Nigerian Research Journal of Chemical Sciences (ISSN: 2682-6054)
Volume 15, Issue 2, 2026

Chemical Composition and Quality Assessment of Limestone from Selected Mining Points at Wamakko, Sokoto State, Nigeria

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Accepted: *October 13, 2026* **Published Online***: October 16, 2026*

ABSTRACT

Limestone is the principal calcium-bearing raw material used in Portland cement manufacture, and its chemical variability can influence raw-mix proportioning and clinker quality. This study assessed the chemical composition and quality of limestone obtained from two selected mining points, MP-A and MP-B, at Wamakko, Sokoto State, Nigeria. Calcium carbonate content was determined by acid dissolution and back-titration, while major and minor oxide compositions were determined by X-ray fluorescence (XRF) spectroscopy. The $CaCO_3$ contents were 86.64% and 59.42% for MP-A and MP-B, respectively. XRF analysis gave CaO contents of 72.1425 ± 0.2327% and 34.6685 ± 0.2536%, respectively. MP-A contained 12.0221 ± 0.0764% SiO_2, 4.0053 ± 0.0177% Al_2O_3 and 1.4255 ± 0.0077% Fe_2O_3, whereas MP-B contained 20.2224 ± 0.0428% SiO_2, 5.8059 ± 0.0209% Al_2O_3 and 3.3817 ± 0.0062% Fe_2O_3. The corresponding loss on ignition values were 12.78 and 32.41%. The XRF-derived silica modulus, alumina modulus and lime saturation factor were 2.21, 2.81 and 183.13 for MP-A and 2.20, 1.72 and 52.70 for MP-B, respectively. The results demonstrate substantial chemical variability between the investigated mining points and emphasize the importance of systematic limestone characterization and appropriate raw-material blending for consistent cement production.

Keywords Cement, limestone, quarry, Wamakko, X-ray fluorescence.

INTRODUCTION

Limestone is one of the most important raw materials in Portland cement manufacture because it supplies the calcium required for the formation of the principal clinker phases. During clinker production, calcium carbonate decomposes at elevated temperature to produce calcium oxide, which subsequently reacts with silica, alumina and iron-bearing components to form calcium

silicates, calcium aluminates and calcium aluminoferrite phases [1,2]. Consequently, the chemical composition of limestone has a direct influence on the chemistry of the cement raw mix and the subsequent clinker formation process. The quality of a limestone deposit is not determined solely by its calcium carbonate content. Variations in SiO_2, Al_2O_3, Fe_2O_3, MgO, alkali oxides, sulphur-bearing compounds and other minor constituents may substantially affect raw-mix proportioning and kiln operation [3,4].

X-ray fluorescence spectroscopy is widely used for determining the major oxide composition of cement raw materials because it provides rapid and quantitative information on the principal oxides used in cement chemistry [5,6]. Recent studies have demonstrated that XRF-derived chemical data are useful for evaluating limestone deposits and controlling the chemistry of cement raw materials [7,8].

Limestone deposits in Nigeria occur in several sedimentary basins and have been investigated for their suitability for cement manufacture. Fatah and Mirza [1] reported considerable variation in the CaO and associated oxide contents of limestone from the Avroman Formation, while Ogunribido and Ogungbamila [2] used XRF to assess limestone from Okeluse, southwestern Nigeria, for cement production. Similarly, Valdon [3] reported substantial variations in the chemical composition of limestone beds from the Guyuk area of northeastern Nigeria. These studies demonstrate that the chemical characteristics of limestone can vary considerably from one geological setting or mining location to another.

The Sokoto Basin contains extensive carbonate deposits that have been utilized as industrial raw materials. The Kalambaina Formation is particularly important because limestone from this formation is associated with cement production in the Wamakko area of Sokoto State. Recent geological and geochemical investigations have further demonstrated the industrial significance of limestone associated with a Cement quarry in the area [4]. The chemical characteristics of individual sections of the quarry, however, may differ because of lithological variation, intercalation with shale or marlstone, depositional conditions and other geological processes.

