

Physical and Geochemical Assessment of Limestone of Amran Group in Arhab Area-North Sana'a for Industrial Uses

Ali M. Qaid¹, Naji Alqubati² and Ali M. Al-Hawbani³

1. Dep. of Civil Engineering technology, Al-Hajar (Qabitah) Community College/ Lahj, E-mail: technologycivil@gmail.com
2. Geological Survey and Mineral Resources Board, Sana'a, E-mail: najaen3209@gmail.com
3. Dep. of Geology & Environmental Sciences, Faculty of Applied Sciences, Tamar University, E-mail: alhwbanyly_82@gmail.com

Abstract

The present study is aimed to evaluate the potential of using limestone of Amran Group in Arhab area in different industries. X-ray diffraction pattern revealed that the limestone from Arhab is dominantly composed of calcite. The physical analysis showed that the bulk density is ranging from 2.4-2.88 g/cm³, water absorption 0.4-2.32%, void ratio 0.01- 0.06, whiteness 87.78-90.2 and specific surface area 3900-4650 cm².g⁻¹. Based on the XRF results, limestones are pure and dominantly composed of calcium carbonate 97.52-99.06%. The concentration of major oxide: CaO is ranging from 54.70 to 55.50 wt%, SiO₂ 0.20 - 0.80 wt%, Al₂O₃ 0.2 – 0.4 wt%, Fe₂O₃ 0.13-0.28wt% and MgO 0.40-0.60 wt%. The other oxides are present with low concentration. Loss on ignition value varied from 42.40 to 43.16 wt% and exhibits strong positive correlation with CaO which attributed to the highest concentration of CaCO₃. Geochemical data combined with Physical analyses data indicated that the limestone of Arhab area is suitable to be raw materials for various chemical industries such as paint, papers, ceramics, steel, pharmaceutical products and plastics after slightly modification of iron oxides in some special industries.

Keywords: Amran Group, limestone, Arhab, calcium carbonate, physical analysis, XRD, XRF

1. Introduction

The study area is located at about 10 km north of Sana'a, between 41573 5 - 434580 E and 1734960 - 1767993 N and covering about 650 km² (Figure 1). Yemen is endowed with huge natural resources of rocks and minerals. Limestone rocks are the most widely distributed rocks in Yemen. In this research limestones of Arhab area were elected because of their high purity (high CaCO₃ content), high physical and mechanical properties, huge of quantities, and low cost of quarrying the raw materials and transportation.

