SSRG International Journal of Geo-informatics and Geological Science
ISSN: 2393 – 9206 /doi:10.14445/23939206/IJGGS-V8I3P107

Volume 8 Issue 3, 52-63, Sep-Dec, 2021

Original article

Petrochemistry And Industrial Assessment of Limestone Occurrences In Tsando District, Yamaltu Deba Area, Gombe Northeastern Nigeria

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Received Date: 08 November 2021
Revised Date: 12 December 2021
Accepted Date: 25 December 2021

Abstract *- Tsando lies on the part of sheet 152/153SE Northeastern Nigeria. Field studies reveal three types of limestone: bioclastic, crystalline, and fossiliferous limestones. Fifteen (15) samples from thirteen (13) different locations were selected on the basis of their inherent characteristics for analysis. Petrographic analysis shows that the predominant mineral is calcite, with little quartz and ferroan calcite with fossils, and geochemical analysis shows that lime (CaO) ranges from 45.561-52.946wt% with an average of 48.570wt% and calcium carbonate ($CaCO_3$) has an average of 86.693wt%. Averages of other major oxides include: silica (SiO_2) 5.779wt%; alumina (Al_2O_3) 2.331wt%; iron oxide (Fe_2O_3) 1.857wt%; magnesium oxide (MgO) 0.607wt%; potassium oxide (K_2O) 0.059wt% and sodium oxide (Na_2O) 0.050wt%. The loss on ignition (LOI) and the limestone saturation factor (LSF) are averagely 38.785% and 270.858, respectively. These geochemical data suggest a moderately pure limestone that satisfies the chemical industrial specification for the production of portland cement.*

Keywords - *Industrial assessment, Limestone, Portland cement, Petrochemistry, Tando.*

I. INTRODUCTION

Limestone rocks are among the most essential of all the sedimentary rocks, and as industrial raw materials, they are essential for economic development. They are mineralogically composed mostly of calcite and can also contain dolomite and many noncarbonates such as glauconite, feldspar, and quartz as impurities [12]. Chemically, it consists of major and minor oxides, and variations in the composition of these chemical oxides and physical properties determine its varied industrial application. Although many occurrences of significant potential industrial mineral resources abound, most of them are not exploited because they do not meet industrial specifications; and in most cases, necessary evaluations and test work on their industrial suitability are unavailable. Such evaluations usually involve the determination of a wide range of inter-related properties, carried out with the knowledge of the requirements of consuming industries, confirmed by comparison with national or international specifications for each potential end-use [19]. Limestone ($CaCO_3$) is a versatile mineral, although its predominant use has been as a construction material [26]. Some of the uses of limestone include; dimension stones in building industries, in the manufacture of portland cement, in lime production, and as fillers in animal feed, paint, paper, plastic, and rubber industries. Many limestone deposits and occurrences are reported in different parts of Nigeria, but the purest limestone documented in Nigeria is that of Mfamosing with a CaO content of 52 to 56% and carbonate content of 99% [4]. Benue Trough, there are abundant, widely dispersed potential sources of limestone that would be sufficient for an industry that might develop around it. This research paper is aimed at studying the industrial quality of limestone occurrence in Tsando.

II.LOCATION OF THE STUDY AREA

The study area is located around Deba, the capital of YamaltuDeba local government area, Gombe State NE Nigeria. It forms part of sheet 152/153SE with coordinates: Longitude: 11°26′00″E - 11°32′00″ and Latitude: 10°10′00″N - 10°16′00″N. It covers a total area of 120.56sq. Km (Figure 1).

A. Geology of the Study Area

Kanawa member of Pindiga Formation is the dominant Formation in the study area, which overlies older Formation to the southeast (Figure 2). The unit is made up of grey shale interbedded with thin fossiliferous limestones. The lithounit ranges in thickness from 120m at Kumo to just a few meters of condensed limestone-dominated beds in the Dumbulwa hills. [31]first described shale-limestone beds of this area as the Kanawa Member of Pindiga Formation. The limestone in the area occurs as both crystalline and fossiliferous. The crystalline limestone is composed of calcite, quartz, dolomite, and subordinate plagioclase, and microcline, while the fossiliferous type consists of calcite and dolomite [11].

Geological field mapping of the study area was conducted to locate limestone occurrences, identify and appraise the lithology and macrofossils associated with the limestone. Thirty-four fresh samples of all the litho units and fossils were collected systematically from exposures within streams at twenty-two different locations in the study area. The location (with coordinates), elevation (in meters), the nature of the samples, description, and group of the samples, and sample identity number were assigned to each. Among the samples collected, field studies reveal three groups of limestone: bioclastic, crystalline, and fossiliferous limestone. Bioclastic limestone was encountered in nine different locations, has broken pieces of limestone, and is highly consolidated with brownish and partly grayish material (plate I). The sample identity numbers include BL1 (Loc 8), BL2 and BL3(Loc 9), BL4 (Loc 11), BL5(Loc 13), BL6 (Loc 14), BL7 (Loc15), BL8, and BL9 (Loc 16) BL10 (Loc 19) and BL11 (loc 21).

Crystalline limestone occurs in five different locations; it is brownish with a gray tinge color and is non-fossiliferous and crystalline in nature (plate II). However, two of the samples (CL3 Loc 18 and CL5 Loc 22) are petrified, characterized by vertical bands. At the same time, the other three samples (CL1 Loc 10, CL2, and CL4 Loc 22)are non-fossiliferous crystalline limestone.

Fossiliferous limestone (Plate III) was encountered in two different locations: loc 3(FL1 and FL2 and Loc 20 (FL3 and FL4).

The limestones occur associated with shale in five different locations (vizLoc 2,9,11,12 and 21) and fine-grained sandstones unit (of the Yolde Formation) in four locations (vizLoc 2,4,5 and 6). Few lenses of gypsum were observed with sediments in locations 11 and 18, and ammonite fossils were encountered in locations 10 and 15. Fifteen representative samples, comprising ten (10) bioclastic types, two (2) crystalline types, and three (3) fossiliferous types, from thirteen identified occurrences, taking into consideration observed morphological differences, were used for analysis. Figure 2 is the map of the study area showing the geology and samples locations.

