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Geochemical Assessment of Oligocene Limestone for Conceivable Use as Raw Material for Portland Cement in Sangaw Area, Iraqi Kurdistan

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ABSTRACT

The sedimentary deposit of Oligocene rocks is outcropped in Ashdagh mountain in south of Sulaymaniyah - Sangaw area and characterized by highly calcareous limestone, especially in the lower parts. The rocks of this age are composed of nine formations included Shurau, Sheikh Alas, Planai, Baba, Bajwan, Tarjili, Anah, Azkand, and Ibrahim those are deposited during Oligocene. These formations grouped into three types included basinal, lagoonal and reefal according to depositional environment. The aim of the study is geochemical assessment for limestone rock as major raw material in Portland cement product. The study based on XRF chemical composition analysis and loss on ignition for thirty-eight handpicked samples of the area and evaluation of major oxides as Portland cement raw material. The chemical results showed that the tested limestone samples are overwhelmingly composed of calcareous limestone. The chemical investigation appears that the entire progression is comprise of calcareous limestone, which comprises loss on ignition averagely above 42.9% and mostly less than 1% in MgO, Fe_2O_3 and Al_2O_3. The limestone classified as high-grade limestone according to the analysis results, which is exceptionally appropriate for Portland cement producing. Geochemical results of samples specified that the limestone contains high ratio of LOI, which it ranges between (38.7% to 43.9%), and other major oxides are in suitable range while alkali metal oxides and SO_3 are present in traces. The ratios of the silica modulus (SM), alumina modulus (AM), and lime saturation Factor (LSF) were calculated. It was found that these proportions of examples are mostly consistent with Iraqi Standard Specifications for Portland cement by adding silica, aluminium and iron oxides. The lime saturation factors (LSF) of assessed samples have extensively differing values, but most values have above the limits required for high quality cement. The overall outcomes confirmed that the Oligocene limestone could be use as major raw material for clinker production in cement industry.

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التقييم الجيوكيميائي للصخور الكلسية لمجموعة الأوليجوسين وامكانية استخدامها كمواد خام أولية في صناعة الأسمنت في منطقة سنكاو / كردستان العراق

سامان غريب رشيد ، عمر أحمد رشيد ، ريذةن جميل

مديرية النفط والمعادن– السليمانية ، أقليم كردستان ، العراق

الملخص

تتكشف الصخور الرسوبية لمجموعة الأوليجوسين في جبل اجداغ جنوب مدينة السليمانية / منطقة سنكاو، حيث تتميز بكونها من الأحجار الكلسية الجيرية خاصة في الجزء الأسفل. تتألف مجموعة ألأوليجوسين من تسعة تكوينات صخرية وتشمل كل من شوراو، شيخ علاس، بالاني، بابا، باجوان، تارجيل، عانه، أزقند وأبراهيم والتي ترسبت خلال فترة ألأوليجوسين. تم تقسيم وتصنيف هذه التكوينات الصخرية الى ثلاثة مجاميع ويشمل السحنات الحوضية العميقة، والمناطق الضحلة والحيد البحري. هدف الدراسة هي التقييم الجيوكيميائي للحجر الجيري وامكانية أستخدامه كمواد أولية في صناعة الأسمنت. أعتمدت الدراسة على التحليل الجيوكيميائي للمكونات الصخرية والفقدان أثناء الحرق بواسطة استخدام اشعة (XRF) التي أجريت على ثمانية وثلاثون نموذجا صخريا تم أخذ ها من الحقل في منطقة سنكاو. أظهرت نتائج التحليل الجيوكيميائي التي أجريت على الأحجار الجيرية انها مكونة بشكل رئيسي من الحجر الجيري الكلسي، وكانت نسب كل من (Al_2O_3 , Fe_2O_3 , MgO) أقل من 1%. يمكن تصنيف الحجر الجيري في المرتبة العليا بامتياز في صناعة الأسمنت أعتمادا على التحليل الجيوكيميائي. كما أثبتت نتائج التحليل الجيوكيميائي للنماذج أن نسبة الفقدان أثناء الحرق عالية ويتراوح مابين (% 43,9 -38.7%) وأن نسبة ألأكاسيد الرئيسة ضمن الحدود المناسبة في حين أن نسبة القلويات والأملاح والكبريتات قليلة جدا. وتم احتساب نسبة معامل السليكا (SM) ومعامل الألمنيوم (AM) وعامل التشبع الكلسي (LSF). وقد تبين ان هذه النسب مطابقة للمواصفات القياسية العراقية بعد أضافة أكاسيد السليكا والألمنيوم و الحديد. وقد تبين ان قيم عامل التشبع الكلسي (LSF) متباينة وأن أغلبية القيم أعلى من الحد المناسب في صناعة الأسمنت. أجمالي النتائج أثبت أن الصخور الكلسية لمجموعة الأوليجوسين تعتبر مواد أولية أساسية في صنع الكلنكر الرئيسية في صناعة الإسمنت.

