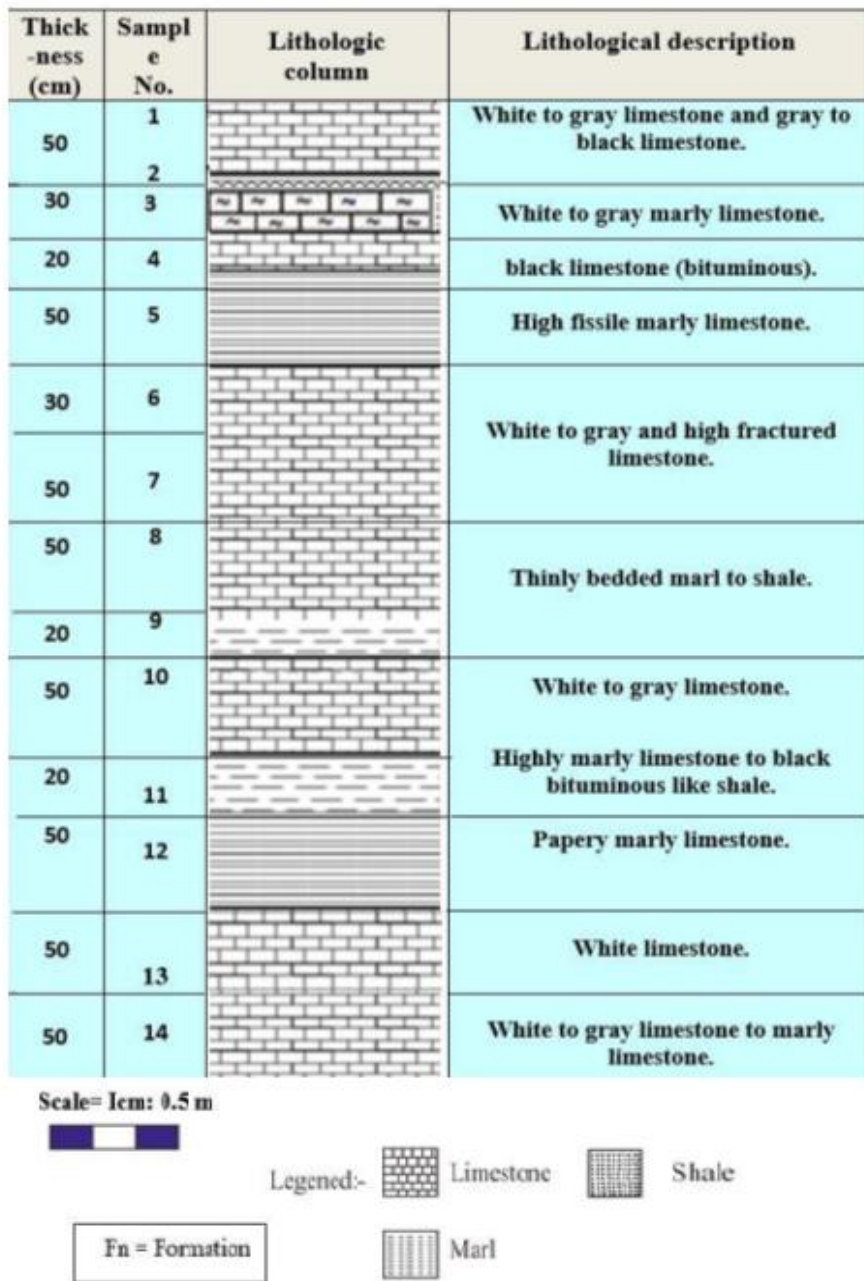


Fig. 2: Stratigraphic Columnar Section of the Qulqula Formation.



