

MINERALOGY AND GEOCHEMISTRY OF CARBONATE ROCK FOR HARTHA RESERVOIR FORMATION IN BALAD OIL FIELD/ CENTRAL IRAQ

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Abstract

Mineralogical and Geochemical investigations were conducted on three wells of the Balad oilfields (Balad-1, Balad-4, Balad-8) in the Hartha Formation (Late Campanian-Maastrichtian), central Iraq. The main objectives of the research are to determine the mineralogy and geochemistry of the Hartha Formation in the Balad oil field. For this purpose, mineralogy tests were performed using X-ray diffraction (XRD), and geochemistry (major oxides) was determined using X-ray fluorescence (XRF) for the study area samples. The change in the physico-chemical state during the deposition period is responsible for the variation in the CaO content with other oxides. Mineralogical results: calcite is the most common mineral, and dolomite is the second common mineral found in the samples under study, along with quartz and clay minerals (kaolinite and illite). The geochemical composition of the Hartha carbonates contains many of the oxides and a relatively small amount of argillaceous material as a result of the transfer, and the pattern of the relations of the main oxide components suggests they are of biochemical origin and not significantly influenced by the detrital source. Chemical classification of Limestone in the sample area of the Balad oilfield shows that magnesian limestone is the most abundant type of carbonatite rock. The deposited limestone of the Hartha in a near-shore, shallow marine habitat is appropriate for receiving some terrigenous minerals.

Keywords: Carbonate Rock; Geochemistry; Hartha Formation; Major oxides XRD; XRF.

1. Introduction

The Hartha Formation, deposited during the Late Campanian and Maastrichtian, is a major Cretaceous geological hydrocarbon-producing formation with carbonates comprising the main lithotype (Aqrabi et al., 2010), located in central and southern Iraq. Rabanit in 1952 defined the Hartha Formation from a well, Zubair-3, within the Mesopotamian region of south Iraq (Owen & Nasr, 1958). The bottom contact of the Hartha Formation is not conforming, and the Shiranish Formation's pelagic sediments frequently cover the Hartha Formation (Buday, 1980). The Hartha

formation thickness in southern Iraq ranges from 200 m to 250 m, and in Iraq's northern region, the thickness of the Hartha Formation was reported to be up to 350 m. The actual thicknesses of high structures in a Stable Shelf are often less than 100 m (Buday, 1980). Their sequences reveal subsurface characteristics, making them significant reservoir rocks in Iraq's central and southern regions. The marine shallow carbonate of the Hartha Formation in western Iraq is also mentioned as producing oil, as indicated by the Ahdab and East Baghdad oilfields, which show perfect economic potential (Sadooni, 1996). The Hartha Formation was deposited in a fore-reef to shoal habitat, along the margins of the Stable Shelf, with local occurrences of lagoonal backreef facies. The reef limestone debris in the detrital limestone strata in the formation in some wells suggests the presence of reef facies surrounding the Khlesia high (Jassim & Goff, 2006). The study of carbonate units is of great importance due to the presence of porous dolomites, which directly reflect reservoir quality. These cycle successions are important producers of hydrocarbons in formations with carbonates comprising the main lithotype (Aqrawi et al., 2010). The replacement of calcite by dolomite in carbonate rocks results in the development of secondary porosity, primarily because dolomite crystals are approximately 20% smaller in size than calcite crystals. These kinds of studies were occasionally conducted by various researchers (Singh & Anand, 1991; Das et al., 2004; Das & Das, 2010). The location of the Balad oil field study area is situated in Salah Al-Deen governorate, roughly 9 kilometers from Balad city, and 60-70 kilometers north of the Baghdad governorate (Figure 1). This study includes three boreholes in the Balad oil field (Ba-1, Ba-4, Ba-8). This paper aims to determine of mineralogy, chemical composition of the Hartha formation.