The chemistry of cement raw materials is commonly evaluated using parameters such as the lime saturation factor (LSF), silica modulus (SM) and alumina modulus (AM). These parameters provide information about the relative proportions of CaO, SiO_2, Al_2O_3 and Fe_2O_3 and are particularly useful in evaluating raw-mix chemistry [5,9]. Variations in LSF and other raw-mix parameters can influence clinker formation, free lime and cement performance [5,10]. The importance of controlling raw-material chemistry has also been emphasized in recent

investigations of Portland and limestone-containing cements [11-14].

The increasing interest in limestone-based cement systems has further highlighted the importance of understanding limestone composition. Limestone can contribute to cement systems both as a calcium-bearing raw material and as a constituent of blended and limestone-containing cements [15-17]. Proper characterization of local limestone resources is therefore important not only for conventional cement production but also for improving raw-material utilization and supporting lower-carbon cement technologies [18-20].

In spite of the importance of the Kalambaina limestone resource to cement production in Sokoto State, there is a need for additional analytical data showing the degree of chemical variation between selected mining points. Therefore, this study investigated the chemical composition and quality characteristics of limestone collected from two selected mining points at the Wamakko, Sokoto State. Specifically, the study determined the $CaCO_3$ content by acid dissolution and back-titration, characterized major and minor oxides using XRF, evaluated the repeatability of the XRF measurements and assessed selected cement raw-material indices derived from the oxide composition.

The novelty of this study lies in its comparative, site-specific assessment of two selected mining points within the Cement quarry, Wamakko. It combines calcium carbonate determination by acid dissolution and back-titration with XRF analysis of major and minor oxides. Unlike a general geochemical assessment, it examines the relationship between $CaCO_3$ content, oxide composition, and cement raw-material indices (LSF, SM, and AM). It also evaluates the repeatability of duplicate XRF measurements to assess the consistency of the analytical results. This integrated approach provides localized comparative data to support preliminary limestone quality evaluation and raw-material blending decisions at the investigated quarry.

MATERIALS AND METHODS

Study Area and Sample Collection

The study was carried out using limestone samples obtained from selected mining locations at Wamakko, Sokoto State, northwestern Nigeria. The quarry is associated with the carbonate-bearing succession of the Kalambaina Formation. Recent work on the area has identified limestone, marlstone and shale lithologies within the quarry sequence and has demonstrated the industrial relevance of the carbonate materials [4].

Limestone samples (2) were collected from selected mining points and designated Mining Point A (MP-A) and Mining Point B (MP-B). Approximately 2 kg of material was collected

from each location, placed in clean labelled sample bags and transported to the laboratory for preparation and analysis.

Reagents and Solution Preparation

Analytical-grade sodium hydroxide (36.5% purity, BDH) and hydrochloric acid (98% purity, BDH) were used for the titrimetric determination of $CaCO_3$. A nominal 0.5 M NaOH solution was prepared by dissolving 5.00 g of NaOH pellets in distilled water and making the solution up to 250 cm^3. The concentration was calculated from Equation 1:

$$C = m / (M_r V) \quad (1)$$

where m is the mass of NaOH, Mr is its molar mass and V is the solution volume.

A nominal 1.0 M HCl solution was prepared by measuring 8.6 cm^3 of concentrated hydrochloric acid and carefully transferring it into a 100 cm^3 volumetric flask, followed by dilution to the mark with distilled water. The acid solution was prepared with appropriate precautions because dilution of concentrated hydrochloric acid is highly exothermic.

Sample Preparation

The collected limestone samples were initially crushed and ground using a mortar and pestle to obtain relatively fine and homogeneous materials. Approximately 100 g of each sample was weighed and dried on a hot plate.

After preliminary drying, the samples were further pulverized using a vibrating cup mill. Portions of the prepared samples were transferred into separate crucibles. A mass of 3.6050 g of MP-A and 3.3090 g of MP-B was weighed into the respective crucibles.