B. Geochemical Analysis

Determinations of major oxides composition of fifteen samples wereundertakenatAshakaCement Company Laboratory Gombe using X-ray Fluorescence Spectrometry (XRF). The samples were grounded by means of pulverized agate mortar to less than 200 mesh size. Fused beads were produced by regrinding the samples to <60µm, drying the raw ground sample, and retaining not more than 0.5% moisture. Two grams (2g) of the dried samples were weighed into the crucible and ignited in a furnace at 950°C. The ignited samples were carefully crushed inside the crucible. Using a spatula from the ignited samples, 1g was carefully weighed into a clean, dry fusion crucible, and 8.0g of flux was added and mixed thoroughly. Thereafter, 1.0ml of LiBr solution was added to the mixed sample. The crucible was then placed on the fusion machine holder, and the "recipe F" button was selected. The start button was pressed and held for 10 seconds, and when released, the machine was automatically set for the fusion process to commence. At the end of the process, the machine indicated "FUSION COMPLETE". Glass disk was used in removing and transferring the bead into the pellet cup. Boric acid and dilatants were mixed to get a homogenous mixture, and pellets were made. These pellets were inserted into a spectrometer for analyses of the elements.

For the loss of ignition (LOI) test, the furnace was set at 970°C, followed by placing an empty crucible on the weighing balance and the reading reset to zero. One gram of the samples was weighed directly into the crucible; then, the crucible with the sample was removed, and the balance machine reset to zero. This is followed by re-weighing the crucible with the sample as M1, followed by carefully placing the crucible and sample in the middle part of the furnace using the crucible tong. The furnace was closed, and the samples were allowed to remain in the furnace for 30 minutes. The samples were then removed and placed into desiccators, and covered to cool. The crucible plus ignited samples were reweighted as M2. Loss on Ignition (LOI) is calculated as:

$$LOI = (M1-M2) \times 100$$

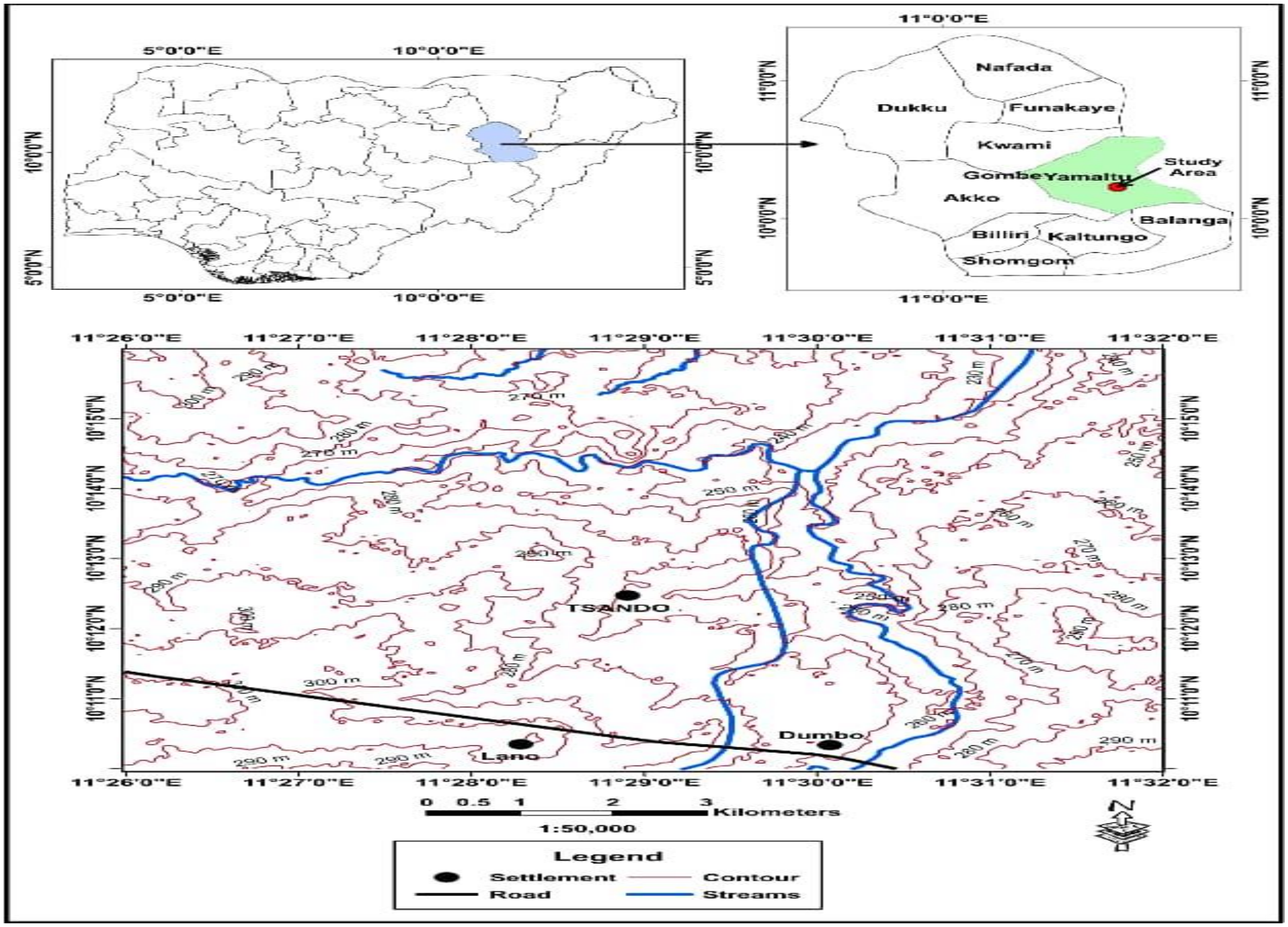

Figure 1. Location Map of the Study Area [13].