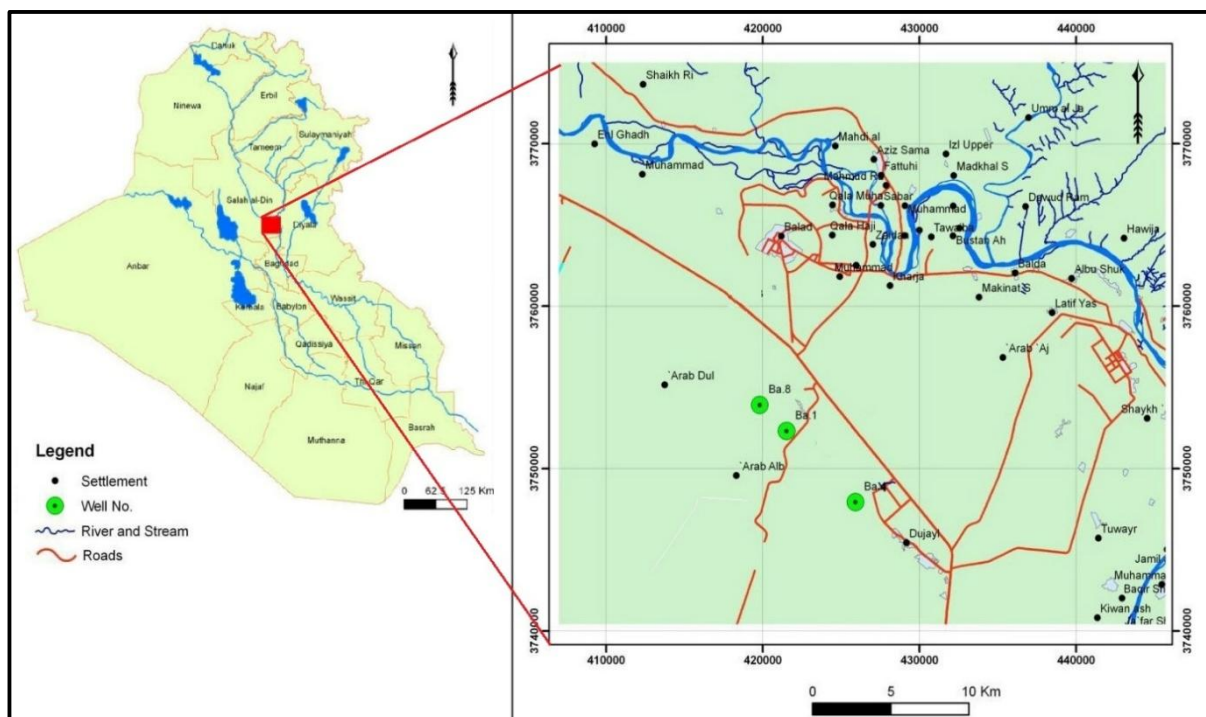


Figure 1. Location of the study area.

