Iraqi National Journal of Earth Science
www.earth.mosuljournals.com

Assessment of Limestone of Jeribe Formation for Ordinary Portland Cement Industry in Bekhair Anticline, Duhok Governorate, Iraqi Kurdistan Region

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Article information

Received: 14- Aug -2022

Accepted: 05- Oct -2022

Available online: 31- Dec- 2022

Keywords:
Jeribe Limestone
Ordinary Portland Cement
Bekhair anticline
Duhok Governorate
Kurdistan region

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ABSTRACT

The limestone of Jeribe Formation in Bekhair anticline, Duhok Governorate, was assessed as raw materials for the Ordinary Portland Cement (OPC) industry. Four sections were selected; the formation consists of fossiliferous limestone and thin layers of dolomitic limestone which are less than one-meter-thick and did not affect the average percentage of MgO in the mixture. The petrographic study showed that the main mineral of limestone rocks is fine calcite with a size of less than 0.25 mm. The physical and mechanical tests of samples indicate low porosity, and the compressive strength range between (168-1291 kg/cm²), which are acceptable for the cement industry and make the rocks easy to crush and grind during the preparation of the mixture. The chemical analysis revealed that the mean concentration of CaO is 51.77% which dominates over other oxides, including MgO (2.06%), SiO_2 (1.95%), Al_2O_3 (0.60%), and, Fe_2O_3 (0.22%), While the remaining oxides (K_2O, Na_2O, TiO_2, MnO, P_2O_5, and SO_3) are considered trace oxides. The lime saturation factors (LSF) of the studied sections range from (1101.74 to 2334.89), which are over the limits necessary for high-quality cement; thus, claystone from Fatha Formation was added to achieve the best point of clinker LSF. The clinker phases C3S, C2S, C3A, and C4AF in the studied sections (Zt, Q, K, and T) range between (49.19-56.70), (17.88-23.11), (10.87-11.30), and (9.20-9.42) % respectively, and they are within the standard acceptable range. According to the results of the analyses, we conclude that the best mixture of limestone ranges between (65.9-69.3) % and claystone ranges between (30.7-34.1) % in the study area are of good quality and meet the requirements of international standards for ordinary Portland cement.

DOI: 10.33899/earth.2022.135129.1022, ©Authors, 2022, College of Science, University of Mosul.

تقييم الصخور الجيرية لتكوين الجريبي لصناعة الاسمنت البورتلاندي الاعتيادي في طية بيخير، محافظة دهوك، اقليم كوردستان العراق

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معلومات الارشفة

تاريخ الاستلام: 14- أغسطس -2022

تاريخ القبـول: 05-أكتوبر-2022

تاريخ النشر الالكتروني: 31-ديسمبر-2022

الكلمات المفتاحية:

ال تكوين الجريبي

الاسمنت البورتلاندي الاعتيادي

طية بيخير

محافظة دهوك

اقليم كوردستان

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الملخص

تم تقييم الحجر الجيري لتكوين الجريبي في طيه بيخير بمحافظة دهوك كمواد خام لصناعة الأسمنت البورتلاندي الاعتيادي. تم اختيار أربعة مقاطع من التكوين، الذي يتكون من الحجر الجيري الغني بالمتحجرات وطبقات رقيقة من الحجر الجيري الدولوميتي بسمك اقل من متر واحد ولا تؤثر على معدل نسبة MgO في الخلطة. أظهرت الدراسة البتروغرافية أن المعدن الرئيسي لصخور الحجر الجيري هو الكالسيت الناعم بحجم أقل من 0.25 ملم. تشير اختبارات الفيزيائية والميكانيكية للعينات إلى انخفاض المسامية، وتتراوح المقاومة الانضغاطية بين (168-1291 كجم/ سم٢)، وهي مقبولة لصناعة الأسمنت وتجعل الصخور سهلة التكسير والطحن أثناء تحضير الخليط. أظهرت نتائج التحليل الكيميائي أن متوسط تركيز CaO هو 51.77٪ الذي يسيطر على الأكاسيد الأخرى، بما في ذلك MgO (2.06٪)، SiO_2 (1.95٪)، Al_2O_3 (0.60٪)، و(Fe_2O_3 (0.22٪)، بينما الأكاسيد المتبقية (K_2O وNa_2O وTiO_2 وMnO وP_2O_5 وSO_3) بتراكيز ضئيلة. ويتراوح معامل الاشباع الكلسي (LSF) للمقاطع المدروسة من (1651.70 إلى 2334.89) وهي تتجاوز الحدود اللازمة للأسمنت عالي الجودة. ولذلك تمت إضافة الحجر الطيني من تكوين الفتحة لتحقيق أفضل مستوى من معامل الاشباع الكلسي LSF للكلنكر. تتراوح نسبة اطوار الكلنكر C3S و C2S و C3A و C4AF في المقاطع المدروسة (Zt، Q، K و T) بين (49.19 - 56.70) ٪ و (17.88 - 23.11) و (10.87 - 11.30) و (9.20 - 9.42) ٪ على التوالي، وهي ضمن النطاق القياسي المقبول. وبناءً على نتائج التحليلات نستنتج أن أفضل خليط هو من الحجر الجيري الذي يتراوح ما بين (65.9-69.3) % والحجر الطيني الذي يتراوح بين (30.7-34.1) % في منطقة الدراسة ينتج كلنكر عالي الجودة موافق لمتطلبات المواصفات القياسية العالمية للأسمنت البورتلاندي العادي.

DOI: 10.33899/earth.2022.135129.1022, ©Authors, 2022, College of Science, University of Mosul.

Introduction

Cement is a mineral powder that, when mixed with water, makes a plastic body that can be easily molded and hardened over time to produce a strong and hard body (Aragaw, 2018). Cement and concrete are essential infrastructural materials in the modern world (Scrivener, 2014). The cement industry's demand has been steadily increasing in tandem with the demand for construction, which drives urbanization (Lim, et al., 2020).

The raw materials for the cement industry are mainly limestone and claystone, which form the clinker mixture, then gypsum is added as an additive to control the setting time of cement. Portland cement clinker has four main phases: alite (C3S), belite (C2S), aluminate (C3A), and ferrite (C4AF), there may also be minor phases of periclase (MgO), lime (CaO), or alkali sulfate in the Portland cement clinker (Suherman, et al., 2002).