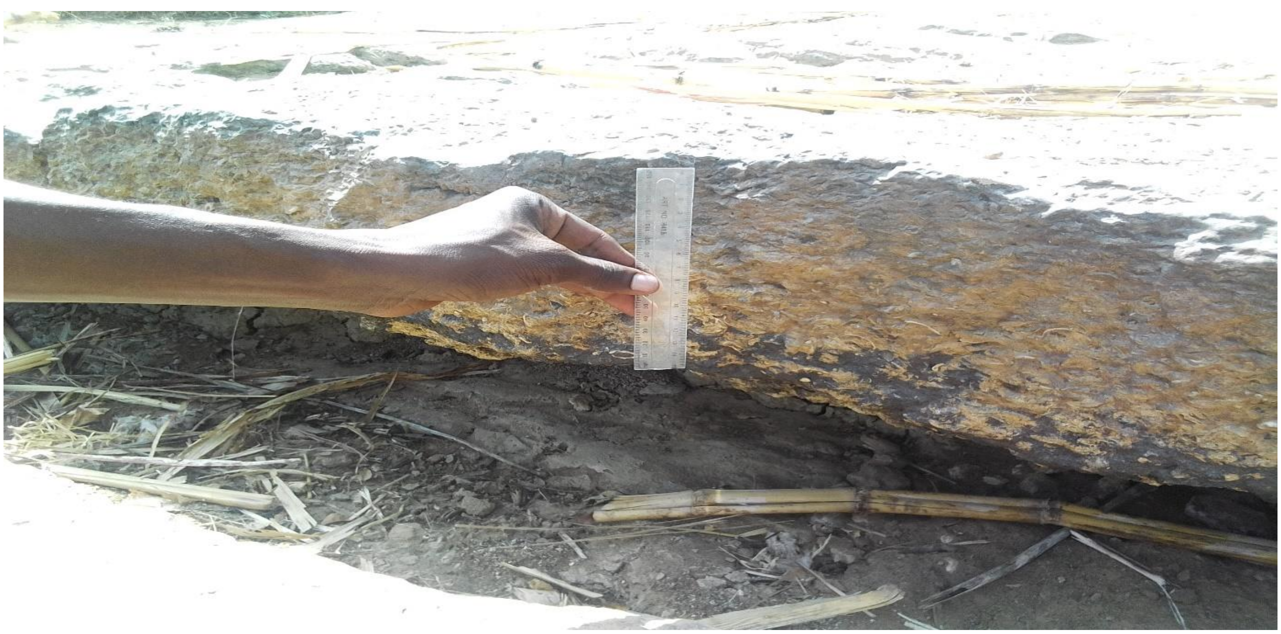

Plate I, Bioclastic limestone in Dumbo stream (10°12ʹ42.5ʹʹN 11°29ʹ38.2ʹʹE).

Plate II, Crystalline limestone in Dumbo stream(10°14'36.3''N 11°28'07.5''E).

Plate III, Fossiliferous limestone in Dumbo stream(10°13'40.9''N 11°29'39.6''E).

III. PETROGRAPHIC ANALYSIS

Thin sections of fifteen samples were produced at the Geology Department of the ModibboAdama University of Technology Yola. The analysis revealed calcite as the dominant mineral found in the studied slides, followed by quartz (mainly detrital) in five slides, while ferroan calcite was observed in two slides. Fossils like brachiopod, bivalves, echinoderm, gastropod, and ostracods were also identified (Table 1 and Plate IV). The presence of ferroan calcite in samples affects the color of the limestone, thereby making it unsuitable for use in glass, paint, and rubber industries.

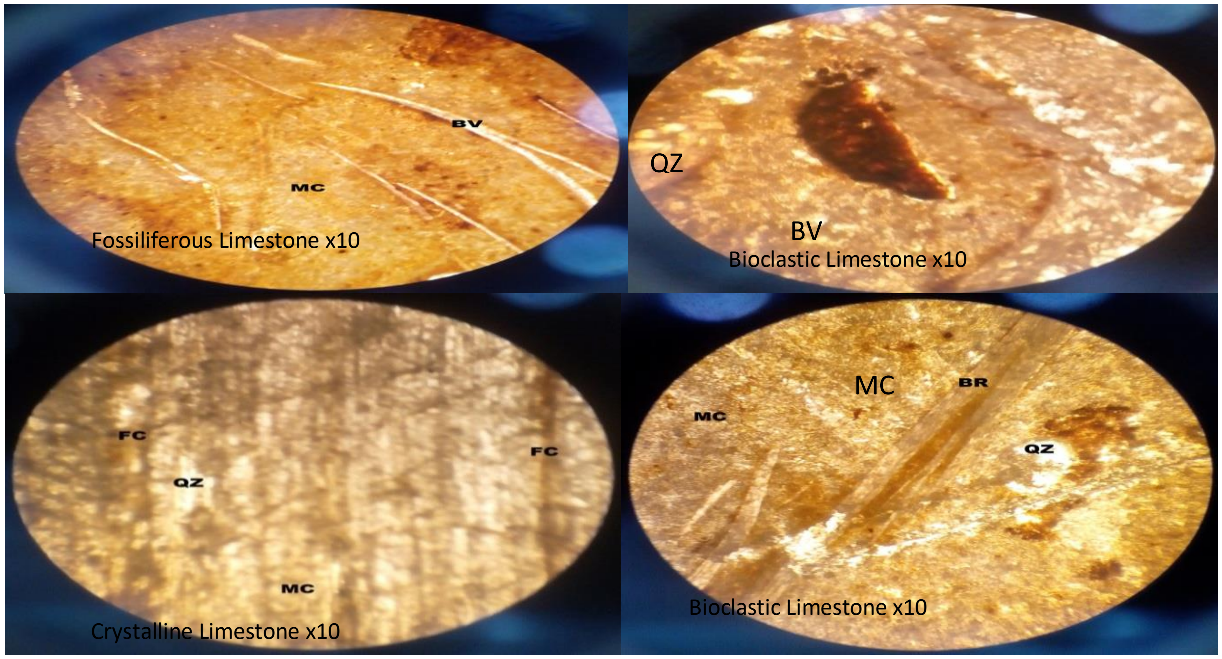

Plate IV. photomicrographs of the studied limestones. BV: Bivalve BR: Brachiopod FC: Ferroan calcite MC: Micrite QZ: Quartz