2. Geology of the study area

The cycle of Late Campanian-Maastrichtian is a huge structure in the evolution of Iraq's geology. This cycle started with the widespread violations that covered the entire country and ended (Buday, 1980). The start of this extensive transgression cycle in the inner Suture Zone is credited overall movement of sedimentary basins towards the Neo-Tethys (Buday, 1980). Neo-Tethyan ophiolite was raised above sea level, shoved further into the northern Arabian Plate, and eroded at high rates throughout the Late Campanian to Early Maastrichtian period. The extensional basins experienced significant subsidence. These areas of subsidence include the Euphrates, the Azraq-Sirhan Grabens, and the Anah and Tayarat Grabens, which stretch to the E-W direction. The rifting stage was when these basins first formed in the early Late Cretaceous Period (Jassim & Goff, 2006). The Campanian to Maastrichtian section comprises the Hartha Formation and the Shiranish Formation in southern and central Iraq. This sedimentary cycle began with a major marine transgression and ended with tectonic uplift and marine regression, where the beginning of the continental collision to the east and folding of the Zagros Mountains caused a downwarp of the Balad area (Sharland et al., 2001). Deposited the Hartha Formation in a carbonate inner shelf and lagoonal back reef setting surrounding the shelf margins during the Late Campanian-Early Maastrichtian era, in the Mesopotamian Zone (Jassim & Goff, 2006). The successions within this cycle represent significant hydrocarbon-producing formations, primarily composed of carbonate lithotypes (Aqrawi et al., 2010). Their sequences exhibit subsurface properties, lithofacies, and petrophysical characteristics, which make them essential reservoir rocks in the central and southern parts of Iraq (Sadooni, 1996). The Hartha Reservoir's observed and computed porosity, when combined with the measured permeability, provided the formation with good reservoir quality (Nooralddin et al., 2022). The Hartha Formation sequence includes the deposition in central and southern Iraq, which occurred due to a significant rise in sea levels (transgression). The sequence concluded with a stage of tectonic uplifting and a subsequent fall in sea levels (Ahmed & Al-Zaidy, 2023; Aziz et al., 2024) studied three oil fields in central Iraq within the Mesopotamian Zone, East Baghdad. Al-Sammarai (2010) split the Balad oil field's Hartha Formation into two primary paleoenvironments: a lagoon environment and a confined marine shelf in central Iraq. The Hartha Formation is one of the most significant carbonate successions in the Late Cretaceous (Jassim & Goff, 2006). Al-Naemi (2012) concluded that the Balad oil field has seen two main faults and many smaller ones, all of which have a substantial effect on the Hartha Formation sedimentary basins. According to well data collected in the field and the stratigraphic features in the seismic sections, listric growth normal faults with branches were the sort of normal faults that impacted the research region (Muhsin & Al-Ridha, 2013). Sadooni (1996) separated the Central Iraqi Hartha Formation into five microfacies: 1) Rudist rudstone and grainstones. 2) Echinoid packstone – grainstone. 3) Foraminiferal packstone–grainstone. 4) Foraminiferal wackstone. 5) Pelletal – peloidal packstone – grainstone. The Hartha Formation's extensive open marine deposits and lack of reef-building facies provide proof that it was produced on a carbonate ramp (Ismail et al., 2022).

3. Materials and methods

The methodology of this work is represented by selecting three wells of the Balad oil field, including Ba-1, Ba-4, and Ba-8 (Figure 2), to determine mineralogy and geochemical

investigations. The core samples were taken from the Oil Exploration Company (OEC), which belongs to the Hartha Formation. Ten samples were examined using X-ray diffraction (XRD) to determine the primary and secondary minerals of the Hartha formation Table 1. For samples, the scanning angles were between 20 and 600, as the most fundamental reflections of primary and secondary minerals typically fall within this range. This analysis was conducted in the Laboratory of the Ministry of Science and Technology in Baghdad. Resulting from this analysis, two types of minerals (Carbonate & clay) are shown in Figure 3 and Table 1. Geochemical analysis was carried out for twenty samples to determine their chemical composition (elements), using X-ray fluorescence (XRF) of the Hartha formation. The studied samples were pulverized to a size of less than 75 microns. Analysis done in the Iraqi-German Laboratory, Department of Geology, College of Science at the University of Baghdad. The analysis resulted from the main oxides (MgO , CaO , Fe_2O_3 , TiO_2 , SiO_2 , Na_2O , P_2O_5 , SO_3 , K_2O , and Al_2O_3) and loss on ignition. The average of this major oxide was compared with the global standard of carbonate rocks (GSCR). Also, we found the results of Loss on Ignition shown in (Appendix A).

The nature of the relationships between major oxides was studied through a diagram of Pearson's correlation and diagrams that illustrate the correlation between them Figure 4. Then, identifying the dominant elements (Ca, Mg) generated by geochemical analysis and calculating their ratio (Appendix A) to determine the limestone classification of Hartha's composition, based on the classification of limestone by Todd (1966), as shown in Table 2.