There are several cement factories in the Kurdistan region, most are located in the Sulaymaniyah Governorate. There is no cement factory in the Duhok Governorate and the Zakho district. There is currently urban development and infrastructure construction in them, and vast quantities of cement are needed for this construction work. Most of this cement is imported from Turkey and a small part from Sulaymaniyah Governorate. Limestone, the primary raw material for the cement industry, is present in several geological formations in Duhok Governorate. These formations are situated in border areas and far from urban areas. Therefore, in this study, the western part of the Bekhair anticline was chosen because it is located between Duhok Governorate and Zakho district and is close to urban areas and the Iraqi-Syrian border as well.

Establishing a cement factory in the study area will supply job opportunities for about 500 people. According to the latest statistics from the Kurdistan Region Statistics Office in 2017, the unemployment rate in the Duhok governorate reached 13.8%, which is more than the unemployment rates in the governorates of Erbil and Sulaymaniyah.

Mosul city is about 120 km from the study area, and its infrastructure was destroyed when ISIS took control of it. The city is currently in the process of reconstruction and requires large quantities of cement, so it is expected that the cement be provided to it, after supplying the needs of Duhok Governorate and Zakho district with the required cement. Syria also, the war and the events that took place for more than ten years destroyed the infrastructure of most of its cities and now requires a lot of cement to reconstruct it. Therefore, after providing local needs with cement, it can also be exported to Syria.

There are no previous studies assessing this formation for the cement industry, and most of the previous studies are related to sedimentology, paleontology, stratigraphic, and structure. While in the areas close to the study area, the rocks of other formations were evaluated for the cement industry; from these studies, Yezdeen (1990) evaluated the suitability of some Tertiary rocks from Wadi Khan in the Sinjar region for the production of ordinary Portland cement. The study area is located at Bekhair anticline in the north of Iraq, which is situated to the northwest of Duhok Governorate at a distance of about 60 km, and to the southwest of the Zakho district at about 20 km (Fig. 1).

The aim is to conduct studies on the chemical, petrographic, physical, and mechanical properties of Jeribe Formation limestone in the Bekhair anticline in Duhok Governorate, to evaluate their viability as raw materials for the Ordinary Portland Cement industry.

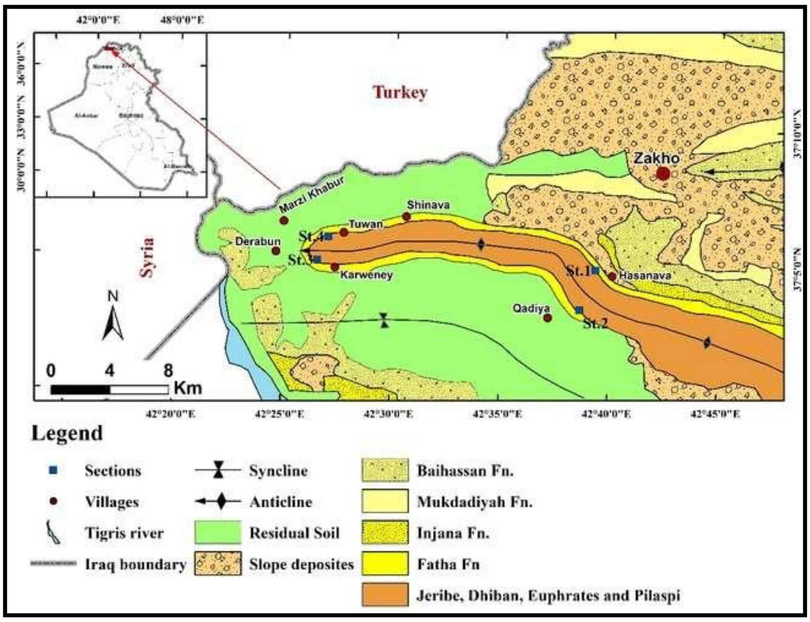

Fig. 1. Geological map and selected sections of the study area (after Al-Mousawi, et al., 2008)

Geology of the study area

Bekhair anticline is an asymmetrical double plunging fold within the Iraqi Foreland High Mountain Belt. It stretches about 72 kilometers from the Zawita/Besari area (the southeastern plunge) to Derabun village near the Iraqi-Syrian-Turkish border (the northwest plunge) (Al-Hubiti and Al-Azzawi, 2009). The following formations are outcroppings in the study area (Fig. 2a).

1- Pila Spi Formation

Lees described the formation for the first time in 1930 from the Pila Spi area on the southeastern margins of the High Folded Zone and Wetzel (1947 in Bellen, et al., 1959) redefined it in Derbendikhan area. According to Taufiq and Domas (1977), the Pila Spi Formation in the Duhok and Bekhair anticline is 143 meters thick. It is the core of the anticline and forms the bulk of the Bekhair anticline due to its hardness (Alawi 1980). The upper part comprises well-bedded, bituminous, chalky, and crystalline limestones, with bands of white chalky marl, with chert nodules. The lower part is composed of dolomitic limestones that are hard, porous, vitreous, whitish, and poorly fossiliferous. A shallow lagoon is a depositional environment of the formation (Asaad, 2022). The Pila Spi Formation is Middle Eocene in age (Sissakian and Youkhanna,1979; Jassim and Buday in Jassim and Goff, 2006).

2-Euphrates Formation

The Euphrates Formation was defined by De Boechkh, et al. (1929) and was amended by Bellen (1957); the type section is in Wadi Fhaimi (western Iraq) (Sissakian and Al-Jiburi, 2014). At the type locality, the formation consists of shelly, chalky, well-bedded recrystallized limestones (Bellen, et al., 1959). The lagoon is the depositional environment of the Euphrates Formation. The thickness is about 50 meters in the study area. The presence of Miogypsina globulina and Miogypsina intermedia indicates that the Euphrates Formation age is late Early Miocene (Burdigalian) (Ctyroky and Karim, 1971 in Jassim and Goff, 2006).