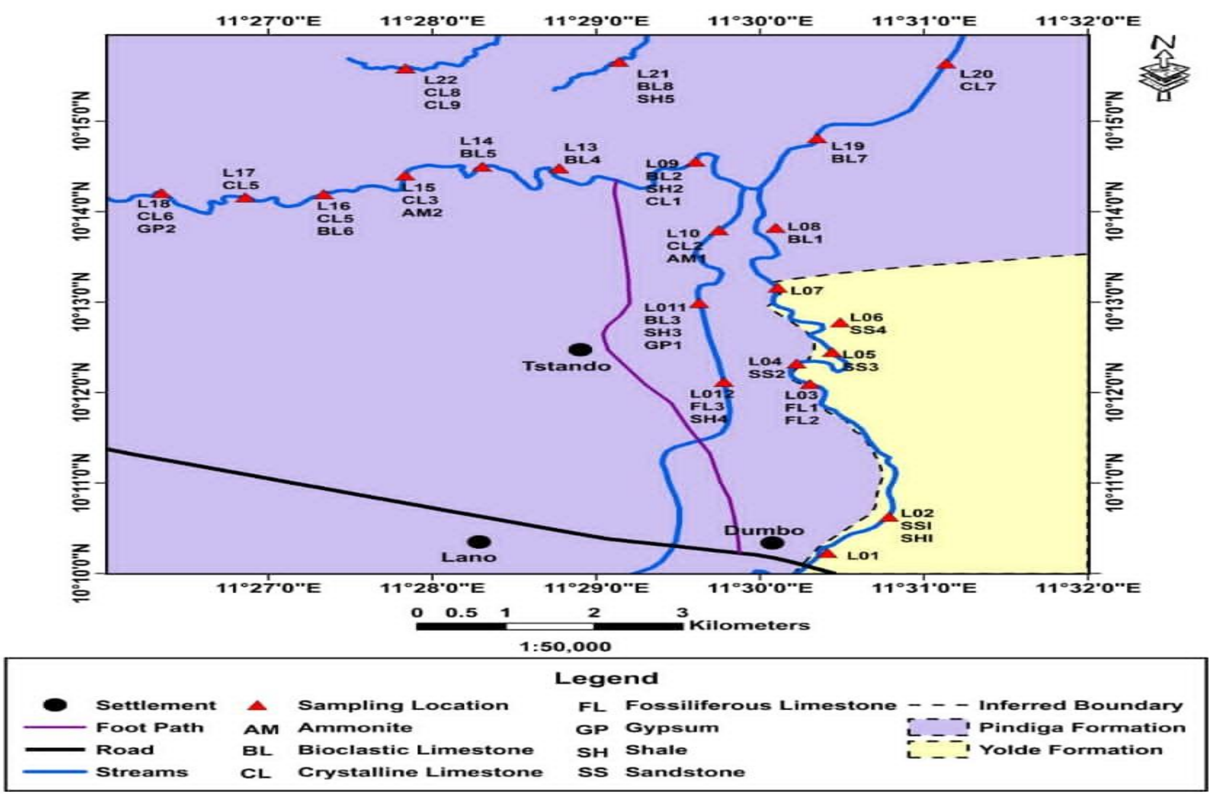

Figure 2. Geological Map of the Study Area (modified from [13])

Table 1, Mineralogical Composition of the Studied Limestone

S/No.	Type of Limestone	Mineral Composition	No of Samples	Samples I.D No.
1	Bioclastic	Micrite and calcite (+fossils)	10	L8BL1, L9BL2 and BL3, L11BL4, L14BL6, L15BL7, L16BL8 and BL9, L19BL10 and L21BL11
2	Crystalline	Calcite, micrite, quartz, and ferroan calcite	2	L10CL2, and L22CL5
3	Fossiliferrous	Micrite, quartz and ferroan calcite (+fossils)	3	L3FL2 L12FL3 and L20FL4

IV. GEOCHEMICAL RESULT

Results of the geochemical analysis of fifteen representative samples are summarized in Table 2. The concentration of major oxides from the analysis shows that lime (CaO) ranges from 45.561-52.946wt% with an average of 48.57wt0% and calcium carbonate ($CaCO_3$) has an average of 86.693wt%. The averages of silica (SiO_2) alumina (Al_2O_3), iron oxide (Fe_2O_3), and magnesium oxide (MgO) are 5.779wt%; 2.331wt%; 1.857wt% 0.607wt% respectively.Averages of other minor oxides includes, SO_3(0.092wt%), K_2O(0.059wt%), Na_2O (0.050wt%), P_2O_5(0.179wt%), Mn_2O_3(0.238wt%), TiO_2(0.123wt%) and Cr_2O_3(0.005wt%). The Loss on Ignition (LOI) ranges from 36.395-41.857%, with an average of 38.785%. The limestone saturation factor (LSF) is an averagely of 270.858.

V. RESULT IN DISCUSSION

The observed chemical parameters were used to classify the studied limestone on the basis of impurities (of [23]), carbonate content (of [28]), and Purity (of [7]).

Going by[23] classification schemes on the basis of principal impurities, the limestone in the Tsando area with average silica content of 5.779% is argillaceous, but according to [28], the studied limestone with 89.758% average proportion of Ca/Mg and 0.013 proportion of Mg/Ca are pure limestone. However, when classified on the basis of purity, according to [7], the tsando limestone with an average of 86.69% $CaCO_3$ and 48.57% CaO is of low purity (Table 3).