The samples were heated in a Carbolite furnace for 30 min at approximately 1000 °C. Following heating, the crucibles were removed from the furnace and allowed to cool for approximately 10 min.

Approximately 1.3 g of each treated sample was subsequently transferred onto foil paper. About 9.1 g of lithium bromide (LiBr) was added to each sample as the fluxing material used in the subsequent fused-bead preparation. The sample-flux mixtures were processed using a Claisse fusion machine at approximately 1600 °C to produce fused analytical materials for XRF determination.

Determination of Calcium Carbonate Content

The $CaCO_3$ content of the limestone samples was determined by acid dissolution followed by back-titration. A measured quantity of limestone was reacted with an excess of hydrochloric acid. The principal reaction can be represented as shown Equation 2:

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2 \quad (2)$$

After the reaction had proceeded, the unreacted acid was titrated against the prepared sodium hydroxide solution. The burette readings obtained for the two samples are presented in Table 1.

Table 1. Titration data and $CaCO_3$ content of the limestone samples.

Sample	Initial reading (cm^3)	Final reading (cm^3)	Titre value (cm^3)	$CaCO_3$ (%)
MP-A	0.00	5.30	5.30	86.64
MP-B	0.00	17.30	17.30	59.42

The $CaCO_3$ contents obtained from the laboratory analytical procedure were 86.64% and 59.42% for MP-A and MP-B, respectively.

X-ray Fluorescence Analysis

The prepared fused samples were analyzed using an X-ray fluorescence spectrometer (PANalytical Axios, PANalytical, The Netherlands). Two analytical runs were performed for each sample. The XRF output provided the concentration of individual oxides for each run together with the mean, standard deviation (SD) and relative standard deviation (SD%).

The oxides determined included CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, SO_3, K_2O, P_2O_5, Mn_2O_3, TiO_2, Cl and Na_2O. Loss on ignition (LOI) and selected cement raw-material parameters were also obtained from the analytical output [22-23].

The mean concentration was calculated as shown in Equation 3:

$$\bar{x} = (x_1 + x_2) / 2 \quad (3)$$

where x_1 and x_2 are the concentrations obtained from the two analytical runs.

Relative standard deviation was expressed as shown in Equation 4:

$$SD\% = (SD / \bar{x}) \times 100 \quad (4)$$

The use of replicate XRF measurements provides an indication of instrumental repeatability and allows the precision of the reported oxide concentrations to be evaluated [22-23].

RESULTS AND DISCUSSION

Calcium Carbonate Content

The titrimetric analysis gave $CaCO_3$ contents of 86.64% and 59.42% for MP-A and MP-B, respectively (Figure 1). MP-A therefore contained a substantially greater proportion of

carbonate material than MP-B.

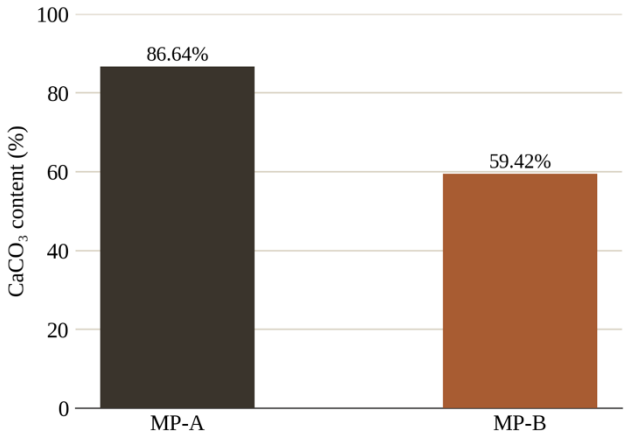


Figure 1. Comparison of $CaCO_3$ content (%) between MP-A and MP-B.

The difference of 27.22 percentage points between the two samples indicates considerable chemical heterogeneity between the selected mining points. Such variation is consistent with previous studies demonstrating that limestone deposits may exhibit substantial compositional differences between beds or sampling locations [1-4].