3-Dhiban Formation

Bellen, et al. (1959) state that Henson was the first who described the formation in 1940, and Bellen amended it in 1957. Dhiban, in the Sinjar area of the Foothill Zone, is the formation's type area. The Formation represents the last evaporated sediments from the late Lower Miocene sub-cycle (Buday, 1980). The formation in the study area is mainly covered, but many layers of evaporites were noticed in it. In its type locality, it comprises beds of anhydrite and gypsum with layers of chalky limestone at the upper part with a thickness of more than 40 meters. Due to the collision of the African/Arabian Plate and the Iranian (Eurasian) Plate, the Tethyan Seaway was closed during the Late Burdigalian; this resulted in the deposition of lagoonal (Dhiban) facies (Sissakian, et al., 2016). The early Miocene is the established age of the Dhiban Formation (Jassim and Goff, 2006).

4-Jeribe Formation

This formation was first described by Damensin (1936) in Bellen, et al. (1959); its type section lies near Jaddala village-Jabal Sinjar, which belongs to (Foot Hill Zone) in Northeastern Iraq. Jeribe Formation comprises crystallized limestone (Fig. 2b), fractured limestone, and thin layers of mudstone in addition to dolomitic limestone, as well as layers of chalky limestone and some marl. In the study area, the Jeribe Formation is around 30 meters thick. It was suggested by Bellen, et al. (1959) that the Jeribe Formation was deposited in both reef and lagoonal (backreef) environments (Jassim and Goff, 2006). The age of the Jeribe Formation is assumed to be the Early Miocene. Nonetheless, the formation was subsequently included in the Middle Miocene Sequence. The existence of the Orbulina datum near the Jeribe Formation's base provides evidence for a Middle Miocene age (Prazak, 1974 in Jassim and Goff, 2006).

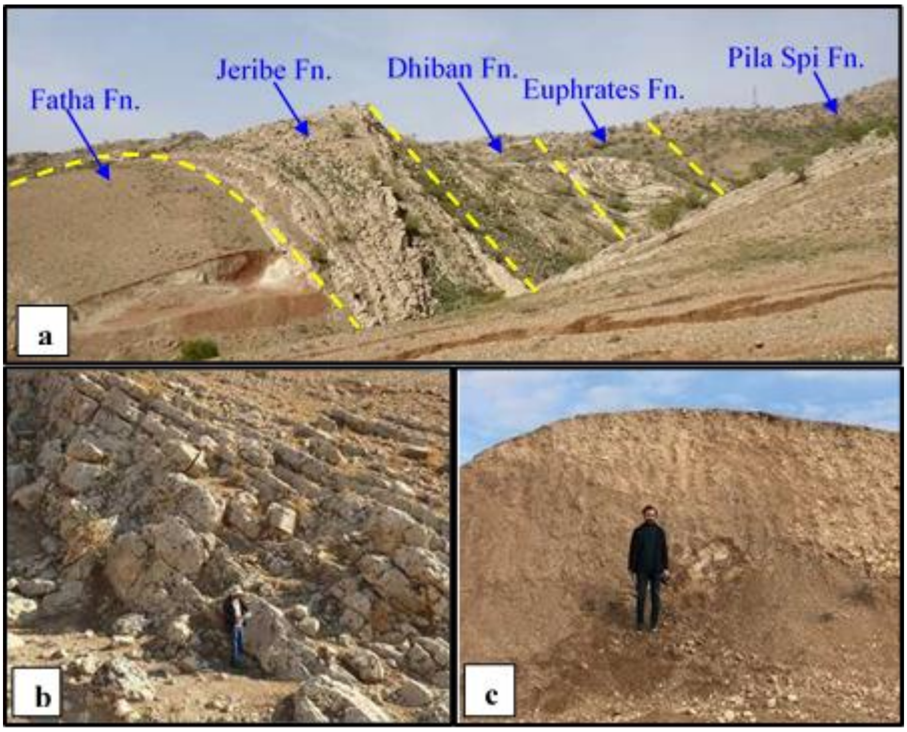

Fig. 2. Photos from the field representing: a) the stratigraphic succession in the southern limb of the Bekhair anticline in the Karweney section. b) Thick to medium bedded limestone of Jeribe Fn. In the Karweney section. c) Red claystone of Fatha Fn. in the Karweney section.

5-Fatha (Lower Fars) Formation

Jassim, et al. (1984) came up with a new name for the Lower Fars Formation in Iraq. They named it the Fatha Formation and placed its type section in the Al Fatha Gorge (10 kilometers north of the town of Baiji, where the Tigris River crosses the Makhul-Hamrin Range) (Al-Juboury and McCann, 2008). It comprises alternating layers of limestone and dolomitized limestone with layers of claystone (Fig. 2c) and with layers of marl. The lower part consists of limestone with green marl and gypsum, as seen in the field. The thickness of the Fatha Formation near Qadya village is more than 200 meters (Sissakian and Al-Jiburi, 2014). The age of the formation is Middle Miocene (Buday, 1980). As agreed, upon by all authors, the depositional environment of the Fatha Formation was a closed lagoon of hypersaline condition (Sissakian and Al-Jiburi, 2014). The age of the formation is Middle Miocene (Buday, 1980).

Methodology

Sampling

The fieldwork was carried out in Bekhair anticline, northwest of Duhok Governorate and southwest of the Zakho district. The fieldwork includes a description of the geology of the area and sampling of four outcropped sections in the coordinates mentioned in (Table 1) extending from the Gulley Zakho Road to the Derabun Village. Samples were taken, stored in plastic bags, numbered, and then transferred to the Department of Earth Sciences and Petroleum workshop at Salahaddin University. It is important to point out that study a section in the middle of the study area was initially planned, but because it is located within the DNO oil field block, therefore sampling there was not allowed due to security reasons.