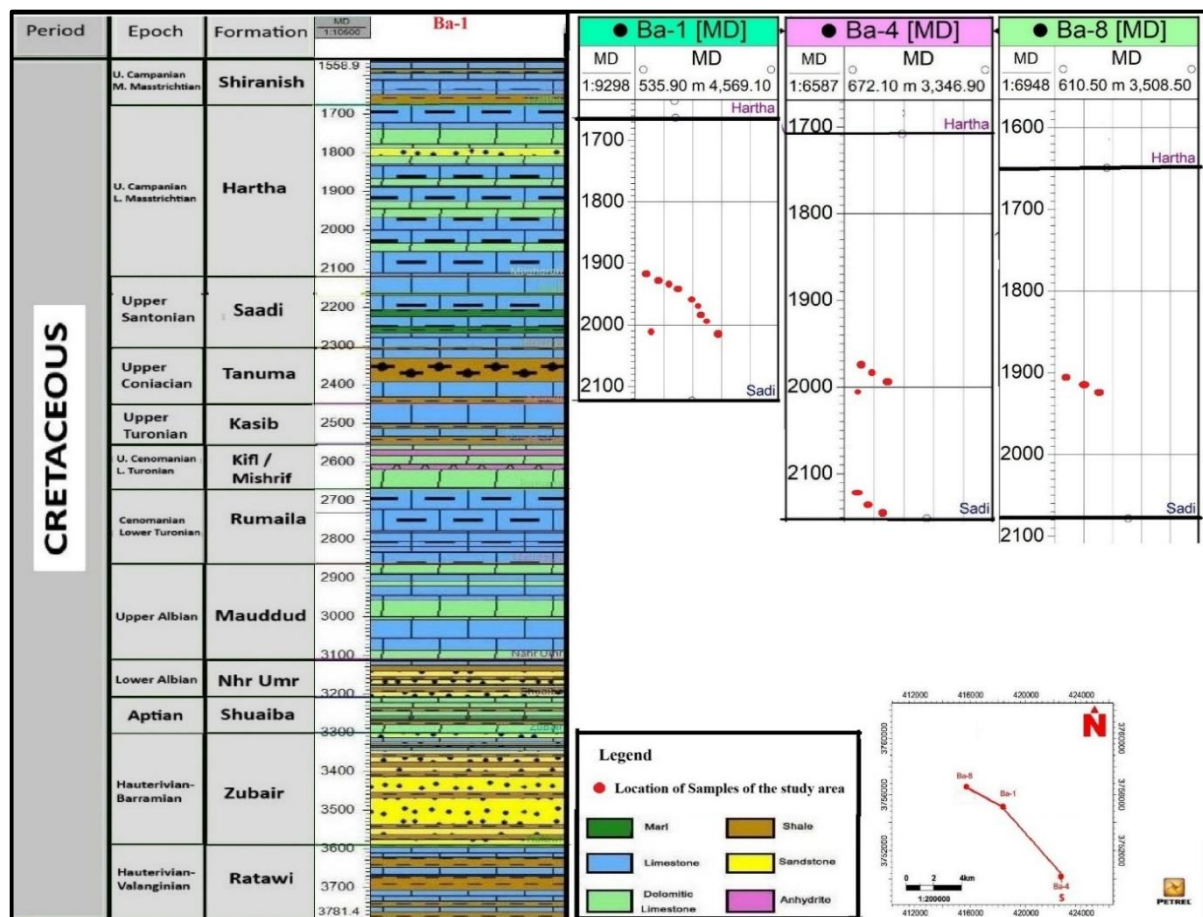
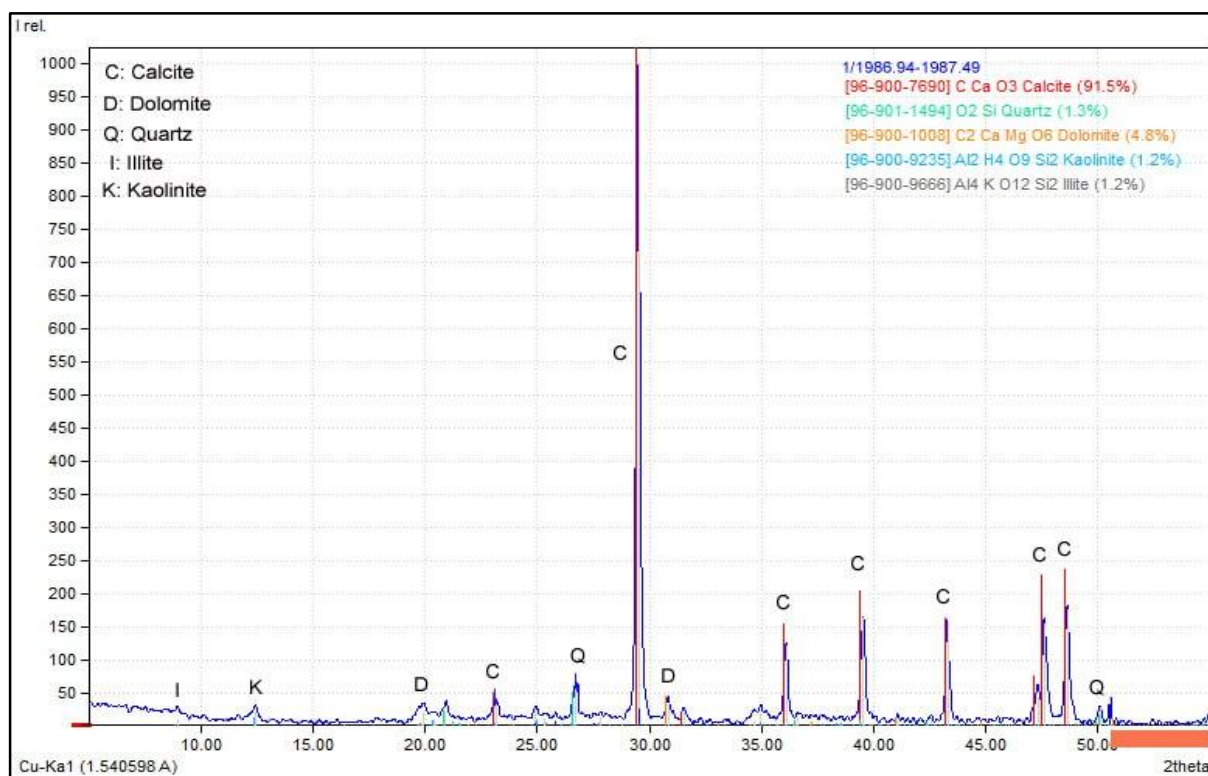