The comparatively high $CaCO_3$ value obtained for MP-A indicates a more carbonate-rich material, whereas the lower value for MP-B suggests a larger contribution from non-carbonate components. However, carbonate content alone is insufficient for complete assessment of limestone intended for cement manufacture because the accompanying SiO_2, Al_2O_3, Fe_2O_3, MgO and minor oxides also influence raw-mix chemistry.

Major Oxide Composition

The XRF results obtained from the two analytical runs are summarized in Table 2.

Table 2. Major and minor oxide composition of limestone from the two mining points.

Oxide	MP-A (%)	SD	SD%	MP-B (%)	SD	SD%
CaO	72.1425	0.2327	0.32	34.6685	0.2536	0.73
SiO_2	12.0221	0.0764	0.64	20.2224	0.0428	0.21
Al_2O_3	4.0053	0.0177	0.44	5.8059	0.0209	0.36
Fe_2O_3	1.4255	0.0077	0.54	3.3817	0.0062	0.18
MgO	1.8103	0.0168	0.93	1.9436	0.0144	0.74

Oxide	MP-A (%)	SD	SD%	MP-B (%)	SD	SD%
SO_3	0.6115	0.0070	1.15	0.3466	0.0081	2.33
K_2O	0.0962	0.0038	3.90	0.2101	0.0001	0.03
P_2O_5	0.5034	0.0157	3.11	0.4802	0.0217	4.52
Mn_2O_3	-0.0345*	0.0023	—	0.0559	0.0007	1.16
TiO_2	0.0456	0.0002	0.53	0.1186	0.0025	2.09
Cl	0.0967	0.0006	0.58	0.0068	0.0003	3.94
Na_2O	0.5389	0.0106	1.97	0.2175	0.0495	22.74
LOI	12.7800	0.0000	0.00	32.4100	0.0000	0.00

**The negative Mn_2O_3 value is an instrumental output and should not be interpreted as an actual negative concentration; it indicates a concentration below the effective analytical/quantification range.*

The XRF results (Figure 2) show a pronounced difference in the chemical composition of the two mining points. MP-A recorded a CaO concentration of 72.1425%, whereas MP-B recorded 34.6685%.

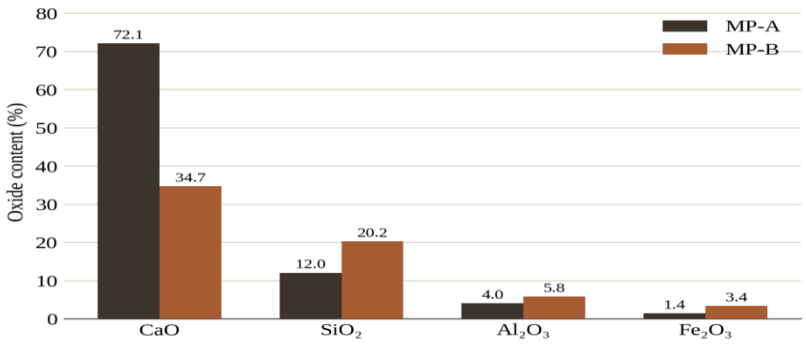


Figure 2. Comparison of major oxide composition (CaO, SiO_2, Al_2O_3, Fe_2O_3) between MP-A and MP-B.

Thus, the CaO concentration of MP-A was approximately twice that of MP-B.

The reverse trend was observed for SiO_2. MP-A contained 12.0221% SiO_2, whereas MP-B contained 20.2224%. The concentrations of Al_2O_3 and Fe_2O_3 were also higher in MP-B, with

values of 5.8059% and 3.3817%, respectively, compared with 4.0053% and 1.4255% in MP-A.

The higher concentrations of SiO_2, Al_2O_3 and Fe_2O_3 in MP-B indicate a greater contribution from silicate-, aluminosilicate- and iron-bearing materials. This type of variation is important in cement raw-material control because the relative proportions of these oxides determine the chemistry of the raw mix and influence clinker formation [5,6,9].