VI. BIVARIANTS PLOTS

Regression analysis generates an equation to describe the statistical relationship between one or more predictors and the response variable and to predict new observations. Certain indicator elements are adjudged to be in abundance if their associated mineral or major element is in abundance or suitable chemical condition for its occurrence is high [21]. This means that the concentration of any element of interest is based on its symbiotic relationship with the concentration of other related compositions in the analyzed material. Therefore, the content of such elements of interest is not measured directly but is relatively approximated or determined on the basis of the abundance or depletion of those constituents related to them. Regression analysis was used to investigate the relationship between the abundance of certain predictor elements and the concentration of a response variable.

A fitted line plot was made to study the relationship between the response and predictor oxides, and then a regression model (linear, quadratic, or cubic) that best describes the relationship between them was chosen using the formula:

S= An estimate of the standard deviation of the error in a model.

R^2 (R-sq) =Coefficient of determination; indicates how much variation in the response is explained by the model. The higher the $R^{2,}$ the better the model fits the data.

Adjusted R2(R adj) = Accounts for the number of predictors in your model and is useful for comparing models with different numbers of predictors.

The concentration of lime was plotted against silica (SiO_2), aluminum oxide (Al_2O_3), iron III oxide (Fe_2O_3), and magnesium oxide (MgO), all of which indicated negative correlations (Figure 2a-d). The negative correlation of calcium oxide with iron III oxide and magnesium oxide signifies an increase in the leaching of CaO by solution in the environment [8]. This, in turn, increases the purity of the limestone.

Table 2. Results of the Major Oxides in wt% of the Analyzed Samples

Samples ID	Oxides (wt%)													Total
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	TiO_2	Cr_2O_3	$CaCO_3$ Ratio	LOI	Ca/Mg	Mg/Ca	LSF	
L3 FL2	7.272	2.672	2.310	46.436	0.661	0.133	0.137	0.005	82.879	37.170	70.25	0.014	185.632	97.438
L8 BL1	4.556	1.879	2.142	50.016	0.381	0.066	0.129	0.004	89.268	39.672	131.27	0.007	305.600	99.305
L9 BL2	8.338	3.762	1.842	45.798	0.761	0.001	0.160	0.006	81.741	36.780	60.18	0.017	158.024	97.748
L9 BL3	8.063	2.896	2.353	45.561	0.581	0.177	0.146	0.005	81.381	36.395	78.42	0.013	165.535	96.395
L10 CL1	3.388	1.324	1.643	51.356	0.673	0.117	0.117	0.005	91.660	41.044	76.31	0.013	423.873	100.493
L11 BL4	7.824	2.850	2.290	45.739	0.751	0.168	0.141	0.006	81.635	36.722	60.90	0.016	170.922	97.339
L12 FL3	4.133	1.588	1.912	50.370	0.651	0.137	0.121	0.004	89.912	40.252	77.37	0.013	342.928	99.634
L14 BL6	4.133	2.626	1.375	48.793	0.628	0.084	0.135	0.005	87.086	38.984	77.69	0.013	234.475	96.993
L15 BL7	4.523	1.862	1.227	50.694	0.777	0.078	0.123	0.005	90.478	40.639	65.24	0.015	323.760	100.435
L16 BL8	5.947	2.300	2.018	48.011	0.575	0.167	0.138	0.005	85.689	38.311	83.49	0.012	232.148	98.009
L16 BL9	6.576	2.824	1.670	47.979	0.789	0.042	0.140	0.005	85.633	38.522	60.81	0.016	210.159	98.861
L19 BL10	8.491	3.099	2.226	45.900	0.478	0.086	0.145	0.007	81.922	36.548	96.03	0.010	159.937	97.615
L20 FL4	7.327	2.984	1.691	47.040	0.785	0.036	0.148	0.005	83.958	37.780	59.92	0.017	187.135	98.219
L21 BL11	3.275	1.242	1.604	51.916	0.330	0.069	0.110	0.005	92.659	41.107	157.32	0.006	444.490	100.142
L22 CL5	2.842	1.061	1.546	52.946	0.277	0.018	0.106	0.005	94.498	41.857	191.14	0.005	518.259	101.160
Range	2.842-8.491	1.061-3.762	1.227-2.353	45.561-52.946	0.277-0.785	0.001-0.177	0.106-0.160	0.004-0.007	81.381-94.498	36.395-41.857	59.92-191.14	0.005-0.017	158.024-518.259	
Average	5.779	2.331	1.857	48.570	0.607	0.092	0.123	0.005	86.693	38.785	89.76	0.013	270.858	

Table 3, Classification of TsandoLimestone and Comparison with other Authors

Chemical Parameter	Range/%	Description	The average value in Tsando limestone
Average proportion Ca/Mg [28]	100.0-39.0	Pure limestone	89.76
	39.0-12.3	Magnesian limestone	-
	12.3-1.41	Dolomitic limestone	-
Mutual proportion Mg/Ca [28]	0.00-0.03	Pure limestone	0.013
	0.03-0.08	Magnesian limestone	-
	0.08-0.18	Dolomitic limestone	-
Percentage $CaCO_3$ [7]	>98.5	Very high purity	-
	97.0-98.5	High purity	-
	93.5-97.0	Medium purity	-
	85.0-93.0	Low purity	86.69
	<85.0	Impure	-
Percentage CaO[7]	>55.2	Very high purity	-
	54.3-55.2	High purity	-
	52.4-54.3	Medium purity	-
	47.6-52.4	Low purity	48.57
	<47.6	Impure	-
Percentage SiO_2 [23]			5.78