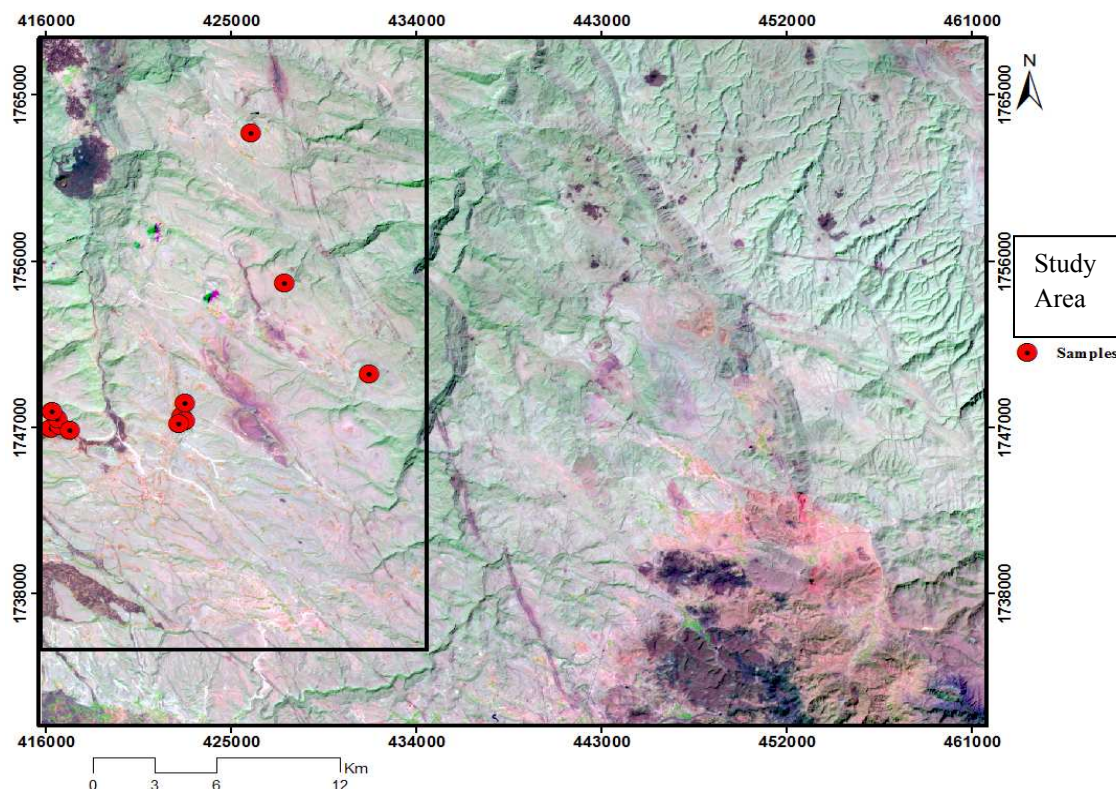


Figure 1. Satellite image (ASTER) shows study area

In Yemen, the applications of limestone are limited only to construction (aggregates, dimension, and ornamental stones) and industrial purposes (cement, lime, animal feed, agriculture). That is due to of the scarcity of applied studies of limestone rocks. Cement manufacturing is considered one of the most important industries that depends on limestone rock, there are 6 plants currently working. For other industries like paint, paper, medicine, etc., limestone is imported from outside the country.

Investigation of carbonate rocks in Yemen could be co-associated in the growth of a number of industrial and agricultural fields that lead to urban development. Limestone is commonly defined as a rock composed of calcium carbonate (CaCO₃) however, most limestone rocks contain significant amounts of magnesium, silicates, manganese, iron, titanium, aluminium, sodium, potassium, sulphur and phosphorus. They are formed from the dissolved organic matter of calcareous organisms and inorganic materials, which rely on their environmental deposits, and are then deposited and compacted into rock by lithification process over geological time [1], [2]. They can occur in various depositional environments, such as non-marine, shallow marine platforms and deep-sea environments [3]. The majority of carbonate sedimentary rocks are deposited from seawater and made by bioclastic accumulation of calcareous organisms [4], [5].

Numerous classification schemes for carbonate rocks have been proposed [6]–[13]. [6], described limestone rocks in terms of discrete grains (such as biogenic fragments) or "allochems" dispersed to a greater or lesser extent in a matrix of microcrystalline calcite ooze (micrite), or a crystalline cement

(spar). Although the wide distribution of calcium carbonate, white calcium carbonate is rare [14]. Iron and manganese oxides, organic matter, and pyrit are the main dark substances that affect the whiteness of calcium carbonate [14], [15]. With over a hundred industrial uses, limestone rock is probably the most useful of all industrial rocks and minerals. These include building (aggregates, rail ballast and dimension stone), mineral fillers (in paper, paint, plastic, rubber and pharmaceuticals), adhesives, abrasives, fertilizers, food additives, environmental applications (acidity neutralization, flue gas desulphurization, soil conditioning and stabilization), and production of cement and lime [16]–[23]. The widely used of calcium carbonate in industrial application because of their special properties: high purity, whiteness, ease of access, average particle size and distribution, etc. [24]–[28]. Limestones are usually used as inert fillers to complete a large variety of final properties without increasing the costs through replacing a more expensive material with an inexpensive one. Fillers also improve the specific characteristics such as hardness, brittleness, impact strength, compressive strength, softening point, fire resistance, surface texture, electrical conductivity, and so on [29], [30].

Figure 2 shows the world lime production in 2019 in million tons [31]. China ranked the first production in the world with the total amount of 300,000,000 tons and the second one is USA with 18,000,000 tons. [16], [17], [32], [33], classify the purity of limestone rocks according to carbonate concentration to: > 98.5% CaCO_3 very high purity, 97.0 to 98.5% high purity, 93.5 to 97.0% medium purity, 85.0 to 93.5% low purity and 50.0 to 85.0% impure (Table 1). Very high purity limestone is defined as carbonate rock that contains greater than 98.5% calcium carbonate (usually as calcite). The British geological survey (BGS) has used this classification in all major limestone resource work carried out in the UK and other parts of the world [34].