The observed variation is comparable with findings reported for other limestone deposits. Valdon [3], for example, reported variation in CaO, SiO_2, Al_2O_3 and Fe_2O_3 in limestone beds from Guyuk, while Ogunribido and Ogungbamila [2] reported variable major oxide compositions in Okeluse limestone. Recent investigation of the BUA Cement quarry also identified variations among carbonate lithofacies within the Kalambaina Formation [4].

The present results therefore provide further evidence that chemical characterization at individual mining points is important for effective quarry and raw-material management.

Repeatability of XRF Measurements

The duplicate XRF measurements demonstrated good analytical repeatability. The SD values were generally small compared with the corresponding mean concentrations.

For example, CaO in MP-A was 72.1425 ± 0.2327%, corresponding to an SD% of 0.32%. For MP-B, CaO was 34.6685 ± 0.2536%, with an SD% of 0.73%. The SiO_2 results also showed relatively low variability, with SD% values of 0.64% for MP-A and 0.21% for MP-B.

The low variation between replicate measurements suggests good short-term instrumental repeatability. This is important because reliable oxide measurements are required for the calculation and interpretation of cement raw-material parameters [5,8]. Similar reliance on replicate or calibrated XRF measurements has been demonstrated in recent cement raw-material studies [6,9].

It should, however, be recognized that instrumental repeatability does not necessarily establish the representativeness of the collected sample for the entire mining point or quarry. More extensive spatial sampling would therefore be required to characterize the full range of chemical variability within the quarry.

Loss on Ignition

A considerable difference was observed in the LOI values of the two samples (Figure 3). MP-A recorded an LOI of 12.78%, while MP-B recorded 32.41%.

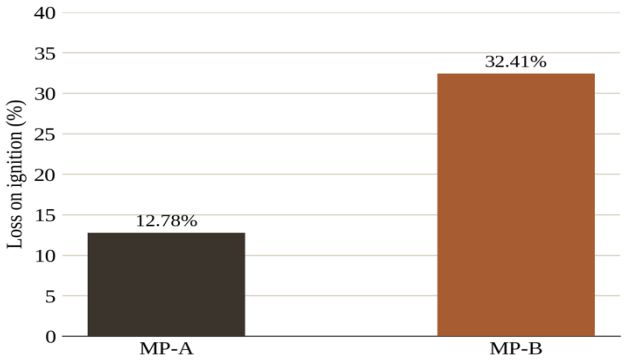


Figure 3. Comparison of loss on ignition (LOI, %) between MP-A and MP-B.

LOI is particularly relevant to limestone because carbonate minerals release CO_2 during heating. The magnitude of LOI can therefore provide useful information concerning the carbonate and volatile fractions of a sample [6]. The considerably higher LOI of MP-B indicates a substantially different volatile/carbonate contribution compared with MP-A.

The relatively high LOI observed for MP-B is also consistent with its lower CaO concentration and higher concentrations of SiO_2, Al_2O_3 and Fe_2O_3. Similar chemical contrasts between limestone-rich and more siliceous raw materials have been reported in cement raw-material studies [6,16].

Cement Raw Material Indices

The calculated cement raw-material parameters obtained from the XRF output are presented in Table 3.

Table 3. Cement raw-material indices of the investigated limestone samples.

Parameter	MP-A	MP-B
LSF (CLRM)	183.13	52.70
LSF (CM)	182.04	52.33
Silica modulus (SM)	2.2137	2.2011
Alumina modulus (AM)	2.8098	1.7168

The silica modulus (SM) is expressed as shown in Equation 5:

$$SM = SiO_2 / (Al_2O_3 + Fe_2O_3) \quad (5)$$

while the alumina modulus (AM) is expressed as given in Equation 6:

$$AM = Al_2O_3 / Fe_2O_3 \quad (6)$$

The LSF (Equation 7) is commonly expressed as:

$$LSF = 100\ CaO / (2.8\ SiO_2 + 1.18\ Al_2O_3 + 0.65\ Fe_2O_3) \quad (7)$$

These parameters are commonly used in cement raw-mix chemistry and provide information about the relative balance of the major clinker-forming oxides [5,9].