Introduction

Limestone is one of the foremost materials involved in the manufacture of Portland cement, which is the most communal type of cement for all-purpose usage in construction. About three-quarters of main raw materials in the kiln feed consist limestone [1]. The suitable limestone reserve is diminishing annually, because of relentless exploitation and high demand in the region and Iraq over the years. However, cement manufacturing is one of the sources of increasing CO_2 emission to environment, but now Iraq needs to more reconstruction due to destroyed during severe wars of the last years.

Cement is a mass-produce material that made by amalgamation some diverse materials after physical and chemical evaluations, burning mixed materials at require temperature until achieve the specific chemical proportions of lime, silica, alumina and iron which known as cement clinker. Principally, cement is a mixture of calcium silicates (Alite and Belite) and lesser quantities of Aluminate and Ferrite that respond with water and cause the cement to set [1].

The main necessity for limestone is met by selected high calcium limestone (or its equivalent calcareous raw material) and clay from mudstone or shale rocks as the focal source of silica and alumina. The product process is finished by powdering around 95% cement clinker together with 5% gypsum, which is used as controller material to impede and prompt hardening time of the cement paste.

Geochemical assessment is the main valuation to indicate suitability of the rock and using as raw material for produce cement. The chemical composition of raw materials that used, is directly controlling the quality of clinker cement. Approximately 80-90% of raw material for clinker kiln feed is grinded limestone. In addition to, aluminosilicate as clayey consist rate is from 10 to 15%, while the precise ratio will vary according to the nature of used materials. Normally, magnesium appears as the main debasement impurity that is present in limestone. Availability of Magnesia (MgO) within the clinker should not surpass 5% and many factory producers approve the content up to 3%, this instruction put an end to usage dolomite and dolomitic limestone in cement industry [2]. Other detritus oxides such as including excessive alkalis, especially sodium and potassium oxides would be an improper and deleterious effect on concrete durability and finally reducing the quality, this react of alkalis with amorphous silica in the mixture forming silica gel which known as concrete cancer.

Some factors such as LSF, SR, and AR are calculated to get an idea about suitability of the limestone and possibility to make a good mixture to form the clinker without problems.

The aim of this study is an explore and chemical inspect to a new resource of the main raw material for Portland cement production in the area and easily

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exported products to other parts of Iraq. The importance of this study is an assessment for another rock unit, located in Sangaw area while most of the cement factories use limestone of Sinjar formation and concentrated in Bazian area. Also, it makes a chance to groundwork for a cement factory and increasing job opportunity in the area. The limestone of Oligocene may have low hardness by compare Sinjar limestone because of the hardness of limestone depends on geological age, typically the younger geological formation has lower hardness limestone [3]. In addition to suitability of the site to build cement factory and availability of the other raw material sources of cement such as clay and gypsum beds of the Fat'ha formation.

1.2 Location

The studied area situated in Sangaw area, Sulaymaniyah governorate, Kurdistan region of Iraq, about 40km southwest of the Sulaymaniyah city and about 6km south of Sangaw district (fig.1). Mostly, rock beds outcropped along the gorge, which covered on top by recent deposit, all samples taken in ten sections (table 1). The area is intersected by many valleys extending from northeast to south and south-west at the nose of Ashdagh and Qarawais mountains. The highest elevation recorded in the studied area is 991m and the least elevation is about 854m.