Figure 2. Lithology of Balad-1 and the locations of samples in Ba-1, Ba-4, and Ba-8.

Table 1. Mineralogical composition of the Hartha Formation.

Well No.	Depth	Non-clay minerals %			Clay minerals %	
		Calcite	Dolomite	Quartz	Kaolinite	Illite
1	1917	93.8	4.7	1.5		
	1925	95.7	2	2.4		
	1940	93.5	5.3	1.2	1.2	
	1986	91.5	4.8	1.3	1.2	1.2
	2017	90.3	4.6	3.1	2	
4	1973	90.8	7.3	1.9		
	1995	96.2	3.8			
	2143	100				
8	1908	97	3			
	1923	92.2	4.1	2.1	1.6	
Minimum		90.3	2	1.2	1.2	
Maximum		100	7.3	3.1	2	
Average		94.1	4.4	1.9	1.5	1.2

**Figure 3.** X-ray diffraction analysis in Balad-1, at a sample depth of 1986m.

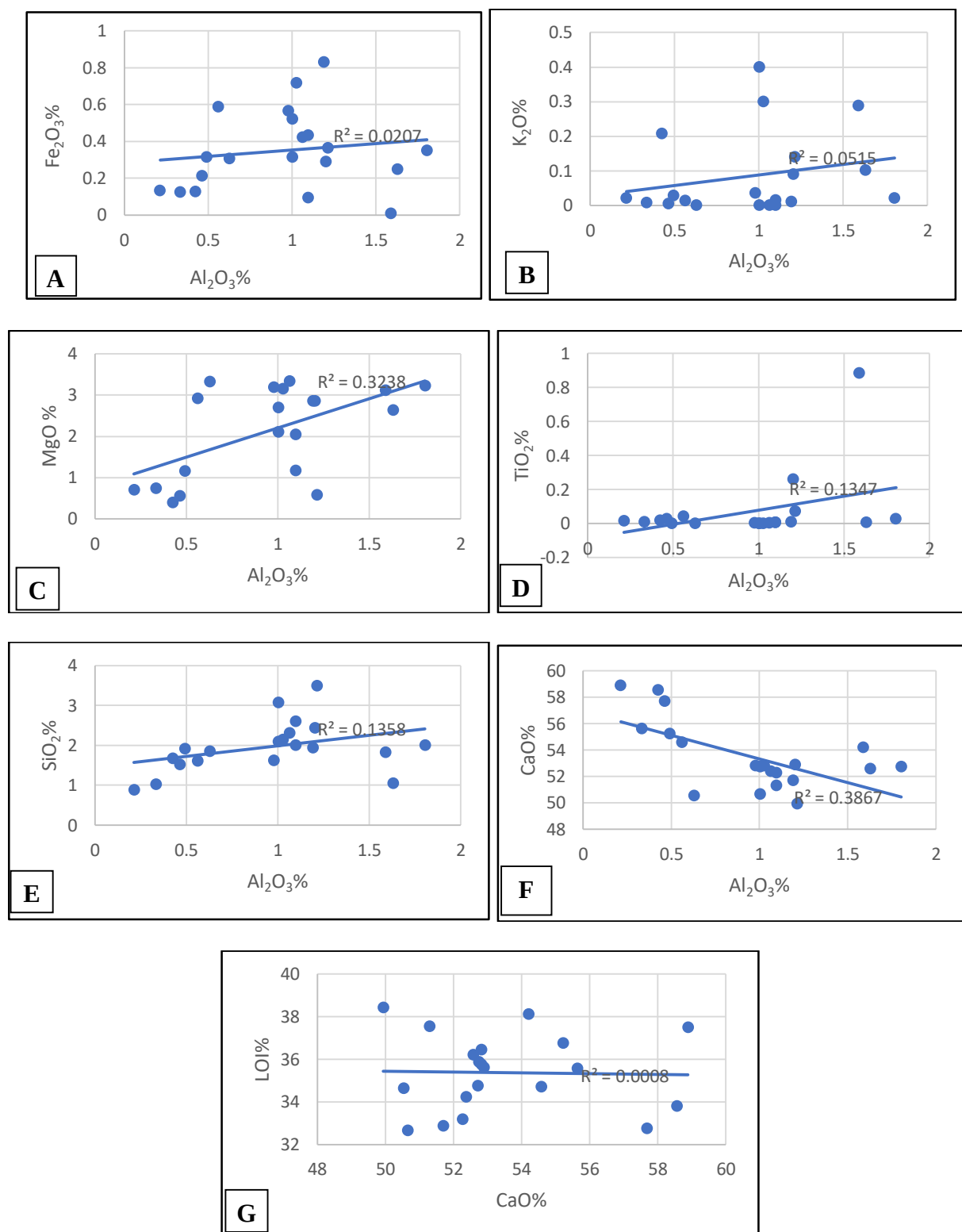


Figure 4. Correlations between major oxides of the Hartha formation in Balad Oilfields, **A)** Relationship between Al_2O_3 & Fe_2O_3 ; **B)** Relationship between Al_2O_3 & K_2O ; **C)** Relationship between Al_2O_3 & MgO ; **D)** Relationship between Al_2O_3 & TiO_2 ; **E)** Relationship between Al_2O_3 & SiO_2 ; **F)** Relationship between Al_2O_3 & CaO ; **G)** Relationship between CaO & LOI .

Table 2. Chemical classification of Limestone (after Todd,1966).

Descriptive Term	Standard Ratio Ca/Mg	Reciprocal Mg/Ca
Pure Limestone	100.00 -39.00	0.00-0.03
Magnesian Limestone	39.00 -12.30	0.03-0.08
Dolomitic Limestone	12.30 -1.41	0.08-0.18

4. Results

4.1. Mineralogy

Mineral analysis of the Hartha formation by X-ray Diffraction (XRD) device is a successful method for determining the minerals present in samples. The X-ray diffractograms of the investigated samples showed that calcite is the main and predominant mineral. With dolomite, quartz, and a trace amount of clay minerals (kaolinite and illite), as shown in Table 1 and Figure 3.

4.1.1. Carbonate Minerals (Non-clay)

Calcite is the major mineral of the Hartha Formation, and most of the samples consist mainly of calcite in a percentage range from 90.3% - 100%, with an average of 94.1%. Dolomite is one of the most important carbonate minerals. Samples of the Hartha formation showed that dolomite is the second mineral, with a percentage range from 2 – 7.3 % and an average of 4.4%. Quartz is minor in the studied rocks, of detrital origins, coexist in many samples; the percentage of quartz ranges from 1.2% – 3.1% with an average of 1.9%. All percentages of these minerals are shown in Table 1.