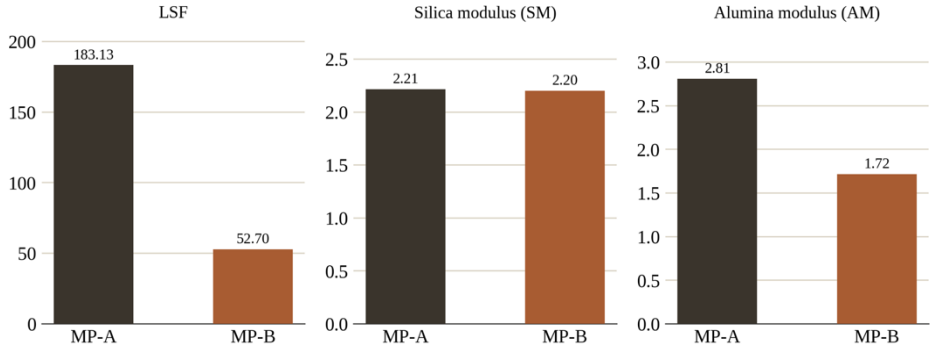


Figure 4. Cement raw-material indices (LSF, silica modulus and alumina modulus) of MP-A and MP-B.

The calculated SM values (Figure 4) were very similar for the two samples, being 2.2137 for MP-A and 2.2011 for MP-B. Despite the considerable differences in the absolute concentrations of the individual oxides, the ratios of SiO_2 to the combined Al_2O_3 and Fe_2O_3 contents were therefore relatively similar. The AM values (Figure 4) showed a greater difference. MP-A recorded an AM of 2.8098, whereas MP-B recorded 1.7168. This difference is attributable principally to the higher Fe_2O_3 concentration of MP-B relative to its Al_2O_3 content.

A pronounced difference was observed in the LSF (Figure 4). The reported LSF values were 183.13 and 52.70 for MP-A and MP-B, respectively. The high LSF of MP-A reflects its very high CaO concentration relative to the acidic oxide components, whereas the considerably lower value for MP-B reflects its lower CaO and higher SiO_2, Al_2O_3 and Fe_2O_3 contents. However, these LSF values should not be interpreted as direct evidence that either limestone sample alone represents an appropriate kiln raw mix. LSF is primarily a control parameter for a complete cement raw mix rather than an isolated limestone material [5,10]. Consequently, the unusually high LSF of MP-A mainly demonstrates its highly calcareous character, while the lower LSF of MP-B reflects its relatively greater contribution of non-calcareous oxides.

Previous studies have shown that variations in raw-mix chemistry and LSF can

influence clinker formation, clinker reactivity and cement properties [5,10]. Labidi and Megriche [7] similarly demonstrated the importance of controlling LSF, silica modulus and alumina modulus during raw-mix design.

Comparison of the Two Mining Points

The overall chemical characteristics of the two samples demonstrate that MP-A is substantially more calcium-rich than MP-B. Its CaO concentration was 72.1425%, compared with 34.6685% for MP-B, while its SiO_2, Al_2O_3 and Fe_2O_3 contents were all lower.

MP-B, on the other hand, contained higher concentrations of SiO_2, Al_2O_3 and Fe_2O_3. From a raw-material blending perspective, the two materials therefore have complementary chemical characteristics. MP-A contributes a larger calcium-bearing fraction, whereas MP-B contributes proportionally greater amounts of the silica-, alumina- and iron-bearing components required in cement raw-mix chemistry. This type of chemical complementarity can be useful during raw-material blending. Rather than treating limestone from different quarry sections as chemically identical, chemical data can be used to determine suitable blending proportions and maintain a more consistent raw-mix composition [5,7,8].

Recent Nigerian studies have similarly demonstrated substantial differences in the chemical composition of limestone and Portland limestone cement materials. Aginam et al. [17] reported differences in CaO, SiO_2, Al_2O_3 and Fe_2O_3 among Portland limestone cement samples from different sources, while Danchuwa et al. [18] demonstrated compositional differences among Portland limestone cements produced in Nigeria.