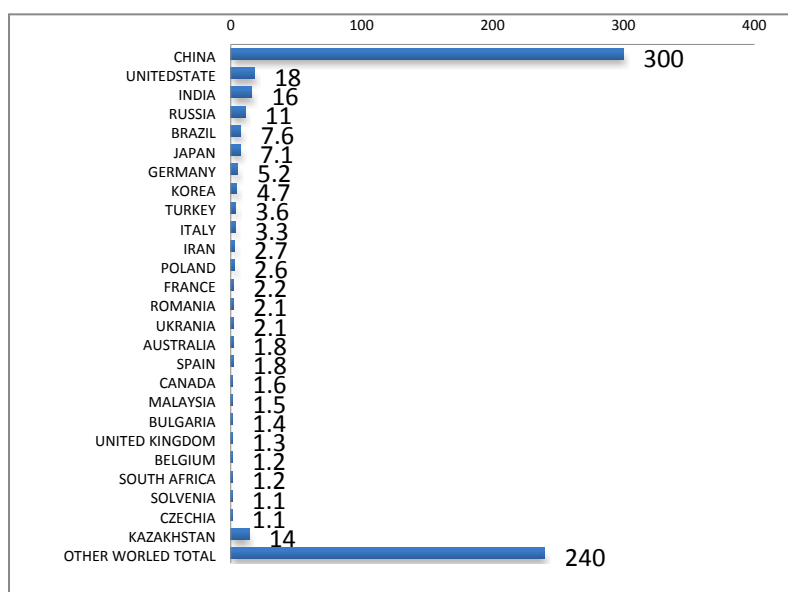


Figure 2. World lime production 2019 in million tons [31]

Table 1. Limestone purity classification according to [16], [17], [32], [33]

Purity classification	CaCO_3 wt%	CaO wt%	Possible industrial uses (grouped by minimum CaCO_3 specifications)
Very high purity	> 98.5	> 55.2	Steel, white glass (subjected to trace elements), rubber, plastics, paint
High purity	97.0-98.5	54.3-55.2	High purity 97.0–98.5 54.3–55.2 Iron, ceramic, Portland cement, whiting, chemical uses
Medium purity	93.5- 97.0	52.4-54.3	Medium purity 93.5–97.0 52.4–54.3 Paper (subject to color), animal feedstuffs (subjected to level
Low purity	85.0–93.5	47.6–52.4	Asphalt
Impure	< 85.0	< 47.6	Mineral wool, natural cements (subjected to silica/clay mineral ratio)

2. Geological Setting

Limestone of Arhab area belongs to Amran Group (Amran Limestone rock) which is one of the most widely distributed sedimentary cover unit in Yemen. Geological Survey and Mineral Resources Board-estimated the geological reserve of carbonate rocks in Yemen as: pure limestone 3.6 billion cubic meters, limestone rocks 10 billion cubic meters, magnizite 58 million Cubic meters and dolomite 3.4 billion Cubic meters [35]. Amran Group is a thick series of dominantly calcareous sediments, but locally including significant sequences of shale and evaporates; the carbonates are commonly massive, fissured rocks (Figure 3). The Amran Group consists mainly of limestone, marly limestone, shale beds and thick beds of evaporites (salt rocks and gypsum), characterized as the Sabatayn Formation in Shabwa “surface outcrops” and Safer “subsurface” areas [36]. The name of this group is derived from Amran town, which in the sequence is very clear (best exposed). [37], classified Amran limestone into two groups: the lower group (the Amran Group Middle-Upper Jurassic) and the upper group (the Sabatayn Group upper Jurassic-lower cretaceous). The maximum thickness of Amran Group may exceed 800 m. Amran limestone is a complex group, with two main depositional environment; a neritic environment (limestone and marly facies) and a shallow-water environment (evaporitic sequence). The tectonic effects on Amran Group at Arhab area is less comparing to other places covered by Amran Group. Weathering and erosion also showed less effect. The elevation of Arhab area ranges from 1735 to 2775 m above sea level (Figure 4).