4.1.2. Clay minerals

Clay minerals are hydrated aluminum silicates. It is found as transported detrital materials by supplying the sedimentation basins from the continents with weathering agents, rivers, or wind (Sudo & Shimoda, 1978). The study area contains small amounts of clay minerals. This could be due to Argillaceous limestone is more common in the Mesopotamian Zone's

Eastern regions (Jassim & Goff, 2006). Kaolinite ranged from 1.2% to 2.0% with an average of 1.5%, and Illite appeared only in the sample depth of 1986, at about 1.2% are shown in Table 1.

4.2. Geochemistry

Geochemical data of the Hartha formation depend on result of X-ray Fluorescence (XRF) analysis of 20 samples for major oxide (CaO, MgO, SiO₂, Na₂O, K₂O, TiO₂, P₂O₅, SO₃, Fe₂O₃, and Al₂O₃) and loss on ignition percentage of carbonate samples distributed in three well of the study area was done and compared to global carbonate rocks, (GSCR) Global Standard of

Carbonate Rocks by (Turekian & Wedepohl, 1961) and local carbonate studies (Ali & Sa'ad, 2018, Taha Taher M., 2019, Mahmood, 2022), Appendix A.

4.2.1. Major oxides

The major oxide (wt %) analyses of twenty carbonate samples were studied contents of CaO ranged between (49.94) % to (58.89) % with an average of (53.51), MgO ranged between (0.397) % to (3.33) % with an average of (2.135)%, Al_2O_3 ranged between (0.213) % to (1.804) % with an average of (0.951)%, SiO_2 ranged between (0.887) % to (3.505) % with an average of (1.961)%, P_2O_5 ranged between (0.003) % to (0.036) % with an average of (0.015)%, SO_3 ranged between (0.025) % to (1.676) % with an average of (0.647)%, K_2O ranged between (0.001) % to (0.401) % with an average of (0.085)%, TiO_2 ranged between (0.000) % to (0.883) % with an average of (0.070)%, Na_2O ranged between (0.966) % to (1.988) % with an average of (1.484)% and Fe_2O_3 ranged between (0.01) % to (0.831) % with an average of (0.350)%, (Appendix A). The average results of major oxides & LOI in the study area were compared with the global standard of carbonate (GSCR) rocks as follows: the average of CaO, Na_2O , and LOI in the studied sample is higher than the GSCR due to the high purity of carbonate rocks. The averages of MgO, SiO_2 , Al_2O_3 , Fe_2O_3 , and K_2O in the studied sample are less than the GSCR. (Appendix A).

4.2.2. Ratio Ca/Mg & Mg/Ca

Todd (1966) used the Ca/Mg distribution and the inverse Mg/Ca in limestones as a criterion for Chemical classifications (Table 2). A classification of the research area, Limestones, is suggested by Todd (1966). (Appendix A) shows that the Mg/Ca ratio in the present study varies from 0.01022 to 0.0989 %, with a mean of 0.060 %, and Ca/Mg ratios vary from 10.106 to 97.829 %, with an average of 27.59%. According to Todd's (1966) classification, the studied samples are located within the field of Magnesian Limestone.

5. Discussion

The main types of minerals are calcite and dolomite, with quartz and a trace amount of clay minerals (kaolinite and illite) (Figure 3 and Table 1). Geochemical analyses of the Hartha carbonite rock include oxides (Na_2O , CaO, Al_2O_3 , Fe_2O_3 , SiO_2 , MgO, and K_2O) and LOI loss on ignition (Appendix A), and compared with GCSR and some of the local studies, where all the results agreed with a high concentration of CaO. The Ca/Mg ratio distribution in the limestone (Appendix A) region indicates that the limestone samples belong to the Magnesian limestone type (Table 2). The high Ca/Mg ratio concentration suggests that there was relatively less seawater evaporation during the limestone deposition period. Far from the beach, the Mg/Ca ratio of carbonate sediments rises, which is associated with the quantity of Mg-rich coralline algae in near-shore water. In this instance, the data points to deposition close to the coastline (Marshner, 1968). Pearson's correlation coefficient was used between major oxides illustrated in Table 3. CaO shows a negative correlation with MgO%, SiO_2 % and Al_2O_3 % indicating the predominance of biochemical origin (Table 3). While CaO and LOI show a negative correlation, which indicates that the content of these oxides is not affected by a distinctive paleoenvironment, this supports skeleton formation (Von Eynatten et al., 2003). Oxides (Fe_2O_3 , K_2O , MgO, TiO_2 , and SiO_2) are