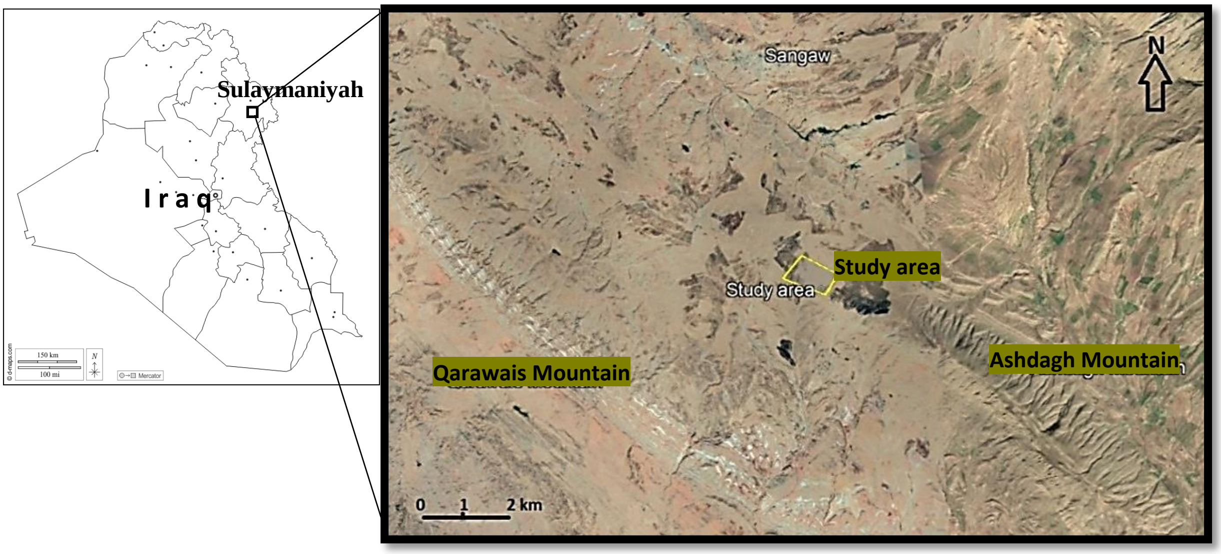

Fig. 1: The location of the studied area (from Google earth)

Table 1: Coordination number of studied sections

Section 1	35°13'48.30"N; 45°11'40.92"E	Section 6	35°13'51.49"N; 45°11'12.97"E
Section 2	35°13'46.59"N; 45°11'35.20"E	Section 7	35°13'54.54"N; 45°11'12.31"E
Section 3	35°13'44.54"N; 45°11'29.59"E	Section 8	35°13'56.61"N; 45°11'19.73"E
Section 4	35°13'43.01"N; 45°11'22.92"E	Section 9	35°14'2.97"N; 45°11'3.11"E
Section 5	35°13'47.42"N; 45°11'14.29"E	Section 10	35°14'6.10"N; 45°11'28.15"E

1.3 Geological Setting

Geologically, the studied area lies within the low folded zone according to the tectonic division of Iraq. Generally, the structures of the area are trending from NW to SW. The Ashdagh mountain is plunged in NW at studied area and a part of the unstable shelf, foothill zone, Erbil - Chamchamal subzone [4]. The attitude of bedding planes is toward N-NW and S-SW with 2-15 degrees dip angle. The western face of Ashdagh mountain is bounded by Qarawais mountain while the eastern side is neighbour with Sagrma mountain. Stratigraphically, the outcrop rocks are attributed to Oligocene Formations, which proved by [5]. The Oligocene formations of the area are Shurau and Sheikh Alas Formations are deposited in back reef to fore reef depositional environment during early Oligocene, Bajwan, Baba and possible Tarjil Formations sedimented in back reef, fore reef, and basinal environments along Middle Oligocene, Anah Formation as back reef deposit which deposited in late Oligocene, they are underlined by Eocene rock (Pila Spi Formation), while it is overlain by Miocene rock units (Jeribi, and Fatha Formations), the lower and upper of Oligocene rock units are bounded by unconformities. The maximum thickness of the Oligocene units in the studied area is about 45m.