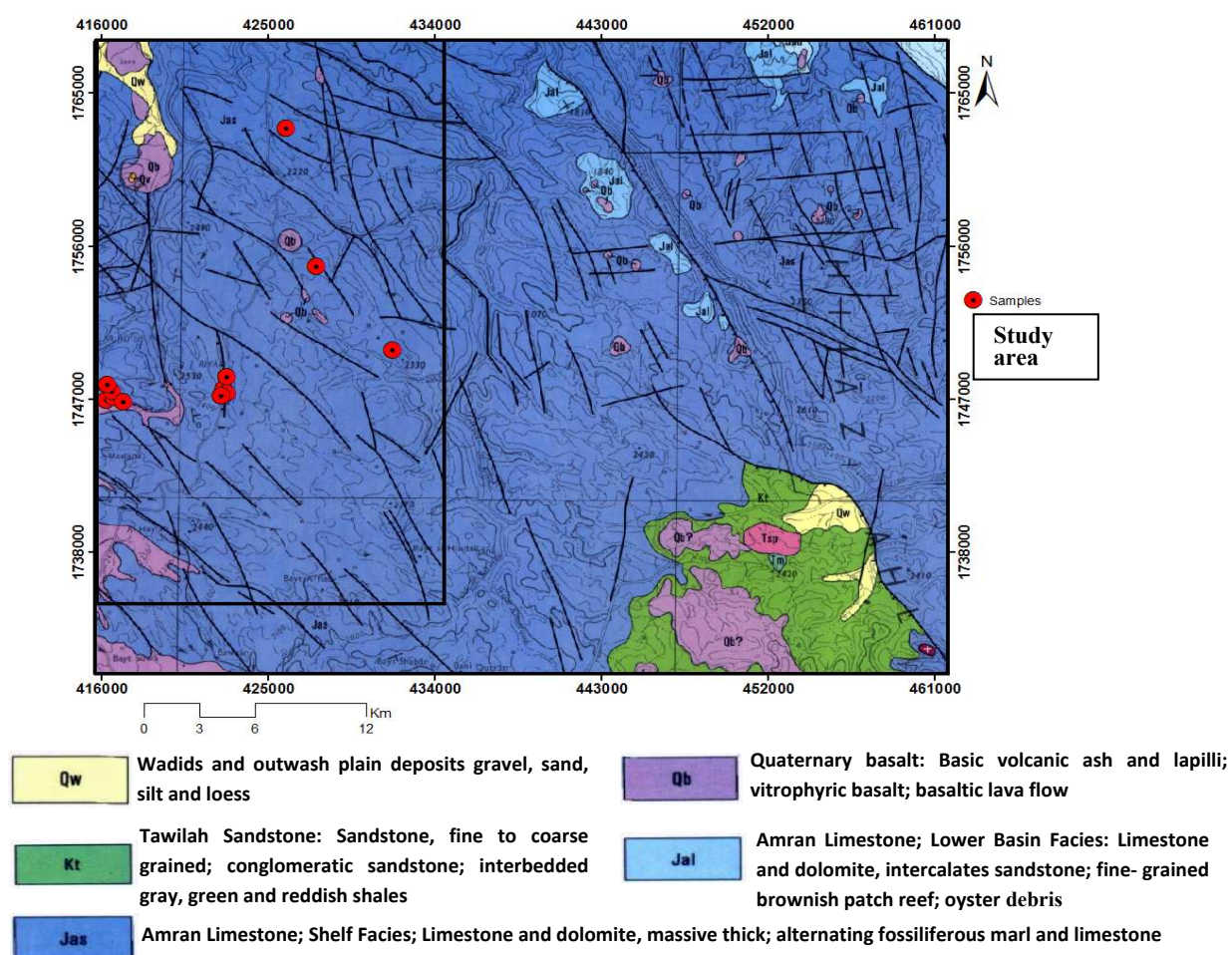


Figure 3. Geological map of study area [39]