Table 1. Coordination (UTM system) of the selected sections with number of samples

Sections	Geographic locations	X	Y	No. of samples
Zt	Zakho tunnel	291619	4106325	19
Q	Qadya village	290433	4103213	27
K	Karweney village	272731	4107250	11
T	Tuwan village	273448	4108997	13

Laboratory works

- Preparing 70 thin sections for petrographical study, calcite and dolomite were distinguished by using the alizarin red solution in the Department of Earth Sciences and Petroleum workshop at Salahaddin University.
- The geochemical analysis was conducted by XRF in the laboratories of Kansaran Binaloud Company in Tehran, Iran, to determine the concentrations of major oxides and the percentage of Loss on Ignition (LOI).
- The physical properties of 12 samples, including apparent porosity, bulk density, and moisture content were conducted in the concrete laboratory at the College of Engineering - Salahaddin University.
- The compressive strength of 12 core samples was measured in the concrete laboratory at Salahaddin University's College of Engineering.
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Results and Discussion

Petrography

The petrographic study for industrial purposes includes the diagnosis of the dominant minerals that makes up the rock, the grain size of these minerals, the porosity of the rock, and the distribution of clay minerals in it. In addition to some diagenesis processes that affected the rock.

Through the petrographic study, it was found that the dominant mineral of limestone rocks in this formation is calcite, the size is less than 0.25 mm which is mainly composed of skeletal grains including benthonic foraminifera, gastropods, and pelecypods in addition to non-skeletal grains mainly peloids with rare ooids (Fig. 3a); it was found that there is a small quantity of dolomite crystals in some of the stained thin sections with alizarin red solution as in (Fig. 3b). Quartz is of fine grain size as in (Fig. 3c). Due to the calcite and quartz's fine grain size, the grinding process will not face any issues, which results in little agglomeration in the crushed mixture and homogeneous clinker as a consequence.

The percentage of porosity is low, most of the pores are filled with secondary calcite cement, and a small amount of moldic porosity was observed in some samples as in (Fig. 3c). The percentage of clay minerals is low and did not form a coating around the mineral crystals. In a few samples, oolites were observed as in (Fig. 3d), and no pseudo spars were noticed.

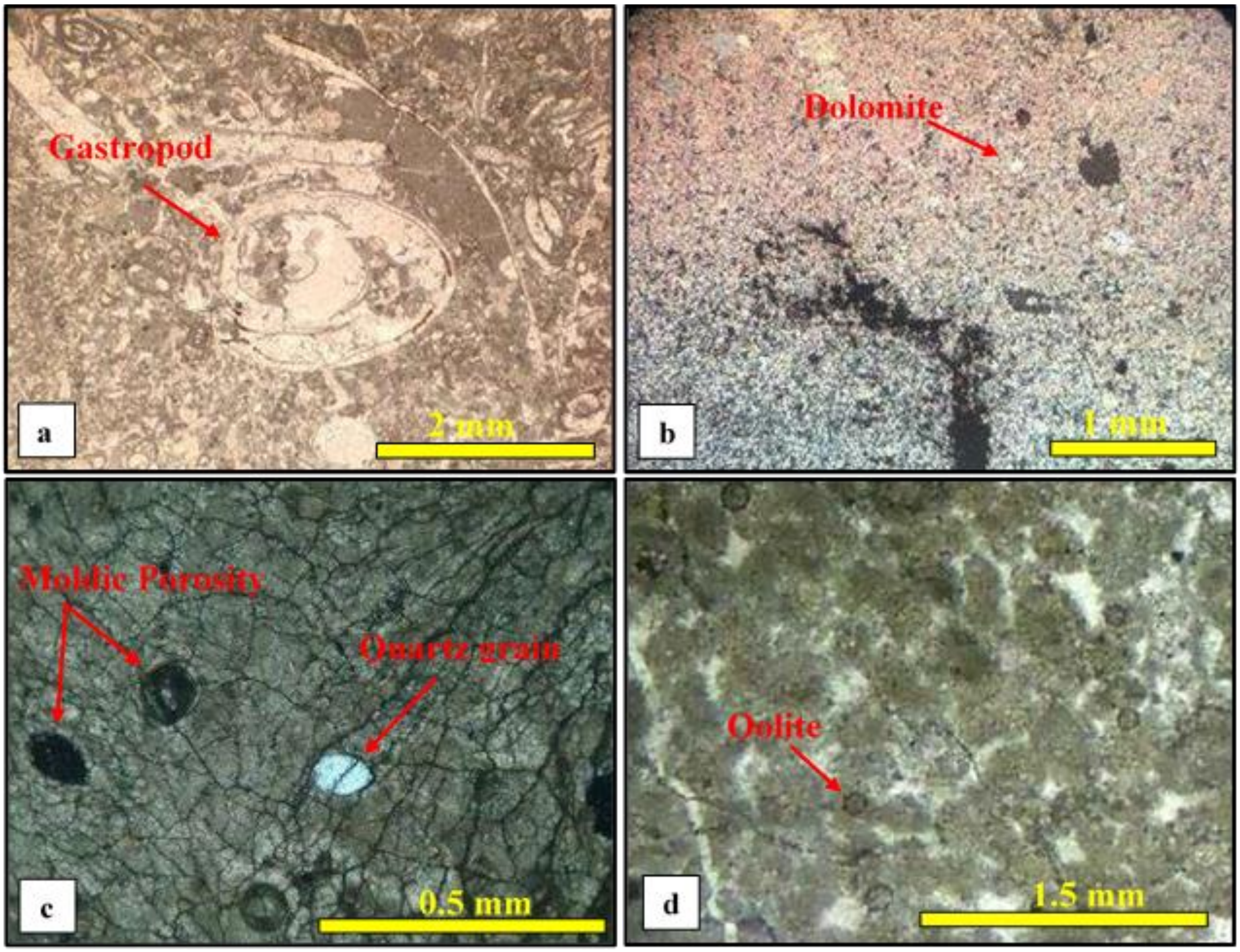

Fig. 3. Photomicrographs of Jeribe limestone showing: a) Gastropod filled with calcite cement (Red arrow) surrounded by benthonic foraminifera and pelecypods, Karweney section, P.P. b) Fine to medium dolomite crystals (red arrow), Qadya section, X.N, A.Z. c) Non-skeletal grain oolite (red arrow), Tuwan section, P.P. d) Quartz grain (red arrow), and moldic porosity (red arrows), Zakho tunnel section, X.N. key: P.P.= Plane Polarized, X.N.= Crossed Nicols, A.Z.= Alizarin Red Stain.

