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Understanding the Influence of Different Limestone Sources on Portland Limestone Cement Characteristics and Advancements in Green Cement

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Abstract: **This paper investigates the influence of varied limestone sources on the chemical compositions and performance of Portland limestone cement. Additionally, it explores recent advancements in green cement technologies, focusing on magnesium oxychloride cement (MOC) and geopolymer concrete. Three samples of Portland limestone cement, produced by different manufacturers that sourced their raw materials from different locations, were used in this study. The samples were labeled Sample A, Sample B, and Sample C. They were classified as CEM II, grade 42.5N in conformity with standard organisations. Properties of concrete materials such as chemical composition, sieve analysis, specific gravity, and fineness were investigated. An X-ray fluorescence test was conducted on the three samples. Workability and compressive strength tests were also carried out on concrete samples. Recent advancements in green cement technologies, including the development of water-resistant MOC and the use of geopolymer concrete, are also reviewed. These advancements present promising alternatives for reducing the construction industry's carbon footprint and enhancing the sustainability of cement-based materials. The results of the investigation revealed a variation in the chemical composition of the three samples. This variation in the chemical composition was found to be from different limestone sources and other raw materials. These variations impacted the performance of the concrete Samples with higher SiO_3, Al_2O_3, and Fe_2O_3. Moderate CaO and low MgO exhibited high slump values and compressive strength. However, all the samples gave desirable consistency and strength. The average of three cubes cured for 7, 14, 21, and 28 days was tested for compressive strength. From the results, sample B gave higher values of 15.17N/mm^2, 24.40N/mm^2, 29.43N/mm^{2,} and 34.07N/mm^2 for strength at 7, 14, 21 and 28days.**

Keywords: **Portland limestone cement, chemical composition, X-ray fluorescence test, workability, strength, green cement, sustainability, magnesium oxychloride cement, and geopolymer concrete.**

I. INTRODUCTION

Portland limestone cement, a blended cement with higher limestone content, has emerged as a significant alternative to traditional Portland cement [1]. This is due to its environmental benefits and potential improvements in concrete performance. By incorporating limestone as a partial replacement for clinker, Portland limestone cement has reduced carbon dioxide emissions and enhanced certain properties of concrete, such as durability and resistance to chloride-induced corrosion [2]. However, the performance of Portland limestone cement can vary significantly depending on the sources of limestone used in its production.

Limestone is a common raw material in cement manufacturing, it is geologically diverse and can exhibit variations in chemical composition and impurities based on its geological origin [3]. These variations can influence the chemical characteristics of the resulting Portland limestone cement and its performance in concrete applications. Understanding the impact of different limestone sources on Portland limestone cement characteristics is crucial for optimizing concrete mix designs and ensuring desirable concrete properties.

Recent advancements in sustainable construction materials have introduced green cement alternatives such as Magnesium Oxychloride Cement (MOC). MOC is produced by mixing magnesium oxide (MgO) powder and a concentrated solution of magnesium chloride ($MgCl_2$), and it is considered carbon-neutral due to its ability to absorb CO_2 from the atmosphere [4]. Despite its higher compressive strength and fast setting properties, MOC has historically been limited by poor water resistance. However, recent developments have improved its water resistance, making it a promising green cement alternative that could significantly reduce construction industry emissions.

There are various strategies for making environmentally friendly cement [5]. With cement production responsible for approximately 8% of global CO_2 emissions, new approaches such as using geopolymer concrete, which replaces traditional limestone-based cement with materials like fly ash and slag, are gaining attention. Geopolymer concrete can reduce emissions by up to 50% and utilize stockpiled industrial byproducts, making it a sustainable option.

Studies have highlighted the importance of chemical composition in determining the reactivity, workability, and strength development of Portland limestone cements [6], [7]. However, comprehensive investigations utilizing advanced analytical techniques such as X-ray fluorescence (XRF) are needed to elucidate the specific effects of varied limestone sources on Portland limestone cement properties.