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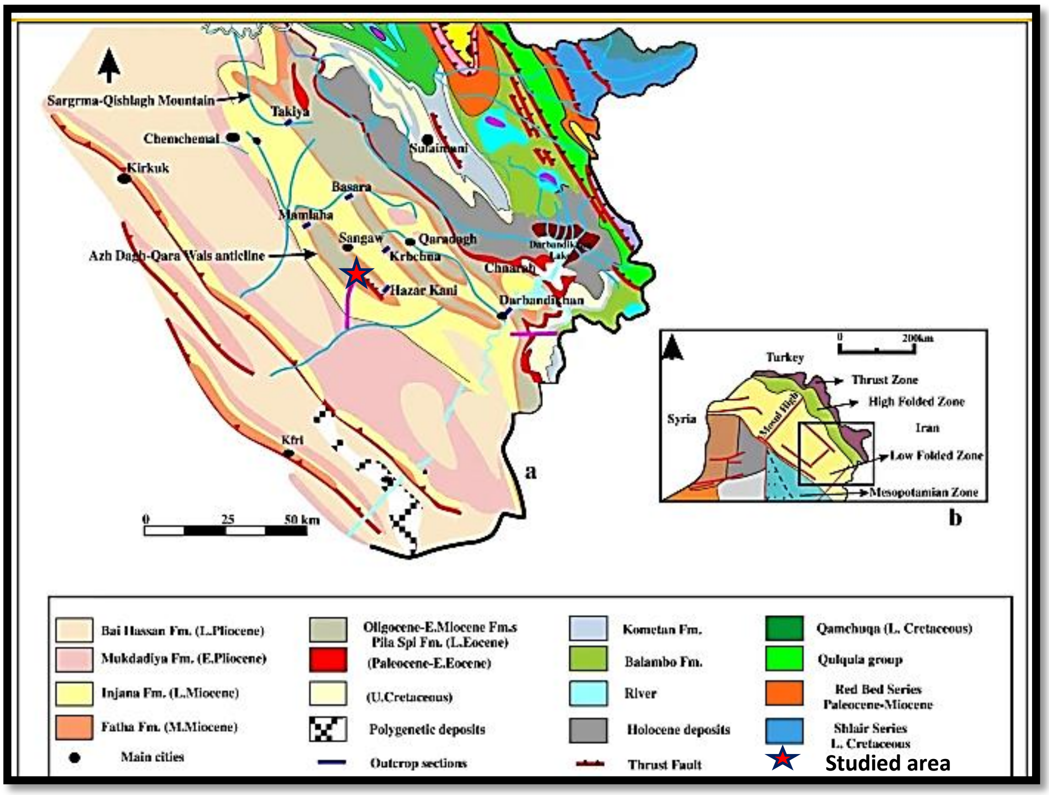

Fig. 2: Geological map of the studied area [4]

2. Methodology

The study started by a detail review through previous studies of the area as the first step of exploration. Industrial study then selected the position of the study based on the rock beds suitability for quarrying and cement investment in there depending on the stratigraphy and geomorphology of the area. The later stage is finding suitable outcrops and defining with full description. Rock sampling were performed for ten sections set which perpendicular to the strike of the bedding planes, from the lowermost beds to the uppermost. The sampling method is based on the variation in physical properties of the rocks such as color, degree of crystallization, hardness, texture and the presence of fossils, in addition to stratigraphy including changes in facies or rock units. Collecting fifty samples of Oligocene limestone during three days of fieldwork and selected thirty-eight samples of them to chemical analysis. Ultimately, all the rock samples were sent to Zarazma Mineral Studies Laboratory in Tehran for the X-ray fluorescence spectrometer chemical test by ICP-OES model. The analysis included major oxides such as calcium oxide (CaO), Magnesium oxide (MgO), Silica (SiO_2), Aluminium oxide (Al_2O_3), Iron oxide (Fe_2O_3), Sulphate (SO_3), Potassium oxide (K_2O), Sodium oxide (Na_2O) and the total loss on ignition (LOI). Those oxides have a great role in assessment of the limestone for cement industry. Other oxides such as MnO, TiO_2 and P_2O_5 are below detection limit (<0.05%).