Fig. 3: Studied Section

MATERIALS AND METHODS

The fieldwork was carried out in Shenrui Mountain in the Halabja area, including the description of six selected sections from which twenty samples were collected from different beds of the Qulqula Limestone Formation. The laboratory work included preparing samples, crushing, sieving at 75 microns, and then analyzing their mineralogical composition using x-ray diffraction, type D2 phaser Bruker X-ray at Tamad Kala Laboratory in Iran. Several samples were scanned at 2θ between $2-60^\circ$. The samples were also analyzed by x-ray fluorescence to determine their content of major oxides (CaO, MgO, Fe_2O_3 , MnO, SiO_2 , Al_2O_3 , Na_2O , K_2O , TiO_2 , SO_3) and trace elements (Sr, Cr, Co, Ni, V, and Zr). The loss on ignition (L.O.I) of the approximate carbon dioxide content of the same portion of the sample was determined by the weight loss on ignition at $(1000-1100)^\circ\text{C}$ for one hour.

RESULTS AND DISCUSSION

Mineralogy

The x-ray diffraction analyses show that the main minerals of Qulqula limestone are calcite (d spacing: 3.04A° , 2.29A° , 2.1A° and 1.91A° respectively), calcite has a range of 66%-80% and an average of 70% (Table 2, Fig. 4). The second main mineral is quartz, which is identified at (4.2A° , 3.34A° , and 2.4A° respectively) and has a range between 17.33% -34%, with an average of 28 % (Table 2, Fig. 4). It is known that quartz is the most resistant mineral to chemical and mechanical weathering and the type of quartz was a guide to the nature of the source rocks (Al Ubaydi, 2008). The silica formation required a concentration of siliceous materials that were dispersed since the time of deposition. This might be the most probable source for silica in these rocks (Baban and Sarraj, 2007).