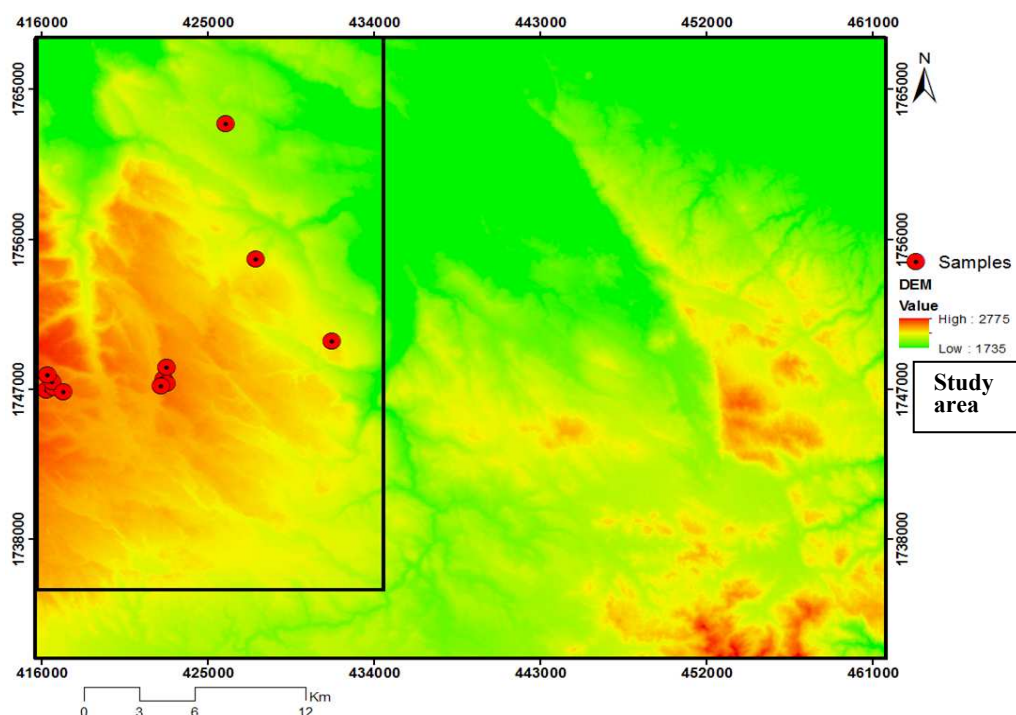


Figure 4. Digital elevation map showing the altitude of the study area

3. Materials and methods

Field work involved collection of representing rock samples with their locations using GPS and other geological information. X-ray diffraction and physical analysis were carried out at Saudi Geological Survey (SGS), Central lab of Geological Survey Board-Sana'a (GSMBR) and Faculty of Engineering, Sana'a University. Samples for XRD were prepared by grinding without treatment to very fine particles then testing using XD-2/ XD-3 Goniometer Type vertical: 00-2 θ scan: 0- θ scan high, single-phase 508Z, 220V, max. 60 kw, rated powder 3Kw. This determines the presence and purity of calcite and common impurities such as quartz, feldspars, clay minerals, pyrite and iron oxides etc. The physical analysis including surface area, bulk density, water absorption, whiteness and void ratio were carried out according to American Standard (ASTM). Chemically, whole rock geochemical analyses for major oxides of part of samples were carried out using X-Ray Fluorescence Spectrometer (XRFs) techniques, WDXRF 1Kw, S8 Tiger Pruker AXA, AG-RH tube at the central laboratories of GSMBR and the other samples were analyzed in SGS. The pH measurement is the measure of the acidity or alkalinity of a solution. The pH value was measured at room temperature using a pH meter.

4. Results and Discussion

The XRD analysis of studied samples of limestone from Arhab area indicates that the limestone is pure and composed mainly of calcite (Figure 5). The Physical test results showed that the whiteness value is ranging between 87.78-90.2. The slight decreasing of whiteness refers to the slightly increasing of dark minerals especially iron oxides content [28]. The bulk density ranges 2.4-2.88 g/cm³, water absorption 0.4-2.32% and void ratio 0.01-0.06. The specific surface area is ranging from 3900-4650 cm²/g (Table 2). The result of the major oxide composition revealed that limestone rock of Arhab is pure with high content of CaO which ranging from 54.70 to 55.50 wt% with an average of 55.22 wt%, accordingly the CaCO₃ content ranging from 97.52 Wt%-99.06 Wt%. The other oxides are present only with a small concentration (Table 2). SiO₂ content varies widely ranging from 0.20 to 0.80 wt%, which is contributed by quartz, Al₂O₃ 0.2-0.4 wt% and the total alkali value is regarded as

low ($\text{Na}_2\text{O} + \text{K}_2\text{O}$) 0.06-1.50wt%. The low levels of Al_2O_3 , K_2O , Na_2O , and SO_3 related to the decreasing spread of clays. MgO contents 0.4-0.6 wt%, the extremely low content indicates a weak dolomitization process of the limestone. The Fe_2O_3 content is ranging from 0.13 to 0.28 wt% (Table 3), and varies from place to place due to the distribution of tectonic discontinuity, bedding planes and clays. A slight increase in iron oxides could be related to surface sampling as iron oxides fill joints and bedding planes. The loss on ignition value ranging from 42.40 to 43.16 wt% with an average 42.88%, suggesting a high carbonate content for the limestone (Table 3). SiO_2 , Al_2O_3 and Fe_2O_3 contents show strong negative correlation with CaO (Table 3 and Figure 6 a,b,c). The negative correlation between CaO and SiO_2 , this is because of the fact that the CaO (from calcite) and SiO_2 (from quartz) are from two different mineral phases and they are not related [38]. LOI shows strong positive correlation with CaO content which may be due to the reason that LOI is contributed mainly by the carbonate content of calcite. It also shows negative correlation with SiO_2 (Figure 6d,e and Table 4). Plotting limestone samples on the $\text{Al}_2\text{O}_3 - \text{CaO} - (\text{MgO} + \text{FeO})$ ternary diagram shows that all samples are rather pure limestones with few Fe – Mg enrichment (Figure 7). The pH range of 8.3 to 8.6 indicates alkalinity (Table 2).