Physical and mechanical properties of limestone

The physical properties of the raw materials used in cement manufacture are just as important as their chemical properties, as they play a role in material selection treatments. The difference in these materials' properties affects the economy of the plant when performing various treatments, beginning with extraction, primary and final crushing, grinding, and the degree of homogeneity (Yezdeen, 1990). SchÖn (2011) states that the internal shape of rock affects its physical properties, such as particle size, pore size, pore connectivity, fracture shape, orientation structure, and texture. Based on IQS No. 31 (1981), the following physical and mechanical properties were examined in this study:

Apparent Porosity: The ratio of the volume of pores to the volume of bulk rock.

$$n = (Vv \div Vt) \times 100$$

n= Porosity, Vv= Volume of voids, Vt= Total volume, the porosity of the studied samples was low, ranging between (1.32 – 7.31 %) (Table 2).

Bulk density: The average density of the rock mass (including pores, etc.) (SchÖn, 2011). (1.68 - 2.70) g/cm3 is the range of the studied samples' bulk density (Table 2). A high bulk density can be related to the low porosity of the studied samples.

Moisture content: The ratio between the weight of the water inside the rock pores (Ww) to the weight of the sample, which is dry (Ws), is expressed in the following form (Hussein, 2012):

$$W\% = (Ww \div Ws) \times 100$$

According to Ghosh and Chatterjee (1977), the moisture content of limestone suitable for use in the cement industry should not exceed 5%. The moisture content of the studied samples ranges between (0.009 - 0.106) % (Table 2).

Table 2. Physical properties of the studied limestone.

Sample No.	Apparent porosity %	Bulk density g/cm³	Moisture content %
Zt-2	2.83	2.64	0.071
Zt-7	2.36	2.66	0.034
Zt-15	1.42	2.64	0.009
Q-3	1.32	2.66	0.011
Q-15	7.31	2.53	0.106
Q-27	5.97	2.57	0.102
K-1	3.50	2.59	0.903
K-3	1.79	2.67	0.093
K-5	2.79	2.46	0.064
T-2	1.72	1.68	0.017
T-3	2.04	2.70	0.031
T-6	3.06	2.61	0.082

Compressive strength: The mechanical property examined in this study is compressive strength which is the maximum stress in compression that certain solid materials can withstand under a gradually applied load without fracturing. Compressive strength (CS) equals force at failure divided by the cross-sectional area

The strength of limestone must be less than 950–1000 Kg/cm²; however, according to international standards, the value of limestone's compressive strength ranges from 458.81–1414.0 Kg/cm² (Chatterjee, 2004). Twelve core samples from the study area have been tested for compressive strength. (Table 3), and they agree with a standard range of limestone listed by Chatterjee (2004). Increases and decreases in compressive strength are related to the porosity of the samples; compressive strength increases as porosity decreases. In addition, joints and fractures affect the compressive strength of the rocks (Fatah and Mirza, 2021).

Table 3. The results of compressive strength of limestone samples

Core No.	Force (KN)	Force (Kg)	Area (cm²)	Compressive strength (Kg/cm²)
Zt-1	473.3	48263.2	37.37	1291
Zt-2	186.9	19058.5	37.37	510
Zt-3	337.2	34384.8	37.37	920
Zt-4	155.4	15846.4	37.37	424
Q-1	141.5	14429.0	35.24	409
Q-2	134.3	13694.8	37.37	366
Q-3	206.5	21057.1	37.37	563
K-1	175.2	17865.4	37.37	478
K-2	238.9	24361.0	37.37	652
K-3	61.7	6291.6	37.37	168
T-1	205.9	20996.0	31.16	674
T-2	53.3	5435.1	31.16	174

Chemical analysis

Cement production is an essential chemical industry that requires a significant amount of raw materials. Using raw materials with the correct chemical composition is essential to producing high-quality clinker, which determines the quality of the resulting cement (Yezdeen, 1990). Portland cement is mainly comprised of compounds lime (CaO), silica (SiO_2), alumina (Al_2O_3), and ferric oxide (Fe_2O_3). The four oxides are the main components of the cement's weight for approximately 90 percent, while the remaining 10 percent comprises magnesia (MgO), alkalis (Na_2O and K_2O), chloride (Cl), SO_3, TiO_2, P_2O_5, and MnO (minor constituents) (Al-Dabbas, et al., 2013).

Chemical analysis of limestone samples

Table (4) shows the chemical analysis of the limestone samples from the Jeribe Formation. The chemical analysis showed that CaO is the dominant oxide range from 25.23% to 55.80%, averaging 51.77%. LOI produced by the decomposition of carbonate minerals as CO_2 ranges from 39.36% to 44.74%, with an average of 43.01%. The amount of MgO which ranged from 0.34% to 18.10%, with an average of (2.06%) reflects the dominance of calcite and deficiency of dolomite (Table 4). Here, the low value of (CaO) and high value of (MgO) in the samples (Zt-9 and Zt-12) are due to the dolomitization and presence of clay fractions in small quantities. When the percentage of MgO in the limestone does not exceed 5% according to IQS No.5 (1984), 6% according to ASTM C150-85 (1986), and 4% according to BS12 (1989), the limestone becomes more suited for cement manufacturing (Al-Ali and Al-Khafaji, 2020). SiO_2 ranges from 0.43% to 11.31%, with an average of 1.95%. Al_2O_3 ranges from 0.19% to 2.72% with average 0.60%. Fe_2O_3 ranges from 0.03% to 2.14% with an average 0.22% (Table 4). Some samples have a high percentage of (SiO_2, Al_2O_3, and Fe_2O_3) due to the presence of clay fractions and quartz grains.

Alkalis content (Na_2O and K_2O) is low, with an average of 0.15%. The SO_3 content for all studied samples is less than 0.25% (Table 4). Excess amounts of SO_3 can lead to expansion, and the standard top limits for ordinary Portland cement are 3.5% (Taylor, 1997). The P_2O_5 content is less than 0.2%. The TiO_2 and MnO are present as traces in the studied limestone samples (Table 4).