3. Result and Discussion

Chemistry of limestone as a main raw material in Portland cement is the most significant part in assessment procedure. Chemical composition results of selected samples from the studied area designated those samples are dominantly composed of calcium oxide that ranges from 49.25 to 55.9% and averagely about 54.6% (table 2), according to the chemical results, the purity of limestone samples are mostly between medium to very high pure [6]. Lime (CaO) is the most important major oxide for Portland cement manufacturing and forming calcium silicate face; require CaO content is allied with a primary strength, which favours final strength that steadily increases over an extended period. Loss On Ignitions (LOI) have a second place, averagely 42.9% in samples. The LOI percentages in those samples showed a strong positive correlation with CaO (fig. 3A) which is generally contributed with carbonate minerals in high ratio. Magnesia, alumina and Iron oxides exhibit minor concentration while Sulfur trioxide and alkalis are existing in the traces ratio for most of the samples. In addition to CaO content, other oxides such as SiO_2, Al_2O_3 and Fe_2O_3 are the main constituents in the chemical structure of cement but should be within allowable limits. The maximum MgO in the samples is about 1.07%, the magnesia content should not exceed 5% which designated by the Iraqi standard specifications for Portland cement. Normally higher magnesia content makes magnesia expansion especially in late of ages, which is

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unfavourable to the soundness of the cement resulting in expansion blow [7, 8]. The rate of iron oxides is not high, averagely less than 1%. The SiO_2 content in samples vary between 0.11 % to 7.92 % most of the samples below 2%, which is generally related to have detrital quartz grains in pores and cracks [9]. We have a very strong negative correlation between calcium and silica oxides in tested samples (Fig. 3B) which is the coefficient about -0.99 (table 3). The negative correlation is related to miscellaneous mineral phases of silicate and calcite, which they are not interrelated [10]. In general, other oxides exhibit low concentration in most of the samples that is sign to pure limestone. Alumina percentages between 1.65% to less than 0.05% and have an inverse correlation with calcium oxide only, while shows positive correlation with magnesium, potash, soda, and iron oxide (table 3) which could be by reason of clay materials existing in the samples or silicates like feldspar.

3.1 Lime Saturation Factor (LSF)

LSF is the elementary calcualtion for raw material composition that used to indicate the excess of lime available from the limestone, and additional lime required to saturate the acidic oxides at standard level, the ratio of these two gives the propotions of limestone and clay requierd in the clinker mix [3]. This factor is drived by Chatterjee, 2004 [11] as a ratio of calcium oxide to other three main clinker oxides as calculated, LSF (when, MgO<2%) = (CaO+0.75*MgO)/ (2.8 SiO_2+1.18 $A1_20_3$+0.65 Fe_2O_3).

In addition to the effects on the burning process, high LSF needs high energy for the burning of the clinker in the kiln, consequently giving higher strength to the cement hereafter make problematic in grinding [12], however leads to high production cost although. The ordinary clinkers always likely to be contained free lime when LSF>1 because the typical LSF value in kiln feed are in range 0.66-1.02 or 66-102% in the Iraqi cement specification [13]. Principally, to form alite (C_3S) in clinkers any grain of the free lime should have be combined with belite completely. The LSF in the studied samples showed high LSF values ranging from 2 to 95 which is related to high CaO and low impurites in the samples. Adding extra Alumino-silicate (clay) sources are required to reach the suitable LSF rate in the clinker and shared all the free lime. The wider range of LSF in the samples require to the more accurate producting process with a consideration LSF ratio of different limestone beds.

3.2 Silica Ratio (SR)

Silica-ratio or silica modulus (SM) is one of the important ratios that have a great effect on burning process also, contributes a part of performance in cement quality and one of the most often applied modulus for control cement types or purposes. The silica ratio describes as the amount of SiO_2 to the total Al_2O_3 and Fe_2O_3. The standard ratio for the modulus is typically among 2.0 and 3.0 in Portland cement clinker [14]. The high silica ratio creates more difficulties in the burning process and exhibit poor coating characteristics. Normally, additional energy is required to run the kiln when the silica ratio increased [15] which is related to high calcium silicates in the clinker by compare to alumina and ferrite. In case silica ratio is lesser than the standard ratio, the clinker leads to the ring formation in the wall of the kiln due to increasing more melt phase, which is made a big issue for production process along time. SR in the studied samples appeared ranges from 0.6 to 10.8 except sample 19 that showed very high SR (39.6) which related to high SiO_2 and very low Al_2O_3 and Fe_2O_3 contents. However, most of the samples comprised in standard range, the higher SR require to be brought down and vice versa for the low SR samples, to stay in SR specified limits for producing cement clinker in high quality.