Field observation, geological map, and satellite image (Figure 1,3) showed that the tectonic discontinuity and cavities are less comparing to other limestone rocks in different places, that associated with decreasing of impurities minerals and increasing the purity. Comparing properties of limestone of Arhab area with [32], [33] classification Table 3 indicated that limestone marked in very high purity to medium purity categories and that meet the requirements of different chemical industries includes: paint, paper, pharmaceutical, ceramic, rubber, adhesive, sealants, agriculture and animal feed with only slightly modification in iron oxide concentrations for some industries.

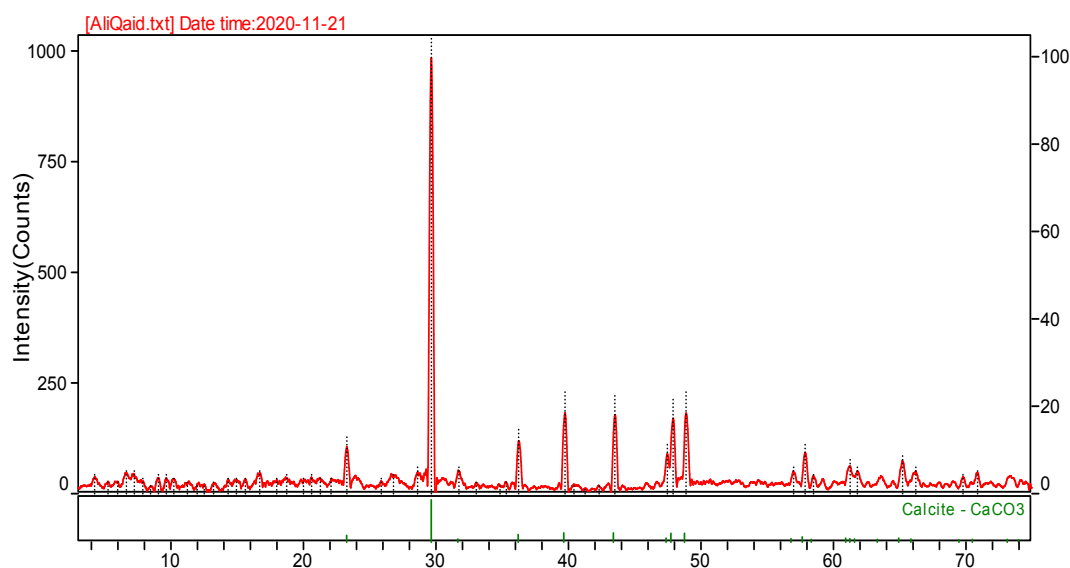


Figure 5. XRD analysis for Limestone from Arhab

Table 2. Average of physical and chemical analysis of limestone from Arhab

Whiteness	Bulk density g/cm^3	Water absorption%	Void ratio	pH	Surface area cm^2/g
87.78-90.2	2.4-2.88	0.4-2.32	0.01-0.06	8.3 - 8.6	3900-4650