The lime saturation factor (LSF) is very high, which means that if this limestone is used for manufacturing cement alone, the proportion of free lime will be very high, affecting the cement's quality. Therefore, claystone must be added to reduce the lime saturation factor. LSF is calculated by the following equations (Chatterjee, 2004):

$$LSF\ (MgO < 2\%) = 100\ (CaO + 0.75\ MgO) / 2.8\ SiO_2 + 1.18\ Al_2O_3 + 0.65\ Fe_2O_3$$

$$LSF\ (MgO > 2\%) = 100\ (CaO + 1.5\ MgO) / 2.8\ SiO_2 + 1.18\ Al_2O_3 + 0.65\ Fe_2O_3$$

The silica ratio (SR) is the ratio of SiO_2 to $Al_2O_3 + Fe_2O_3$. The silica ratio is typically between 1.9 and 3.2 (Duda, 1985). The Aluminum Ratio (AR) determines the ratio of aluminum oxide to ferric oxide (Al_2O_3/ Fe_2O_3). AR acceptable range is between 1.3 and 2.5 (Taylor, 1997). Some samples have SR and AR values, not within the acceptable range. Therefore, to improve them to accord with standard specifications, claystone is added to produce the mixture.

Table 4. Chemical analysis of limestone samples with LSF, SR, and AR values.

No. of mixed samples	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	MnO	P_2O_5	SO_3	LOI	LSF	SR	AR
Zt-1-6	4.79	1.16	0.49	0.03	51.22	0.80	0.19	0.06	0.01	0.02	0.06	41.12	343.19	2.90	2.37
Zt-7	1.01	0.39	0.07	0.00	54.48	0.54	0.02	0.01	0.02	0.02	0.05	43.31	1646.37	2.20	5.57
Zt-8	1.03	0.40	0.07	0.00	54.64	0.44	0.02	0.06	0.02	0.00	0.02	43.14	1616.05	2.19	5.71
Zt-9	11.31	2.72	2.14	0.14	25.23	18.10	0.41	0.05	0.10	0.04	0.22	39.36	144.43	2.33	1.27
Zt-10	1.95	0.52	0.10	0.01	53.63	0.62	0.06	0.05	0.00	0.02	0.03	42.91	881.23	3.15	5.20
Zt-11	1.38	0.36	0.05	0.01	54.06	0.59	0.02	0.07	0.02	0.02	0.05	43.31	1261.25	3.37	7.20
Zt-12	2.74	0.67	0.29	0.02	35.76	15.37	0.08	0.06	0.03	0.02	0.03	44.74	679.86	2.85	2.31
Zt-13	2.36	0.59	0.09	0.01	53.06	0.71	0.06	0.05	0.01	0.02	0.02	42.92	727.89	3.47	6.56
Zt-14	1.81	0.44	0.08	0.01	53.74	0.62	0.03	0.02	0.02	0.03	0.02	43.07	961.22	3.48	5.50
Zt-15	2.35	1.15	0.62	0.04	52.42	0.34	0.96	0.07	0.03	0.08	0.09	41.78	631.56	1.33	1.85
Zt-16	1.85	0.64	0.10	0.01	53.39	0.58	0.07	0.09	0.06	0.01	0.01	43.08	897.05	2.50	6.40
Zt-17	0.96	0.24	0.06	0.00	54.47	0.38	0.01	0.06	0.00	0.01	0.01	43.66	1818.98	3.20	4.00
Zt-18	2.72	0.73	0.12	0.02	52.52	0.71	0.08	0.09	0.04	0.01	0.01	42.80	620.11	3.20	6.08
Zt-19	1.41	0.31	0.06	0.00	54.53	0.42	0.01	0.06	0.00	0.02	0.04	43.08	1259.99	3.81	5.17
Q/1-9	0.48	0.19	0.05	0.00	54.90	0.75	0.01	0.01	0.05	0.04	0.01	43.35	3464.89	2.00	3.80
Q/10-20	0.73	0.31	0.06	0.00	54.71	0.54	0.01	0.01	0.00	0.06	0.09	43.44	2250.69	1.97	5.17
Q/21-23	1.95	0.54	0.11	0.01	53.44	0.71	0.05	0.03	0.03	0.01	0.05	42.97	874.94	3.00	4.91
Q/24-27	1.28	0.53	0.12	0.01	54.20	0.65	0.02	0.03	0.00	0.02	0.05	43.00	1275.54	1.97	4.42
K/1-3	1.70	0.60	0.23	0.01	53.80	0.40	0.08	0.01	0.01	0.02	0.02	42.98	963.06	2.05	2.61
K/4-5	2.50	0.60	0.33	0.05	52.80	0.74	0.08	0.02	0.01	0.02	0.01	42.50	673.46	2.69	1.82
K/6	1.50	0.50	0.22	0.04	53.40	0.70	0.08	0.01	0.01	0.04	0.01	43.30	1093.15	2.08	2.27
K/7-8	0.67	0.43	0.03	0.00	54.13	0.36	0.02	0.09	0.00	0.04	0.03	43.78	2263.93	1.46	14.33
K/9	0.80	0.30	0.24	0.03	55.80	0.40	0.04	0.01	0.01	0.05	0.01	42.20	2040.00	1.48	1.25
K/10-11	1.46	0.66	0.09	0.01	53.48	0.58	0.09	0.05	0.07	0.17	0.01	43.17	1094.65	1.95	7.33
T/1-4	1.17	0.45	0.13	0.01	49.67	4.35	0.04	0.07	0.03	0.08	0.01	43.87	1360.21	2.02	3.46
T/5	0.43	0.27	0.06	0.00	53.37	1.65	0.01	0.02	0.02	0.03	0.16	43.87	34.96.89	1.30	4.50
T/6-7	2.11	0.95	0.25	0.03	43.82	8.33	0.13	0.06	0.01	0.04	0.05	44.13	783.08	1.76	3.80
T/8	1.08	0.40	0.07	0.00	54.42	0.46	0.04	0.06	0.00	0.04	0.02	43.20	1546.38	2.30	5.71
T/9-11	1.64	0.63	0.13	0.01	53.55	0.66	0.10	0.05	0.01	0.05	0.01	43.08	997.16	2.16	4.85
T/12-13	1.18	0.42	0.05	0.00	54.45	0.38	0.06	0.03	0.00	0.15	0.08	43.14	1428.33	2.51	8.40
Average of all samples	1.95	0.60	0.22	0.02	51.77	2.06	0.10	0.05	0.02	0.04	0.04	43.01	1303.19	2.42	4.79

Chemical analysis of the clay sample

Clay is a raw material in ordinary Portland cement and is regarded as the primary source of SiO_2, Al_2O_3, and Fe_2O_3 (Fatah and Mirza, 2021). Chemical analysis of a sample of claystone from the Fatha Formation in the study area shows that the SiO2 is the most abundant oxide with 37.99% and a good percentage of Al_2O_3 and Fe_2O_3 (Table 5). CaO is 17.37%. MgO is 5.61%.