The present findings are particularly relevant to the Cement quarry because recent geological work on the same quarry has demonstrated variations in limestone, marlstone and associated lithologies [4]. The present chemical data therefore complement the existing geological observations by showing that selected mining points can also differ substantially in their major oxide composition.

Implications for Cement Manufacture

The chemical differences observed between MP-A and MP-B have implications for quarry management and cement raw-material preparation. Cement manufacture requires a relatively controlled balance of CaO, SiO_2, Al_2O_3 and Fe_2O_3 to obtain the desired clinker chemistry [5,6]. MP-A, with its substantially higher CaO concentration, can be regarded as the more calcareous of the two investigated materials. MP-B contains considerably higher concentrations of the principal acidic oxides, particularly SiO_2, together with increased Al_2O_3 and Fe_2O_3.

Consequently, the two materials should not necessarily be regarded as interchangeable feed materials without considering their chemical contribution to the raw mix. Appropriate blending based on routine chemical analysis could provide a means of compensating for the compositional variability between different quarry sections.

Such quality-control practices are consistent with current approaches to cement raw-material proportioning, in which rapid analytical information is used to adjust the raw mix and maintain target chemical modules [8,9]. This is increasingly important because optimization of raw materials can contribute to efficient cement manufacture and, in broader terms, to reduction of energy use and environmental impacts associated with cement production [13,19-21].

The findings should nevertheless be interpreted within the scope of the present investigation. Only two mining points were evaluated, and therefore the results cannot be considered representative of the entire BUA Cement quarry. A larger sampling campaign involving multiple benches, depths and spatial coordinates would provide a stronger basis for developing a quarry-wide chemical variability model.

Overall Assessment of Limestone Quality

Based on the results obtained, MP-A is characterized by a substantially higher CaO and $CaCO_3$ content, while MP-B contains higher concentrations of SiO_2, Al_2O_3 and Fe_2O_3. The results therefore demonstrate that the two selected mining points represent chemically distinct limestone materials.

The $CaCO_3$ values of 86.64% and 59.42% further support the distinction between the samples. However, quality assessment for cement manufacture should not rely on $CaCO_3$ alone. A complete evaluation should consider CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, SO_3, alkalis, LOI and the resulting raw-mix parameters.

The observed chemical variability is consistent with the general finding that limestone quality can vary substantially within and between deposits [1-4]. It also reinforces the importance of routine XRF analysis and controlled blending in cement raw-material management [5,7,9].

CONCLUSION

The chemical composition of limestone from the two selected mining points at Wamakko, Sokoto State, Nigeria, differed substantially. Mining Point A recorded a $CaCO_3$ content of 86.64% and an XRF-derived CaO concentration of 72.1425 ± 0.2327%, whereas Mining Point B recorded 59.42% $CaCO_3$ and 34.6685 ± 0.2536% CaO. Mining Point A was characterized by higher CaO and lower SiO_2, Al_2O_3 and Fe_2O_3 contents, while Mining Point B exhibited the opposite trend. The LOI values were 12.78% and 32.41% for MP-A and MP-B, respectively. The duplicate XRF analyses showed good repeatability, with generally low SD and SD% values.

The calculated silica modulus values were similar for both samples, whereas substantial differences occurred in the alumina modulus and LSF. The high LSF obtained for MP-A and lower value obtained for MP-B reflect their markedly different CaO-to-acidic-oxide ratios and demonstrate the importance of considering the complete oxide composition rather than $CaCO_3$ content alone.

Overall, the investigated mining points exhibit substantial chemical variability. MP-A represents the more calcium-rich material, while MP-B is relatively enriched in silica, alumina and iron oxides. The results demonstrate the importance of systematic chemical characterization and controlled blending of limestone from different quarry sections to support consistent cement raw-mix chemistry.

Further studies involving a larger number of samples distributed across different quarry benches and depths are recommended to establish the spatial distribution of chemical variability within the Cement quarry and provide a stronger basis for quarry-wide raw-material management.

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