Table 3. Chemical analysis of major oxides of limestone from Arhab

Sample no.	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	TiO ₂	P ₂ O ₅	K ₂ O+Na ₂ O	LOI	CaCO ₃
1	55.00	0.70	0.20	0.13	0.50	<0.05	0.01	0.06	42.72	98.16
2	55.33	0.40	0.36	0.26	0.42	<0.05	0.17	0.12	42.86	98.75
3	55.40	0.40	0.22	0.17	0.43	<0.05	0.09	0.10	42.98	98.88
4	54.70	0.80	0.40	0.26	0.60	<0.05	0.01	0.15	42.40	97.63
5	55.50	0.20	0.21	0.13	0.41	<0.05	0.05	0.11	43.06	99.06
6	55.50	0.25	0.26	0.15	0.44	<0.05	0.18	0.14	42.95	99.06
7	55.40	0.30	0.14	0.13	0.40	<0.05	0.11	0.10	43.16	98.88
8	55.20	0.43	0.21	0.19	0.41	<0.05	0.07	0.11	42.98	98.52
9	55.32	0.39	0.23	0.17	0.43	<0.05	0.10	0.10	42.95	98.73
10	55.32	0.35	0.26	0.26	0.53	<0.05	0.02	0.13	43.00	98.73
11	55.36	0.48	0.20	0.20	0.50	<0.05	0.03	0.10	43.08	98.81
12	55.12	0.65	0.29	0.21	0.51	<0.05	0.05	0.12	42.80	98.38
13	55.26	0.48	0.31	0.23	0.55	<0.05	0.04	0.15	42.80	98.63
14	54.64	0.69	0.32	0.28	0.60	<0.05	0.01	0.10	42.60	97.52
15	55.31	0.48	0.27	0.24	0.55	<0.05	0.04	0.14	42.92	98.72

Table 4. Correlation matrix of Pearson of major oxides of limestone from Arhab

Variables	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	P ₂ O ₅	K ₂ O+Na ₂ O	LOI
CaO	1.00							
SiO ₂	-0.90	1.00						
Al ₂ O ₃	-0.55	0.51	1.00					
Fe ₂ O ₃	-0.52	0.46	0.79	1.00				
MgO	-0.73	0.75	0.61	0.70	1.00			
P ₂ O ₅	0.58	-0.61	-0.08	-0.29	-0.71	1.00		
K ₂ O+Na ₂ O	0.05	-0.07	0.61	0.51	0.34	0.11	1.00	
LOI	0.89	-0.85	-0.78	-0.53	-0.73	0.41	-0.21	1.00