Table 5. Chemical analysis for the studied clay sample

Oxides	Wt. %	Oxides	Wt. %
SiO_2	37.99	K_2O	1.64
Al_2O_3	10.95	Na_2O	0.28
Fe_2O_3	5.63	MnO	0.05
TiO_2	0.60	P_2O_5	0.13
CaO	17.37	SO_3	0.01
MgO	5.61	LOI	19.63

Theoretical raw mixture

The extraction of limestone for the cement industry is performed by large machines, such as shovels and dumpers, for large quantities as a whole formation, not for one or two samples. Therefore, the chemical composition of the mixture was determined by calculating the average of samples suitable for the cement industry in each of the study sections. According to the results presented in (Table 6) drawn from the average of the studied sections, LSF has high values; consequently, this limestone cannot be used alone to manufacture cement, and claystone is added to produce the mixture. Liska and Hewlett (2019) state that the LSF for clinker ranges between (93-98). According to IQS No. 5 (1984), amended in (2019), LSF for cement ranges between (66-102%), while most Iraqi factories work with LSF ranges between (90-100) (Yezdeen, 1990). In this study, LSF = 98 is used to calculate the proportion of raw mix composition. The expected mixing ratios of Jeribe limestone from studied sections with the clay of the Fatha Formation are shown in (Table 7).

Alkalies (sodium and potassium) in Portland cement clinker are primarily derived from clay components in the raw mix. Their total amount is expressed as Na-equivalent (Na_2O + 0.64 K_2O), and ASTM C150 classifies cement with less than 0.6 percent Na-equivalent as low-alkali cement and cement with more than 0.6 percent Na-equivalent as high-alkali cement. An alkali concentration of 0.6 percent or less is usually determined to produce damage due to the alkali-aggregate reaction in ordinary concrete (Mehta, 2006). The Na-equivalent values of mixtures are more than 0.6 percent, which needs a 5% bypass in cement plant design (Table 7).

Table 6. Average composition of the studied sections

Oxides	Zt	Q	K	T
SiO_2	2.69	1.11	1.44	1.27
Al_2O_3	0.74	0.39	0.52	0.52
Fe_2O_3	0.31	0.09	0.19	0.12
TiO_2	0.02	0.01	0.02	0.01
CaO	50.23	54.31	53.90	51.55
MgO	2.87	0.66	0.53	2.64
K_2O	0.14	0.02	0.06	0.06
Na_2O	0.06	0.02	0.03	0.05
MnO	0.03	0.02	0.02	0.01
P_2O_5	0.02	0.03	0.06	0.06
SO_3	0.05	0.05	0.02	0.06
LOI	42.73	43.19	42.99	43.55
LSF	963.51	1966.52	1354.71	1602.01
SR	2.86	2.24	1.95	2.01
AR	4.66	4.57	4.94	5.12

Table 7. Theoretical raw mixture and cement clinker composition, parameters, phases, and properties (HM= Hydraulic modulus, MBT= Minimum burning temperature, LPH= Liquid phase at the burning zone, and BI= Burnability index)

	Requirements	Lst. (Zt)+clay	Lst. (Q)+clay	Lst. (K)+clay	Lst. (T)+clay
Theoretical raw mix	Clay	0.307	0.341	0.334	0.332
	Limestone	0.693	0.659	0.666	0.668
	SiO_2	13.53	13.68	13.63	13.48
	Al_2O_3	3.88	3.99	4.00	3.99
	Fe_2O_3	1.94	1.98	2.00	1.95
	CaO	40.14	41.72	41.72	40.19
	MgO	3.71	2.35	2.22	3.63
	K_2O	0.60	0.57	0.59	0.59
	Na_2O	0.13	0.11	0.11	0.13
	SO_3	0.04	0.04	0.11	0.06
	LOI	35.64	35.16	35.20	35.60
	Total	99.61	99.60	99.58	99.62
Clinker composition	SiO_2	21.15	21.23	21.17	21.06
	Al_2O_3	6.06	6.19	6.21	6.23
	Fe_2O_3	3.04	3.07	3.11	3.05
	CaO	62.75	64.76	64.79	62.80
	MgO	5.80	3.64	3.45	5.67
	K_2O	0.94	0.89	0.91	0.91
	Na_2O	0.20	0.17	0.18	0.20
	SO_3	0.06	0.06	0.17	0.09
	LOI	0.00	0.00	0.00	0.00
	Total	100.00	100.00	100.00	100.00
	Requirements	Lst. (Zt)+clay	Lst. (Q)+clay	Lst. (K)+clay	Lst. (T)+clay
Clinker parameters	LSF*	98.00	98.00	98.00	98.00
	SR	2.33	2.29	2.27	2.27
	AR	1.99	2.02	1.99	2.04
Na-equivalent		0.80	0.73	0.76	0.78
Clinker phase	C3S %	49.46	56.70	56.43	49.19
	C2S %	23.11	18.33	17.88	23.02
	C3A %	10.87	11.16	11.14	11.30
	C4AF %	9.20	9.29	9.42	9.23
Clinker properties	HM	2.07	2.12	2.12	2.07
	MBT (°C)	1396.10	1423.64	1423.72	1392.92
	LPH at 1338 °C	25.38	23.33	23.43	25.27
	LPH at 1400 °C	31.38	29.59	29.78	31.73
	BI	2.46	2.74	2.74	2.40

Clinker parameters

The quality parameters (moduli) must be measured periodically to ensure that the clinker quality remains within an acceptable range. The clinker quality parameters are lime saturation factor (LSF), silica Ratio (SR), and alumina Ratio (AR).