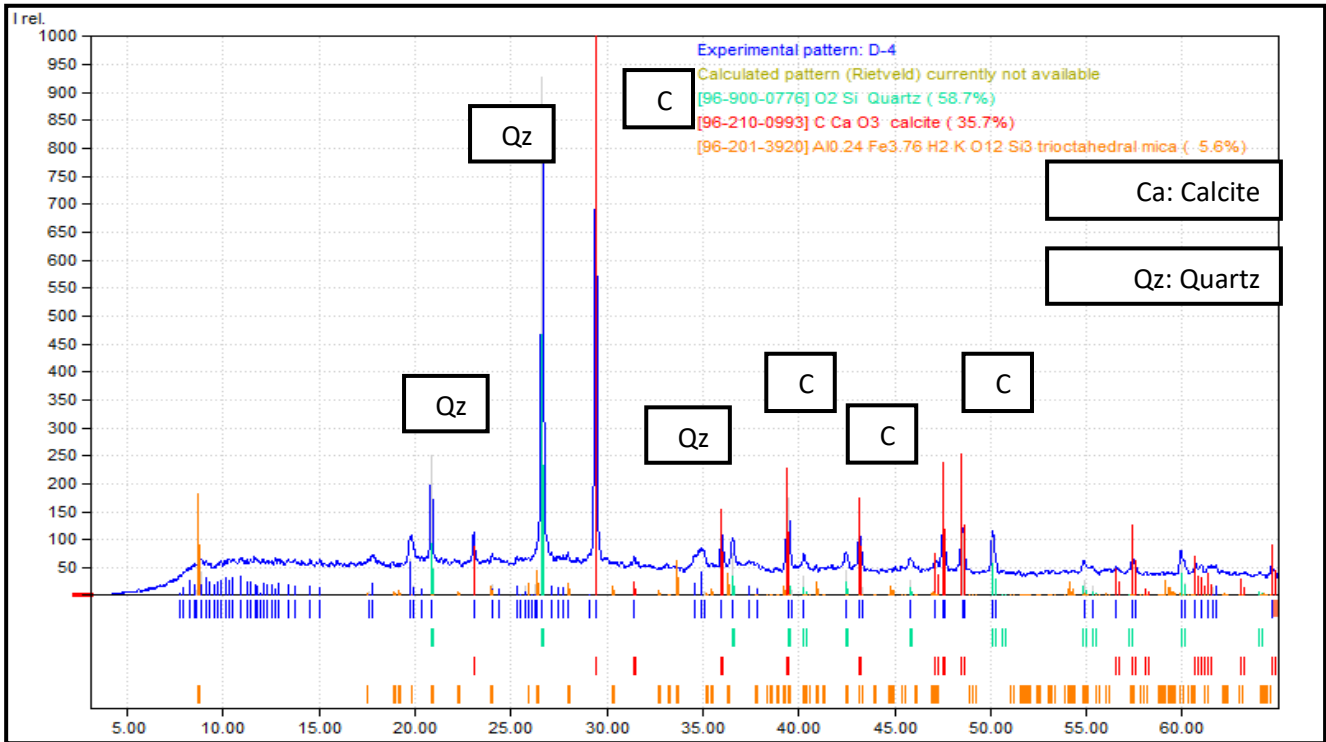


Fig. 4: X-ray diffractogram of Bulk Sample.

Table 2: The Relative Percentage of the Minerals in Selected Samples.

sample	calcite	quartz
A	66%	31%
B	70.8%	28.6%
C	80.211%	17.33%
D	66.465%	32.3%
E	72.88%	25.2%
F	64.475%	34%
Average	70.14	28.07

GEOCHEMISTRY

Major Oxides:

The objective of the analysis of the major elements is to understand the inter-elemental relationships, which would reflect the source rocks of the Qulqula Formation.

1. Calcium Oxide (CaO): The concentration of (CaO) in the studied samples of the Qulqula Formation has a range between (27.95 and 51.22 %) with an average of 39.58% (Table 3). There is a wide variation in the concentrations of CaO in all studied sections, which is attributed to the alteration of limestone to marly limestone in some locations. CaO showed a negative relationship with SiO₂, Al₂O₃, Fe₂O₃, MgO, and K₂O. The negative relationship with SiO₂ is attributed to the silicification, as well as with Al₂O₃. This revealed that clay minerals increase when carbonate constituents

decrease due to dissolution. The clay minerals filled the cavities and pore spaces produced by dissolution processes. This might lead to the same conclusion for the strong negative relation of CaO with TiO₂ and K₂O (Al-Khafaji and Al-Saad, 2019) (Fig. 5).

2. Magnesium Oxide (MgO): The (MgO) ranges between (0.27 and 1.33 %) with an average of 0.59 % (Table 3). Two sources represent the main supplier of magnesium in these rocks. (MgO) in the carbonate, the fraction is present as a replacement for Ca in calcite (isomorphous substitution). On the other hand, the Mg content in the insoluble fraction could mainly be related to the clay minerals since it is one of the major constituents of this fraction (Raza, 2009). MgO shows a strong negative correlation with CaO (Fig. 5).
3. Iron Oxide (Fe₂O₃): The range of iron oxide is between (0.65 and 3.01%) with an average of 1.83% (Table 3). The general role for variation of (Fe₂O₃) is the mobilization and fixation of iron during oxidation. The released iron is as oxides/hydroxides and Mg and Al, Ti, and K (Pendias, 1992) replace it. Fe₂O₃ showed a positive correlation with Al₂O₃ (Table 3).
4. Manganese Oxide (MnO): The range of (MnO) content is between (0.004 and 0.008 %) with an average of 0.006 % (Table 3). There is a strong negative correlation of MnO with CaO, and a strong positive correlation with MgO due to the substitution of Ca and Mg by Mn in carbonate minerals. MnO has a positive correlation with Fe₂O₃ because the manganese and iron are combined in an oxidizing condition (Falkum and Grundving, 2006).
5. Silica (SiO₂): it ranges between (11.32 and 30.39%) with an average of 20.86% (Table 3). According to X-ray analysis, the high SiO₂ in some samples could be related to the terrigenous influx as well as the silicification process. Limestone shows that SiO₂ concentrations are strongly negatively correlated with CaO due to a gradual decrease in quartz and subsequent enrichment of calcite. On the other hand, silica is positively correlated with Al₂O₃ and Na₂O (Fig. 5), which are the main constituents of clay minerals and quartz (Velde, 2010). The presence of quartz is in a negligible quantity. There are two origins of SiO₂ in the formation, according to some samples: clay minerals are the first source, and the siliceous skeleton is the second detritus source. The fact that SiO₂ and Al₂O₃ have such a good positive association suggests they are the main components of clay. The high SiO₂ levels in certain samples are likely related to silicification.
6. Alumina (Al₂O₃): Alumina has a range between (0.32 and 2.32%) with an average of 1.32% (Table 3). It shows a negative relationship with CaO (Fig. 5). The concentration is relatively low, which may be due to the presence of shale. The content of aluminum in carbonate rocks is mainly controlled by the amount of the non-carbonate constituents. The variation in (Al₂O₃) contents is related to the variation in the insoluble residue contents. It is logical to state that all the (Al₂O₃) content is related to clay minerals and the very few other insoluble silicate minerals (e.g. Al-Khafaji and Al-Saad, 2019).