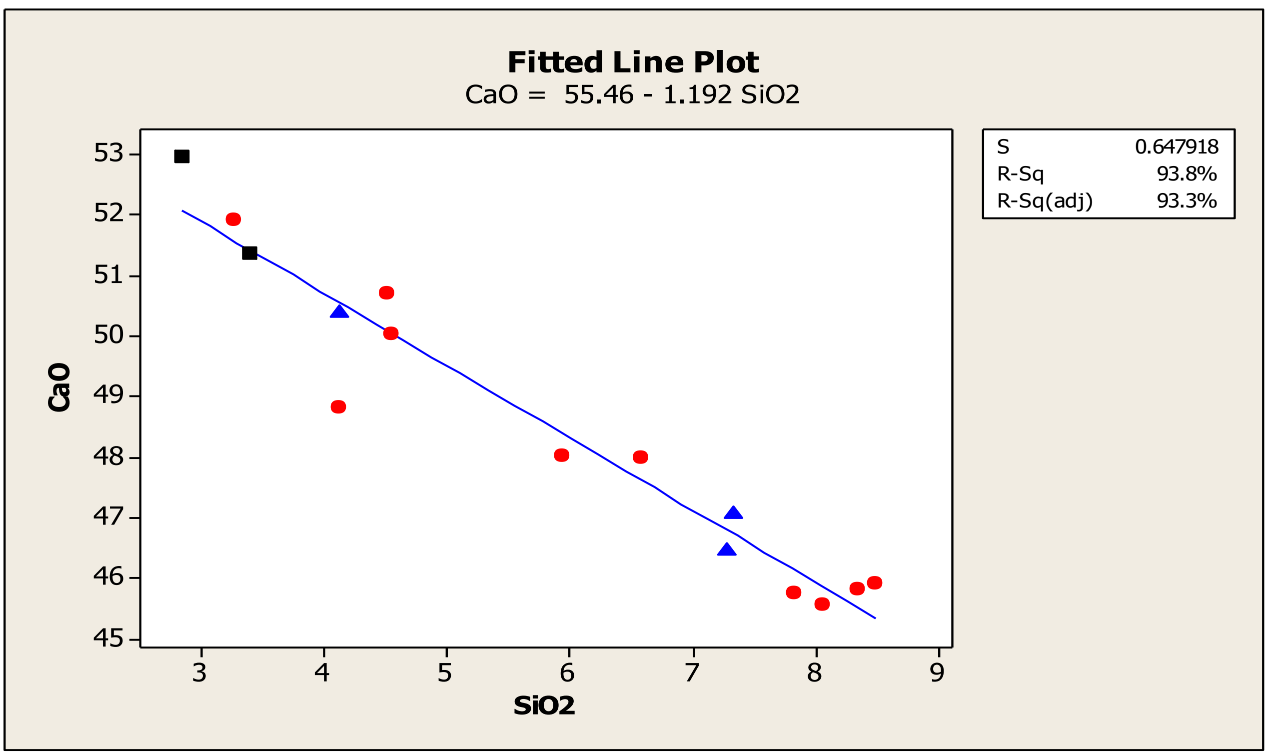

Figure 2a, Fitted line plot of CaO against SiO_2 (Negative correlation).

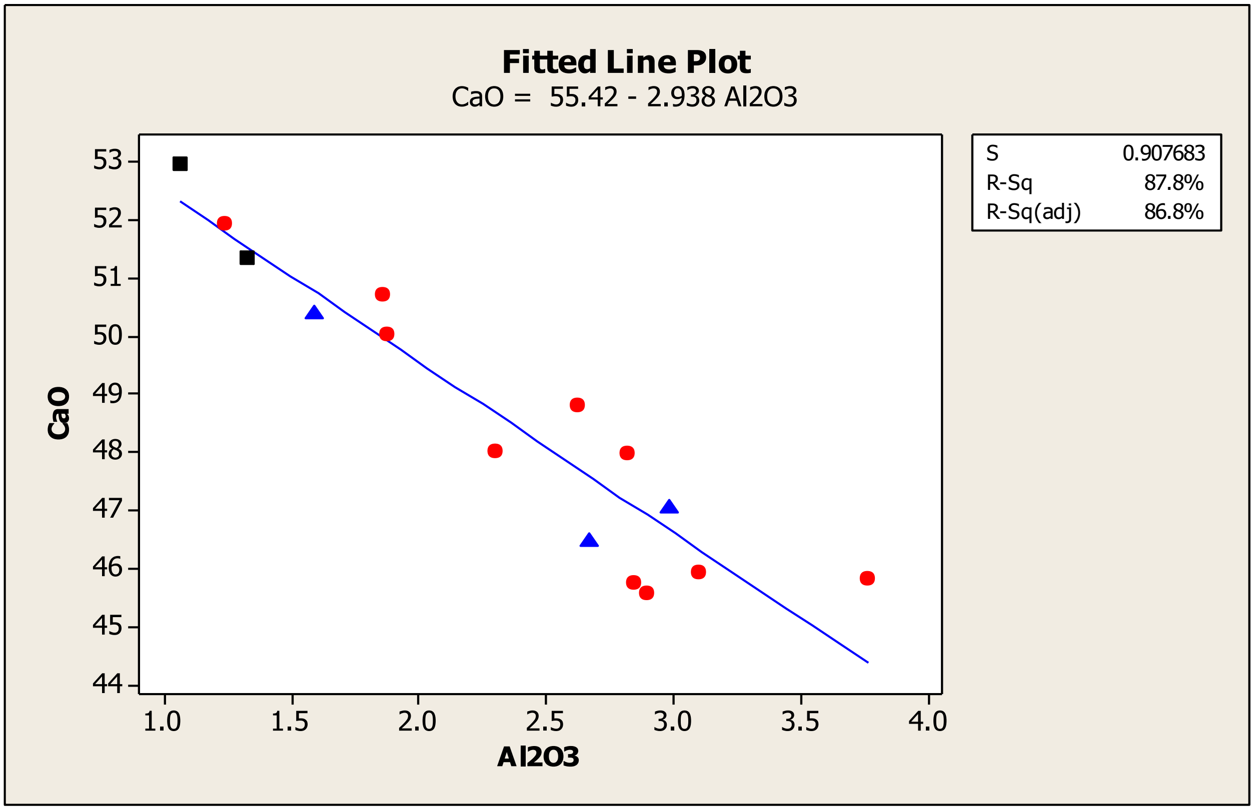

Figure 2b, Fitted line plot of CaO against Al_2O_3 (Negative correlation)