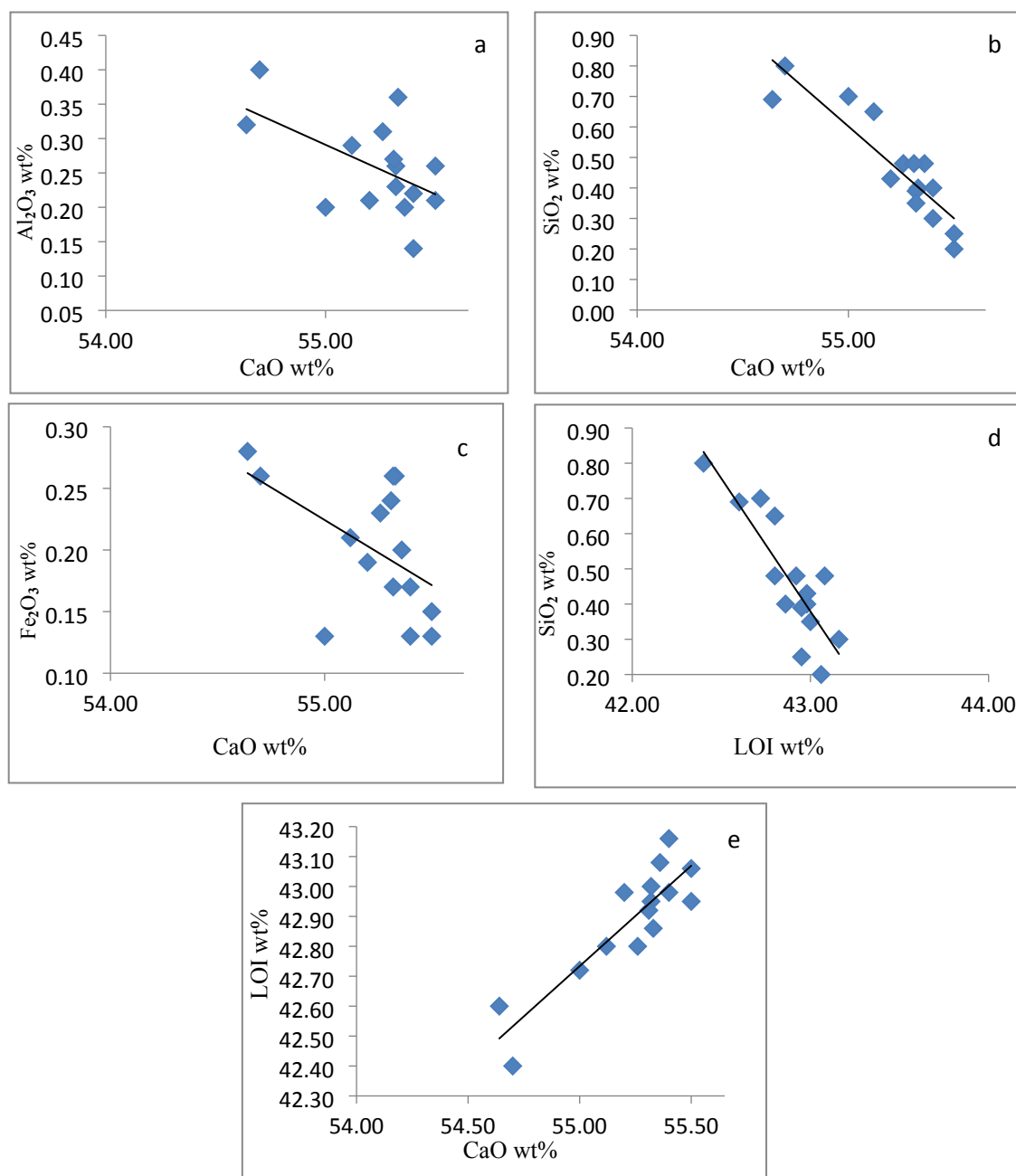


Figure 6. Diagrams of selected major oxides of limestone from Arhab (a) CaO wt% vs. Al_2O_3 wt%, (b) CaO wt% vs. SiO_2 wt% (c) CaO wt% vs. Fe_2O_3 wt% (d) LOI vs. SiO_2 wt% (e) CaO wt% vs. LOI%.

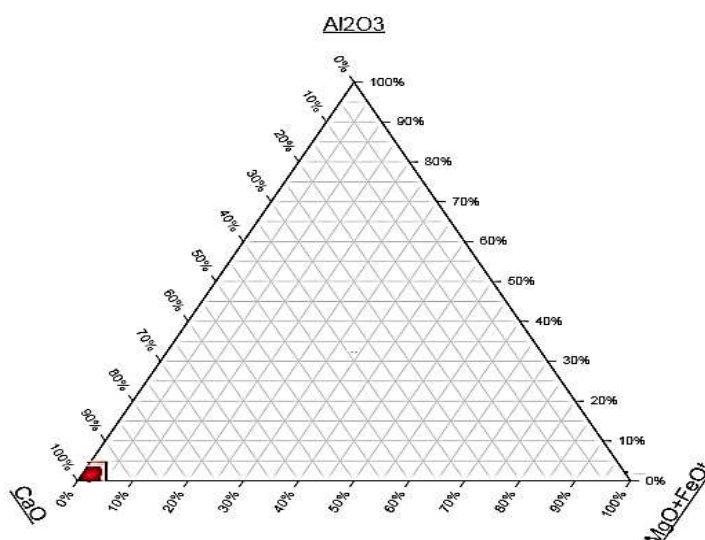


Figure 7. Chemical analyses of samples of limestone from Arhab area plotted on $\text{Al}_2\text{O}_3\text{--CaO--(MgO+FeO)}$ ternary diagram

5. Conclusions

XRD and chemical composition results showed that the limestone from Arhab area has very high - high purity, mainly composed of CaCO_3 with ranging from 97.52 to 99.06 wt% and the harmful oxides SiO_2 , Fe_2O_3 , MgO and Na_2O_3 are present with low concentration. The physical test results include whiteness, void ratio, water absorption, surface area plain and bulk density show high properties. In addition to chemical and physical specifications, there are many advantages of limestone from Arhab such as huge outcrop, can be mined open cast with low cost, and their position near the plants and markets.

Field observation, geological map, and satellite image revealed that the tectonic effects are less comparing to the same rock in other places. Based on the geochemical and physical properties, the limestone in Arhab can be used in a wide spectrum of industrial applications including plastic, glass made, steel, ceramic, paper, paints, animal feed, and production of lime, etc. with slightly modification for iron oxides in rare cases.

This is a preliminary study with limitations in industrial tests used (due to its currently unavailability). The study emphasized the interest in local limestone, which is available in economic quantities with very high quality that meet the requirements of many industries currently based on imported limestone. It also stressed the importance of local limestone in industrial uses rather than construction, which is extracted randomly.

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