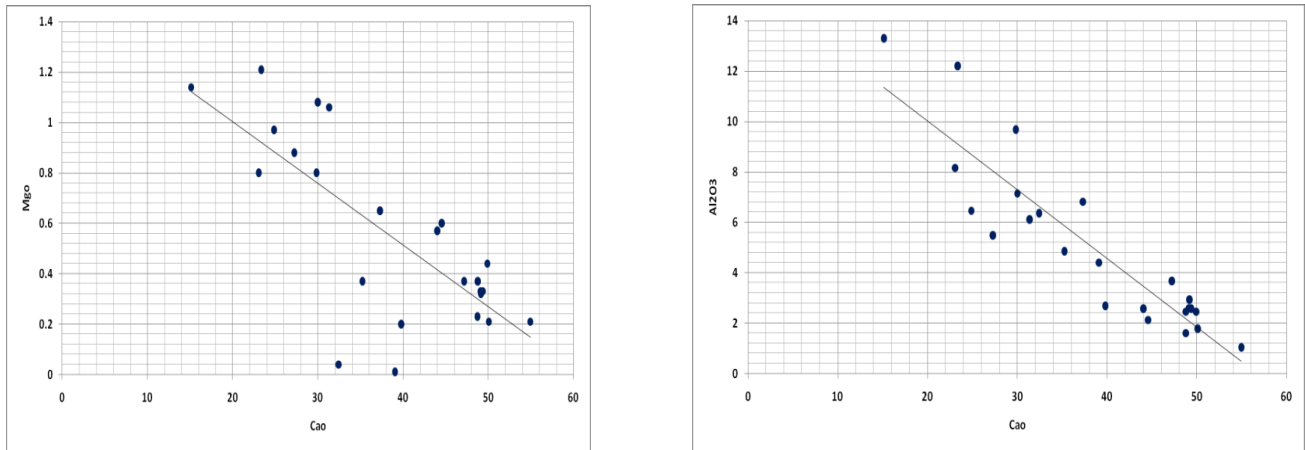


Fig. 5: The relationship diagrams CaO with, MgO and Al₂O₃.