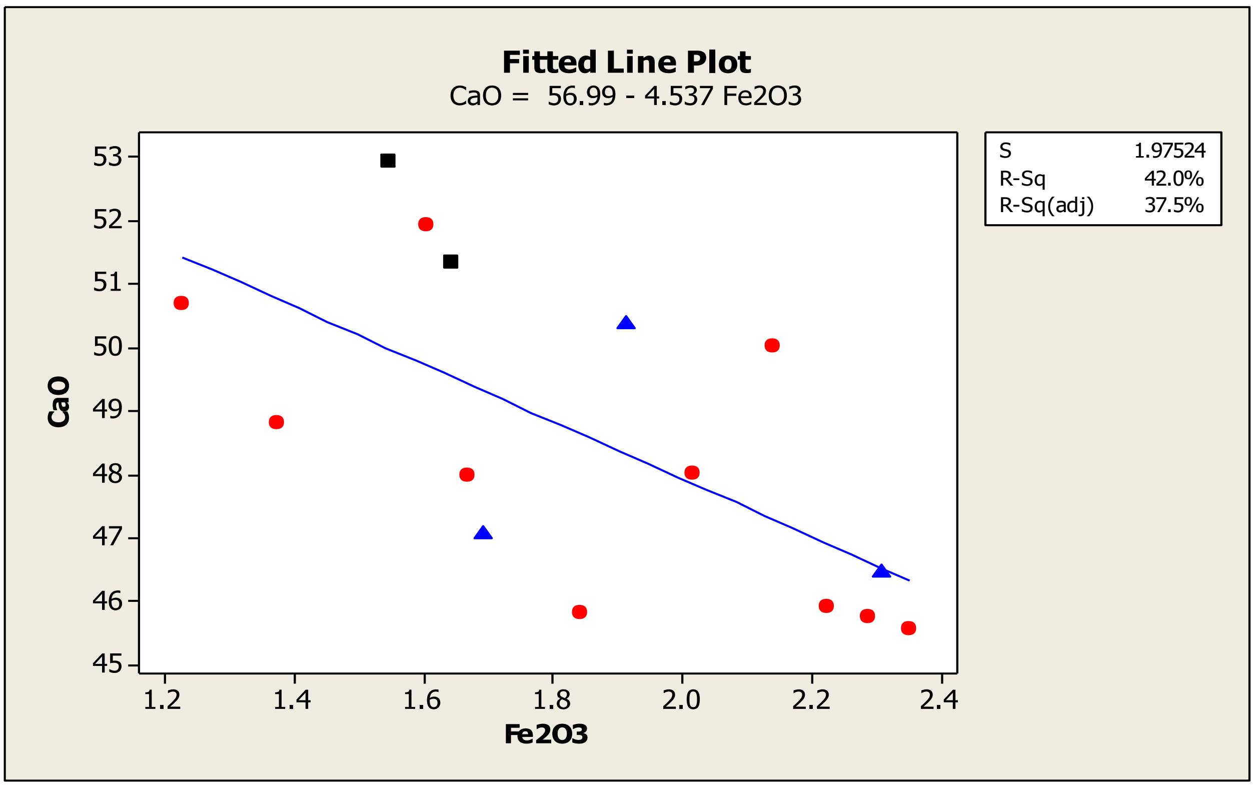

Figure 2c, Fitted line plot of CaO against Fe_2O_3 (Negative correlation)

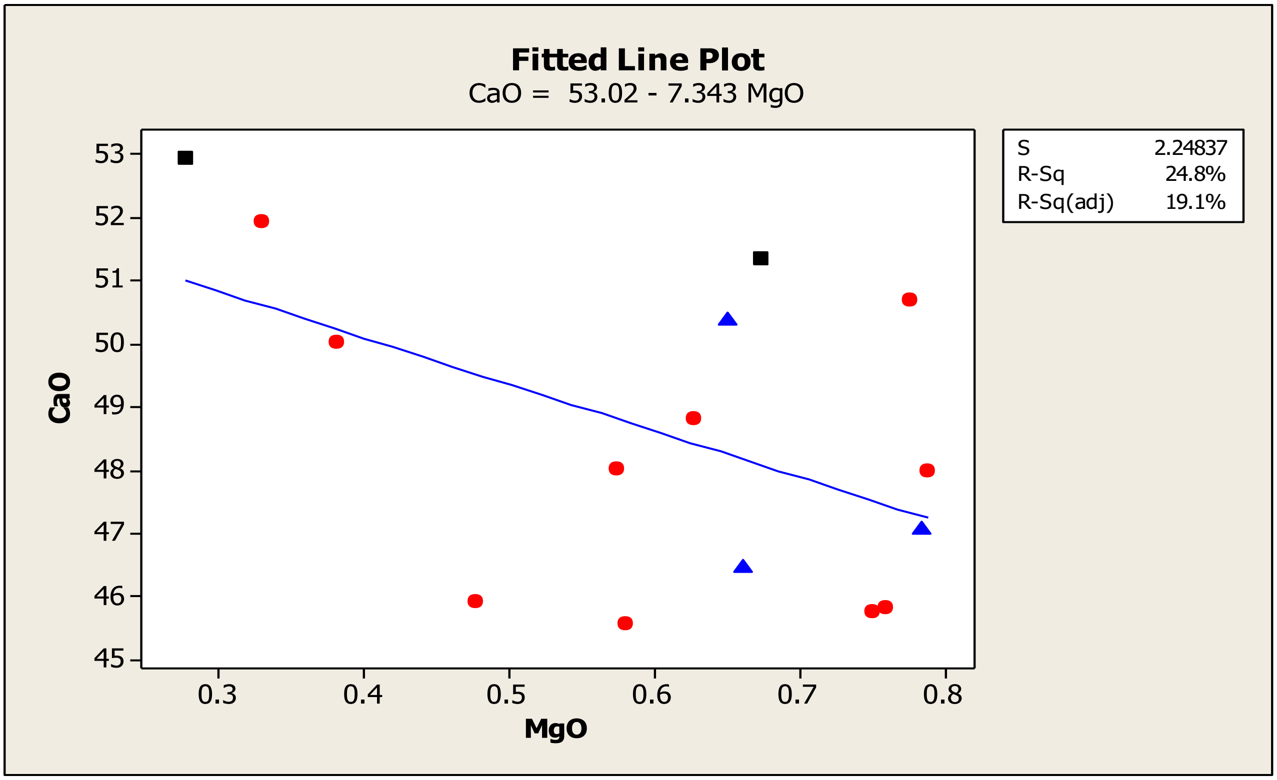

Figure 2, Fitted line plot of CaO against MgO (Negative correlation)