3.3 Alumina Ratio (AR)

The Alumina-ratio (AR) represents the proportion of Al_2O_3 to the Fe_2O_3. The standard alumina ratio (AR) is ranges from 1 to 4 in regular Portland cement clinker [16]. The ratio shows the suitable relative proportions of the aluminate and ferrite phase of feeding materials in the kiln which effects on the minimum burning temperature and affects the colour of the product [17]. The clinker will be easier burning when the ratio is about 1.4 due to forming more liquid clinker at a lower temperature [9]. AR results for the existing samples are from 0.6 to 3.58. Regularly, using clay and iron oxide to regulate the chemistry of the limestone was to preserve AR within standard limits for the clinker.

4. Implications in Cement Production

The chemical composition and white the color of the most studied limestone indicated that the samples are pure limestone. The majority of mineral content of the limestone samples appear simple whereas CaO showed a strong positive correlation with LOI in the same time CaO with SiO_2 presented a strong negative correlation, which completely related to the mineralogical composition (fig. 3). The most objective of calculating the resource chemical ratios is to indicate the quantitative proportions of the raw components with the purpose of generate the favourite compositions for the clinker. Based on the geochemical data and significant factor ratios for all of the limestone units in the studied area need to add some quantity of sand, clay and iron to compensate the suitable percentage of silica, alumina and iron oxides to the standard clinker. The lime saturation factor, silica and aluminium modulus from geochemical data showed that the limestone need to add further silica and aluminium source to produce clinker.

The values of LSF showed a wide variety ranging from 2 to 95, normally all of the studied samples had values above the required limits and vice versa for SR

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and AR values which most of the samples located below of the standard limit for high quality cement. The most suitable additive to treatment the mixture is claystone or alumina-silicate source, the Lower-Fars formation outcropped near the studied area, which contains a succession of claystone and gypsum beds. In addition to the gypsum source in the area increased the site suitability for manufacturing cement, the gypsum could be adding to the mixer to control the setting properties and confirm the compressive strength of cement. Na_2o, K_2O and SO_3 as oxides are very low in all samples that will not have a prominent effect on the final quality of the cement produced or on the manufacturing process; inversely the alkalis in smaller quantities in studied limestone are made it suitable for all types of cement. While they may belong to silicate minerals.

Table 2: Chemical composition of Oligocene limestone in studied area (detection limit for all oxides is 0.05%).

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI	Sum
1	4.31	0.93	0.34	52.35	0.52	0.05	0.07	0.24	41.14	99.95
2	5.78	1.24	0.58	51.09	0.57	0.08	0.07	0.32	40.15	99.88
3	0.85	0.18	0.13	55.04	0.35	0.09	0.06	0.05	43.25	100
4	7.85	1.65	1.11	49.25	0.79	0.08	0.05	0.38	38.7	99.86
5	0.92	0.25	0.1	55.12	0.19	0.06	0.05	<	43.31	100
6	1.06	0.33	0.09	55	0.18	<	0.05	0.08	43.21	100
7	2.79	0.86	0.24	53.5	0.29	<	0.05	0.24	42.03	100
8	2.32	0.7	0.35	53.84	0.24	<	0.05	0.19	42.31	100
9	2	0.53	0.24	54.22	0.23	<	0.05	0.13	42.6	100
10	4.32	0.28	0.12	53.22	0.18	<	<	0.06	41.82	100
11	0.4	0.1	0.06	55.43	0.13	0.32	<	<	43.56	100
12	2.39	0.21	0.25	54.24	0.18	0.12	<	<	42.61	100
13	0.7	0.19	0.1	55.33	0.15	0.05	<	<	43.48	100
14	0.62	0.19	0.17	55.35	0.18	<	<	<	43.49	100
15	1.42	0.42	0.2	54.64	0.19	0.1	<	0.09	42.94	100
16	0.71	0.22	0.23	55.16	0.16	0.07	0.06	0.05	43.34	100
17	1.29	0.4	0.23	54.62	0.24	0.24	<	0.07	42.91	100
18	1.54	0.53	0.23	54.52	0.18	<	0.05	0.12	42.83	100
19	4.38	0.05	0.06	53.36	0.12	0.05	0.05	<	41.93	100
20	0.48	0.1	0.07	55.45	0.16	0.18	<	<	43.56	100
21	0.51	0.17	0.09	55.41	0.11	0.17	<	<	43.54	100
22	2.08	0.66	0.2	54.03	0.31	0.14	<	0.15	42.43	100
23	7.6	1.65	0.76	49.19	1.07	0.41	0.1	0.42	38.61	99.81
24	0.55	0.13	0.13	55.44	0.09	0.1	<	<	43.56	100
25	0.32	0.1	0.08	55.57	0.12	0.15	<	<	43.66	100
26	0.43	0.12	<	55.56	0.14	0.09	<	<	43.66	100
27	0.1	<	<	55.87	0.06	0.07	<	<	43.9	100
28	0.79	0.14	0.08	55.33	0.07	0.11	<	<	43.48	100
29	0.11	<	0.23	55.75	<	0.1	<	<	43.81	100
30	<	<		55.9	0.08	0.1	<	<	43.92	100
31	0.11	<	0.05	55.47	0.05	0.74	<	<	43.58	100
32	<	<		55.92	0.07	0.07	<	<	43.94	100
33	0.21	<	0.05	55.74	0.1	0.11	<	<	43.79	100
34	<	<	<	55.9	0.08	0.09	<	<	43.93	100
35	0.06	<	<	55.85	0.08	0.12	<	<	43.89	100
36	<	<	<	55.9	0.08	0.1	<	<	43.92	100
37	0.16	0.06	0.1	55.65	0.19	0.12	<	<	43.72	100
38	0.21	0.09	<	55.62	0.22	0.1	0.06	<	43.7	100