7. Sodium Oxide (Na₂O): The range of (Na₂O) is between (0.22% and 0.55 %) with an average of 0.38% (Table 3). The strong positive correlations between Na₂O, and CaO, SO₃ may be due to the high carbonate content, while the poor negative correlations with K₂O and Al₂O₃ are due to the clay minerals associated with carbonate in the rock.
8. Potassium Oxide (K₂O): The range of (K₂O) is between (0.32% and 1.02 %) with an average of 0.82 % (Table 3). The low content of (K₂O) in carbonate rocks may be attributed to the low content of clay minerals associated with carbonate of Qulqula formation.
9. Titanium Oxide (TiO₂): The range of (TiO₂) is between (0.11% and 0.19 %) with an average of 0.15 % (Table 3). The concentration of (TiO₂) in the samples is generally low. TiO₂ shows a strong negative correlation with CaO and TiO₂ with a strong negative association with CaO, suggesting that these associations provide strong support for the limestone's terrigenous origin (Nagendra et al., 2011).
10. Sulfur Oxide (SO₃): The range of (SO₃) is between (0.02 and 0.63 %) with an average of 0.315 % (Table 3). The low concentration of (SO₃) in all samples indicates that sulfur content in carbonate rocks has no phase mineral (Al-Khafaji and Al-Saad, 2019).
11. L.O.I.: The loss on ignition has a range between (22% and 33 %) with an average of 27.5% (Table 3). Since the samples contain 70% calcite, the burning loss is mostly carbon dioxide, and crystallization water does not account for a large portion due to the low concentration of clay minerals. The abundance of organic matter, as well as the carbonate component of the sediment, led to a rise in the percentage of loss on ignition (L.O.I), which is largely due to carbon dioxide (CO₂) produced from the carbonate fraction of the sediment. (Raza, 2009).

Table 3: Chemical analysis of major oxides (wt %) of Qulqula Formation (20 samples).

Sample no.	CaO	MgO	Na ₂ O	K ₂ O	Mno	TiO ₂	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	SO ₃	L.O.I	TOT
A1	46.99	0.28	0.32	0.55	0.005	0.17	1.02	22.32	0.76	0.02	32	104.4
A2	44.31	0.33	0.34	0.44	0.006	0.16	1.33	25.44	1.24	0.05	28	101.6
A3	39.77	0.27	0.22	0.73	0.004	0.19	1.22	28.34	1.45	0.21	28	100.4
B.	44.78	0.36	0.22	1.02	0.008	0.19	0.32	30.39	2.04	0.19	30	109.5
B1	42.64	1.03	0.31	0.73	0.004	0.17	0.39	28.33	1.76	0.32	31	104.6
B2	44.67	1.22	0.44	0.54	0.008	0.14	0.34	29.33	1.54	0.27	33	111.4
B3	50.11	1.33	0.42	0.54	0.006	0.15	0.4	30.11	1.08	0.44	30	114.4
C1	28.45	0.45	0.28	1.05	0.008	0.17	0.44	32	0.88	0.23	27	90.9
C2.	44.87	0.27	0.55	1.02	0.007	0.16	0.32	17.44	1.32	0.63	29	95.5
C3	47.33	0.62	0.48	1.02	0.004	0.17	0.54	18.44	1.32	0.55	28	98.4
C4	32.9	0.27	0.41	0.88	0.008	0.19	0.44	33.4	1.04	0.45	30	99.9
D.	47.44	0.32	0.49	0.87	0.004	0.17	1.39	20.22	1.77	0.04	33	105.7
D2	51.22	0.34	0.41	1.04	0.006	0.18	1.66	18.55	2.11	0.02	32	107.5
D3	33.04	1.03	0.38	0.22	0.004	0.14	1.54	22.6	2.62	0.04	28	89.6
E.	44.56	0.51	0.27	0.34	0.008	0.19	2.32	11.32	3.01	0.08	32	94.6
E1	45.4	1.22	0.22	0.55	0.004	0.17	1.66	30.33	2.44	0.03	28	110
E2	42.43	0.33	0.22	0.32	0.004	0.12	1.54	28.89	2.64	0.11	30	106.6
F1	38.7	0.46	0.34	0.32	0.008	0.11	1.22	28.45	0.67	0.23	24	94.5
F2	27.95	0.42	0.32	0.41	0.007	0.12	0.88	28.44	0.68	0.44	28	87.6
F.3	46.33	0.3	0.3	0.32	0.008	0.11	1.05	20.56	0.65	0.06	22	91.6
MAX	51.22	1.33	0.55	1.05	0.008	0.19	2.32	33.4	3.01	0.63	33	126.7
MIN	27.95	0.27	0.22	0.22	0.004	0.11	0.32	11.32	0.65	0.02	22	63
MAEN	39.58	0.58	0.38	0.64	0.006	0.15	1.03	20.58	1.83	0.315	27.5	92.5