Key

Bioclastic limestone
Crystalline limestone
Fossiliferous limestone

A geochemical map of concentrations of the calcium oxides (CaO) in the study area reveals that the purity would be higher in the northcentral portion (Figure 3).

VII. INDUSTRIAL APPLICATIONS

Cement production, according to Oates (1996), requires 44.4% of CaO; 14.3% of SiO_2; 3.0% of Al_2O_3; and LOI of 35.9% (Oates, 1991). Attempts at assessing the industrial application of the Tsando limestone reveal that its low SiO_2 (5.78) and Al_2O_3 (2.3) will be compensated from sand and shale, respectively, but its CaO (48.570%) and LOI (38.785%) makes it suitable for cement production.

VIII. CONCLUSION

The limestone of the study area is associated with Kanawa shale members of the Pindiga Formation, and geological mapping reveals three different types of limestone: bioclastic, crystalline, and fossiliferous limestone. The petrographic analysis identifies calcite (as the dominant mineral), quartz, ferroan calcite as a mineralogical constituents with fossils. Geochemical analyses suggest that the limestone is moderately pure with a lime (CaO) average composition of 48.570wt%. The limestone falls below the permissible industrial specification for use in glass, paint, and rubber, and sugar industries but can satisfy the chemical industrial specification for the production of Portland cement upon adjusting silica and alumina content by the addition of sand and feldspar or shale.

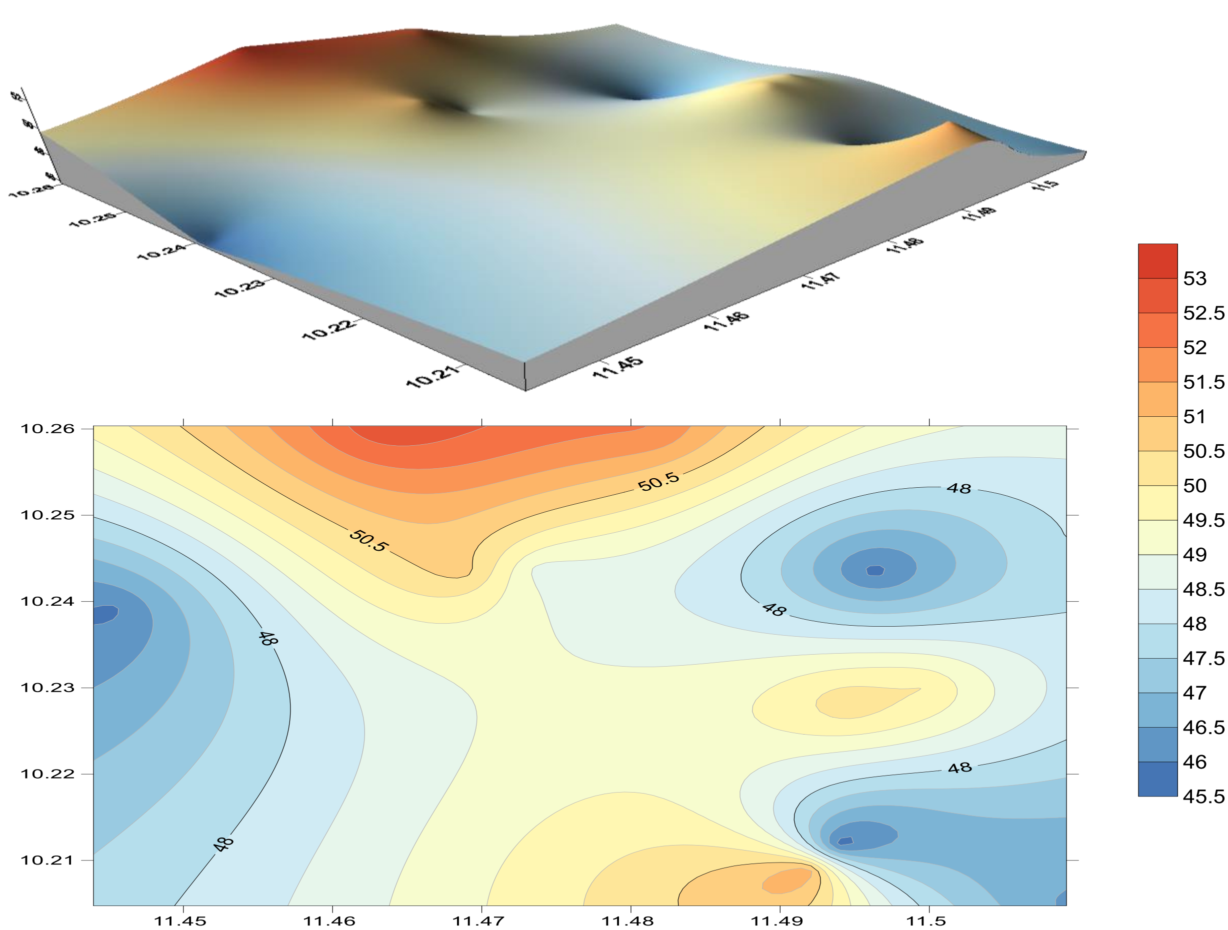

Figure 3. Geochemical Map Showing Distribution of CaO.

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