The LSF governs the clinker's alite-to-belite ratio; the ratio of alite C3S to belite C2S is greater in clinkers with a high LSF compared to those with a low LSF. (Fatah and Mirza, 2021). The silica ratio, SR, is the ratio of silica to alumina and iron oxide, with a value of (2.4-2.6) for typical clinker (Newman and Choo, 2003).SR ratio was (2.33, 2.29, 2.27, and 2.27) for the studied sections (Zt, Q, K, and T) respectively (Table 7). The proportion of calcium silicates C3S and C2S that can be produced in the clinker increases with the silica ratio, but less liquid can be produced since the combination of C3A and C4AF will be decreased (Hewlett and Liska, 2019).

The AR controls the clinker's aluminate to ferrite phase ratio, significantly affecting cement properties. It also controls the quantity of liquid produced at relatively low temperatures. Ordinary Portland cement clinker typically has an AR between 1.0 and 4.0. (Rao, et al., 2011). The AR in the raw mixtures was (1.99, 2.02, 1.99, and 2.04) for the studied sections (Zt, Q, K, and T), respectively (Table 7); This means all studied sections have acceptable ranges.

Clinker phase

In cement chemistry C = CaO, S = SiO_2, A = Al_2O_3, and F = Fe_2O_3. C3S=3CaO.2SiO_2, C2S=2CaO.SiO_2, C3A=3CaO.Al_2O_3, C4AF=4CaO.Al_2O_3.Fe_2O_3. The clinker phases C3S, C2S, C3A, and C4AF in the studied sections range between (49.19-56.70), (17.88-23.11), (10.87-11.30), and (9.20-9.42) %, respectively (Table 8).

Table 8. Main phases in the clinker

Phase% / Mixtures	C3S	C2S	C3A	C4AF
Zt	49.46	23.11	10.87	9.20
Q	56.70	18.33	11.16	9.29
K	56.43	17.88	11.14	9.42
T	49.19	23.02	11.30	9.23
Newman and Choo (2003)	45-65	10-30	5-12	5-12

Clinker properties

Some factors affect the properties of clinker, which are:

Hydraulic modulus (HM): It refers to the ratio of calcium oxide (CaO) to the total of the three oxides (SiO_2, Al_2O_3, and Fe_2O_3). The value of this factor generally lies within the range (1.7-2.3). Low strength cement has a hydraulic modulus of less than 1.7, and a rise in these values demands the use of more heat to complete the clinker-burning process (Duda, 1985). The H.M. of clinker in the studied sections (Zt, Q, K, and T) was (2.07, 2.12, 2.12, and 2.07), respectively (Table 7), showing that the H.M. of all sections falls within the acceptable range.

Minimum burning temperature (MBT): It is defined as the degree of liquid phase formation in the kiln and has the following form: (Yezdeen, 1990):

$$MBT°C = 1330 + 4.51 * C3S – 3.74 * C3A – 12.64 * C4AF$$

The ratio of lime to silica makes the value go up, and it is better that it doesn't go below 1250 °C. Because this temperature (C3S) has just begun to appear. (Chatterjee, 1979). The MBT of clinker in the studied sections (Zt, Q, K, and T) was (1392.10, 1423.64, 1423.72°, and 1392.92), respectively (Table 7); as a result, the MBT of the four mixtures is acceptable.

Liquid phase at the burning zone (LPH): It is the percentage of the liquid formed when burning that has the most significant influence on the ability to form clinker; its content is dependent on the temperature and composition of the raw mixture, as well as the amount of impurities (Yezdeen, 1990). Its percentage can be calculated within the burning zone at different temperatures such as 1338 °C, 1440 °C, and 1450 °C. In this study, the liquid phase was calculated at a temperature of 1338 °C and 1400 °C according to the following formulas:

$$\textbf{LPH \% (at 1338 °C)} = 6.10F + MgO + K_2O + Na_2O, \text{ when } A/F \geq 1.38$$

$$\textbf{LPH \% (at 1400 °C)} = 2.95\, Al_2O_3 + 2.20\, Fe_2O_3 + MgO + K_2O + Na_2O$$

The amount of liquid phase in the burning zone varies between 23% and 29% (Shabana, 2013). At a temperature of 1338 °C, the LPH of the studied sections (Zt, Q, K, and T) was (25.38, 23.33, 23.43, and 25.27), respectively (Table 7). While LPH, at a temperature of 1400 °C, has higher values (31.38, 29.59, 29.78, and 31.73) for the studied sections (Zt, Q, K, and T) respectively (Table 7), and they are out of the acceptable range. According to Gouda (1979), the excess in the liquid phase causes the clinker's high resistance to grinding and the resulting high energy consumption. Therefore, the burning temperature of this clinker should not exceed 1400 °C.

Burnability index (BI): This is a measure of the clinker's ease of burning; the higher the index number, the more difficult the clinker is to burn (Peray, 1979).

$$\mathbf{BI = C_3S / C_3A + C_4AF}$$

The BI value ranges between (2.6-4.5) (Al-Ali, 2004). For the clinker of the studied sections (Zt, Q, K, and T), the respective BI values were 2.46, 2.74, 2.74, and 2.40 (Table 7), showing that the two sections fall within the acceptable range.

Conclusion

- The petrographic study showed that the main mineral of the limestone rocks in this study is calcite, the porosity in these rocks is low, and the pores were filled with secondary calcite cement.
- According to their physical and mechanical properties, these rocks are appropriate for the cement industry, as they are easily crushable and grindable and produce a homogenous mixture.
- Chemical analysis of limestone samples revealed that the major oxide proportions in the rocks are suitable as raw materials for the cement industry.
- The studied limestone has a high LSF, indicating that it is very pure; to reduce this ratio and produce a mixture that conforms to international standards for cement manufacturing, claystone from the Fatha Formation in the study area was added, and fixed LSF (98) was used to calculate clinker compositions and estimate raw mix proportions.
- The majority of clinker chemical parameters, such as LSF, SR, AR, Clinker phase (C3S, C2S, C3A, and C4AF), and clinker properties, such as hydraulic modules, minimum burning temperature, and burnability index, are within the standard specification for manufacturing Portland cement.
- When the clinker is burned at 1338°C, the values of the liquid phase are within the acceptable range. While, at 1400°C, the values are higher and outside the range, which causes the clinker's high resistance to grinding and high energy consumption; therefore, this clinker should not be burned over 1400°C.

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