Trace elements:

Trace elements are considered as indicators to determine the depositional environment and origin of carbonate rocks. The results of trace element analysis in the studied samples are listed in Table (4).

1. Strontium is available in the studied samples. It ranges between 300 and 1000 ppm with an average of 650 ppm. A comparison between the strontium content of limestones, with some other published data, is shown in Table (5). It shows a positive correlation with CaO. The limited dolomitization can be confirmed by the low concentration of Sr element and a negative correlation with other oxides (Wilson, 1989) (Fig. 6).
2. Chromium concentrations range between < 1 to 200 ppm with an average of 40 ppm (Table (4)). The strong positive correlation between Cr with CaO, MgO, Fe₂O₃, Al₂O₃, TiO₂, Co, and Ni (Fig. 7) displays that it is related to the clay minerals in the rock.
3. Cobalt concentrations range between < 3 and < 30 ppm with an average of 5ppm (Table 4). The mean cobalt in the five studied sections is similar to the average contents given by different workers (Table 5). This element has very few concentrations in the studied samples and no positive relationship has been observed with trace elements and oxides.

4. Nickel concentrations range between (10 and 190 ppm) (Table 4). It is usually found in the clay minerals as adsorption on the clay grain surface. Nickel exhibits opposite cobalt behavior in the studied samples where the relationship is a positive correlation (Fig. 6). Table 5) shows a comparison between the Ni content in all studied sections of limestone and other carbonate rocks of other published data.
5. Vanadium ranges between 80 and 880 ppm with an average of 440 ppm (Table 4), and through the positive relationship with the oxides (CaO, MgO, Fe₂O₃, MnO, SiO₂, Al₂O₃, Na₂O, K₂O, TiO₂, SO₃), it is possible to reveal the origin of the rocks (Pendias, 2011) that vanadium found in the crystal unit of clay minerals as a substitution for Al.
6. Zirconium in the studied samples shows a range between < 3 and 100ppm with an average of 45 ppm (Table 4). Zirconium is almost found as zircon that is a resistant mineral transported from the source rocks. Zirconium in carbonate rocks of the studied formations shows a positive correlation with SiO₂, Al₂O₃ (Fig. 8). The existence of zirconium in high values of some samples indicates the secondary acidic igneous rocks of carbonate rocks (Majid, 2015).

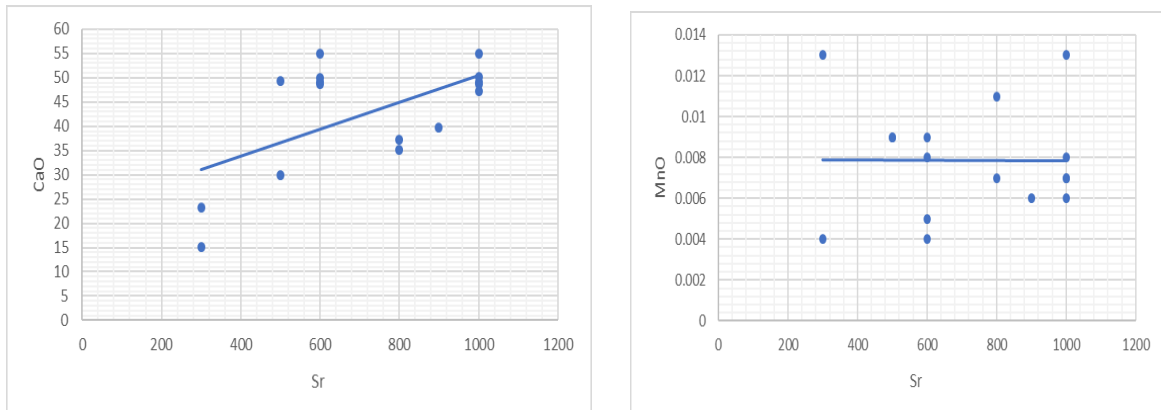


Fig. 6: The relationship Diagrams of Sr with CaO, MnO.