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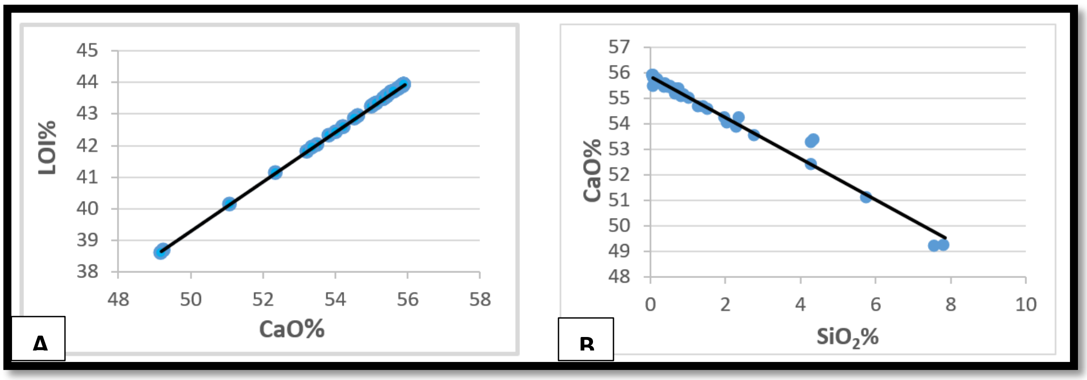

Fig. 3: A) CaO and LOI positive correlation, B) CaO and SiO_2 negative correlation in studied samples.

Table 3: Compositional correlation coefficients of studied samples.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
SiO_2	1								
Al_2O_3	0.86	1							
Fe_2O_3	0.84	0.92	1						
CaO	-0.99	-0.93	-0.89	1					
MgO	0.85	0.91	0.88	-0.92	1				
SO_3	-0.01	0.25	-0.03	-0.09	0.11	1			
Na_2O	0.51	0.52	0.42	-0.56	0.76	0.87	1		
K_2O	0.89	0.99	0.88	-0.94	0.9	0.35	0.55	1	
LOI	-0.99	-0.93	-0.89	1	-0.92	-0.09	-0.56	-0.94	1

5. Conclusion

The limestone chemical composition showed a high degree of homogeneity and pure limestone reserve, which is suitable to produce high quality Portland cement. The high CaO content and strong positive correlation with loss of ignition exhibited to calcite as a main mineral structure in the samples while other oxides showed depletion percentage except SiO_2 that presented a slightly higher ratio in some samples. The lime saturation factor, silica and aluminium ratios approved that Oligocene limestone in the studied area cannot be used for cement lonely without adding silica, aluminium and iron contents which most suitable material is the clay. The clay and gypsum of Lower-Fars formation where outcropped near the studied area is might suitable for the completion clinker raw material.

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