plotted versus Al_2O_3 (Figure 4a, b, c, d, and e), and all are linked with weak positive correlations. Weak negative correlation can be observed between CaO and Al_2O_3 (Figure 4f), which indicates that almost all calcium comes from marine sources and is not greatly impacted by detrital sources (Hofer et al., 2011). The weak linear relationship between CaO and LOI (Figure 4g) demonstrates that carbonates are the primary source of CaO (Von Eynatten et al., 2003). The CaO concentration plotted against SiO_2 , Fe_2O_3 , MgO, and Al_2O_3 shows a negative relationship. Therefore, this suggests that once $\text{SiO}_2/\text{MgO}/\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ increases, the CaO portion drops (Hofer et al., 2011). Bivariant charts of Al_2O_3 against CaO shows negative relationship and, while positive correlation of Al_2O_3 against MgO, this indicates that, in comparison to pure limestone, the amount of calcium decline in limestone when it is rich in magnesium, shows that magnesium is enhanced when the CaO is eliminated through leaching solution process, resulting in the formation of magnesium limestone (Von Eynatten et al., 2003).

Table 3. Pearson's correlation coefficient between major oxides and L. O. I.

Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	L.O. I
Na ₂ O	1									
MgO	0.33	1								
Al ₂ O ₃	0.15	0.57	1							
SiO ₂	-0.05	0.1	0.37	1						
P ₂ O ₅	0.12	-0.14	-0	-0.09	1					
SO ₃	-0.19	-0.06	-0.49	-0.21	-0.19	1				
K ₂ O	-0.24	0.04	0.23	0.09	0.13	-0.11	1			
CaO	-0.3	-0.54	-0.62	-0.68	0.2	0.07	0.04	1		
TiO ₂	-0.13	0.21	0.37	0.03	0.02	-0.33	0.39	0.05	1	
Fe ₂ O ₃	0.02	0.49	0.14	0.18	-0.11	0.08	0.07	-0.39	-0.39	1
L.O. I.	0.05	-0.14	0.2	0.04	-0.25	-0.19	0.27	-0.03	0.38	-0.34 1

6. Conclusions

The mineralogical investigation of the Hartha Formation in selected wells shows that Calcite is the main kind of mineral, and dolomite, quartz, and clay minerals (kaolinite and illite) as secondary constituents. Moreover, the carbonate samples include relatively little argillaceous material, as well as the correlations between the main oxide components imply that they are mostly of biochemical origin. It also appears during the study that Magnesian limestone is the most common rock type among the samples under study, according to the geochemical categorization of carbonate rocks based on Ca/Mg ratios. Statistical analysis revealed weak relationships between major oxides: SiO_2 , MgO, Fe_2O_3 , K_2O , TiO_2 , CaO, and Al_2O_3 , as well as

weak relationships between CaO and LOI. This suggests that the content of these oxides is not influenced by a particular paleoenvironment and that the CaO is mostly of marine origin and not significantly influenced by the detrital source. In Bivariant charts, the negative relationship between Al_2O_3 and CaO, alongside the positive correlation between Al_2O_3 and MgO, indicates that calcium decreases in magnesium-rich limestone relative to pure limestone. This suggests that magnesium becomes enhanced when CaO is eliminated through leaching during the solution procedure, forming magnesium limestone.

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Author Contributions

The authors contributed to the study's conception and design. Material preparation, data collection, and analysis were performed by Farah M. Jaddoa and Enaam J. Abdullah. The first draft of the manuscript was written by Farah M. Jaddoa. The authors read and approved the final manuscript.

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