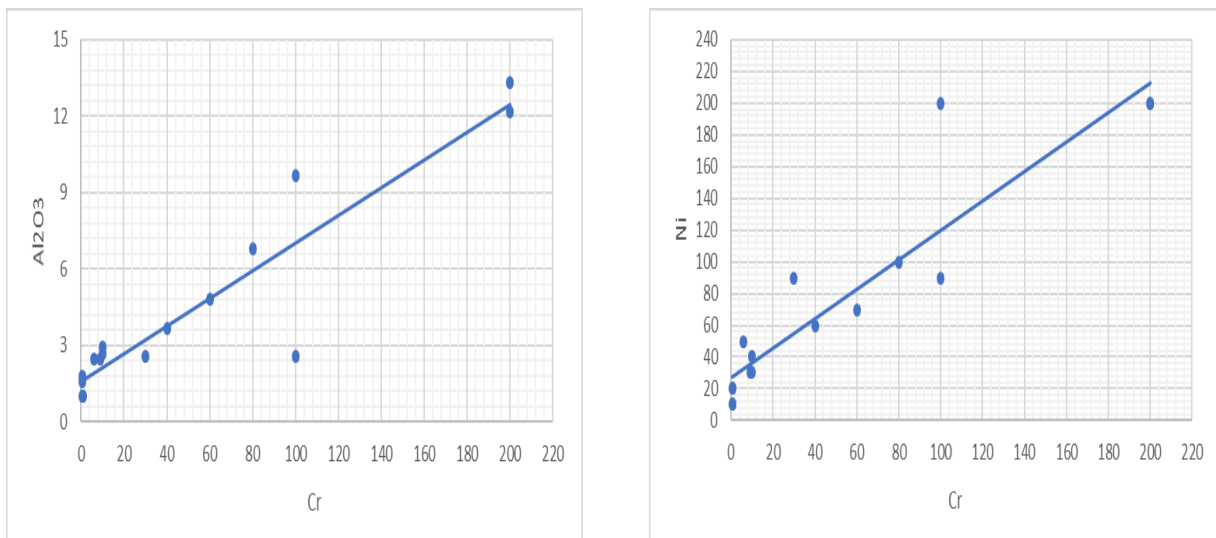


Fig. 7: The relationship diagrams of Cr with Al₂O₃, and Ni.

Table 4: Chemical Analysis of Trace Elements (ppm) of (20 samples) of the Qulqula Formation.

Sample no.	Sr	Cr	Co	Ni	V	Z
A1	500	<3	<3	40	150	30
A2	700	30	<3	10	800	<3
A3	1000	< 1	<3	20	650	50
B.	600	<6	<7	90	400	70
B1	300	100	<3	190	300	60
B2	600	60	<10	70	200	30
B3	500	80	<3	90	100	<3
C1	700	<7	<6	80	200	70
C2.	500	80	<3	40	400	40
C3	700	40	<10	40	600	100
C4	1000	60	<3	30	80	60
D.	600	10	<3	60	190	40
D2	900	40	<30	10	80	20
D3	500	30	<3	40	700	20
E.	600	10	<3	70	400	60
E1	600	40	<30	20	500	20
E2	300	200	<3	190	300	60
F1	700	<6	<3	60	260	40
F2	1000	< 1	<3	20	80	20
F3	500	60	<3	30	100	80
Min.	300	<1	<3	10	80	<3
Max.	1000	200	<30	190	800	100
Mean.	650	40	<5	65	440	45

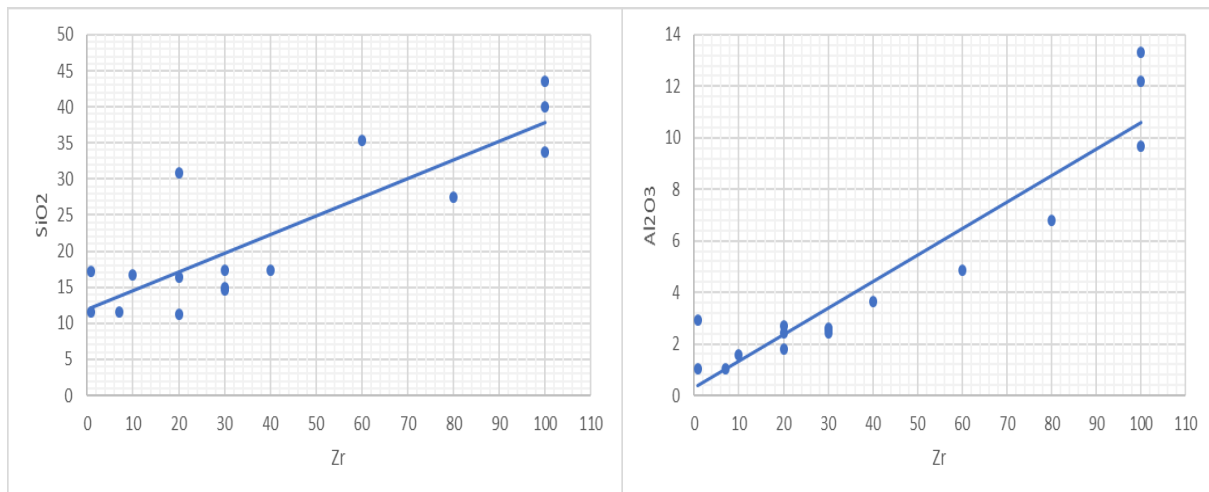
Fig. 8: The relationship diagrams of Zr with SiO₂, Al₂O₃.

Table 5: shows a comparison between the Ni, Co, and Sr content of limestone beds of the studied sections, with some other published data.

Standard	Type of rock or sed.	Ni	Co	Sr
		Argill Lst	60	25
Sarkan	Black Lst	22.14	22.71	360
Awera	Black Lst	30.11	21.56	552
Grian2	Black Lst	28.33	30.11	610
Hawa	Black Lst	22,14	33.45	440
Rankama and Sahama,1950	Carbonate rocks			490
Ostrom,1957	limestone	17		595
Al-Hashimi, 1972	limestone			618
Al-Jawadi, 1978	PilaSpi Limestone			341
Younis, 1979	Miocene Carbonate	13.58		868
Al-Haza'a,1996	Limestone Zahra Fm.	91	84	275

CONCLUSIONS

- 1- The dominant minerals of the studied sections are calcite with an average concentration of 70.137 % and quarts 28.071%.
- 2- The chemical result of the major oxides shows a high concentration in CaO and a relatively high concentration of LOI that reflects the existence of limestone commonly in the overall studied samples.
- 3- The low content of (Na₂O) in studied samples indicates recrystallization of the expected limestone.
- 4- The concentration of alumina Al₂ O₃ is relatively high which may refer to the presence of clay minerals.
- 5- The low concentration of (SO₃) indicates that sulfur content has no phase mineral, so there is no effect on forming new minerals other than expecting new rocks.
- 6- The limitation of dolomitization can be confirmed by the low concentration of Sr element.
- 7- The high alteration of limestone to marly limestone and marl, and then to marl like shale, was discovered by the strong negative association between CaO and other oxides MgO, Fe₂O₃, MnO, SiO₂, Al₂O₃, K₂O, and TiO₂.

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