This paper aims to address this gap by investigating the chemical composition and performance of Portland limestone cement samples sourced from different limestone deposits. The findings will contribute to a deeper understanding of how geological variations in limestone sources impact the characteristics of Portland limestone cement and guide concrete industry practices toward more sustainable and effective cement utilization. Also to explore the potential of green cement alternatives like MOC in reducing construction emissions.

II. LITERATURE REVIEW

After carrying out a comprehensive literature review on Portland limestone cement and Portland cement, it is clear that the sources of limestone significantly influence the chemical compositions and properties of the resulting cement. Studies have shown that limestone's geological variations lead to different levels of calcium carbonate ($CaCO_3$) and impurities, which affect the reactivity and performance of the cement [2], [3]. Moreover, the integration of sustainable practices in cement production, such as the development of MOC and the use of geopolymer concrete, highlights the industry's potential to reduce its environmental impact.

Producing a tonne of conventional cement in Australia emits about 0.82 tonnes of CO_2, primarily due to the calcination process, where limestone (calcium carbonate) is heated to produce quicklime (calcium oxide), releasing CO_2 in the process [5]. Efforts to create a zero-carbon cement industry have led to the exploration of alternatives like carbon-negative cement, made with magnesium oxide, which can absorb CO_2 from the air. Additionally, mineral carbonation processes and improved building designs that use less concrete can further reduce emissions.

The incorporation of industrial byproducts such as fly ash and silica fume in cement production not only enhances the performance of cements like MOC but also addresses the issue of industrial waste management. By adding these byproducts, researchers have developed a water-resistant MOC that retains its compressive and flexural strength even under prolonged exposure to water [4]. This breakthrough makes MOC a viable alternative for a broader range of construction applications, potentially replacing conventional cement in the future.

In a research on some common Portland limestone cement of grade 42.5 [8] and [6] concluded that the samples had similar compositions even though some variations were observed. Their investigations were however limited since they employed traditional techniques instead of spectrometry methods like X-ray fluorescence method which has a high level of precision and accuracy.

The chemical composition of Portland limestone cement primarily consists of CaO, SiO_2, Al_2O_3, and Fe_2O_3 with a minor amount of Na_2O, K_2O, and MgO coming from clay fraction of raw materials and SO_3 from gypsum [9].

In their research, [7] established that the main chemical component of limestone is calcium carbonate, which undergoes thermal decomposition during the cement manufacturing process to form calcium oxide (lime). [10] in line with the findings of [11] pointed out that marl or clay and limestone are the two main ingredients of Portland limestone cement because they have major oxides that make them suitable.

III. MATERIALS AND METHODS

A. Cement: Three samples of Portland limestone cement, labeled as follows: Sample A, Sample B, and Sample C, were collected from different manufacturers. The samples were classified as CEM II, grade 42.5N, in conformity with [9] and [12]. The raw materials for these cements were sourced from different limestone deposits, resulting in variations in their chemical compositions. Limestone is readily available near cement factories, hence manufacturing companies source their raw materials basically limestone around their factory locations [11].

B. Fine Aggregates: Fine aggregate passing 4.75mm sieve was used for this research, it was sourced from River Niger in Onitsha, Anambra State Nigeria and labelled Sample 1. The fine aggregate met the specifications of [13] and is in conformity with [14]. It was kept clean and dry to prevent bulking of aggregates.

C. Coarse Aggregates: Coarse aggregate passing 25mm sieve size was used for this research with an average size of 19mm. it was sourced from Ezza in Abakility, Ebonyi State, Nigeria. Sieve analysis test and specific gravity test were carried out in accordance with [13] to determine the suitability of the material in concrete.

D. Water: The water used for mixing concrete in this research was clean, drinkable and free from impurities. It was sourced within the Concrete Laboratory at Nnamdi Azikiwe University, Awka. It was tested in conformity with [15]. The PH value of the water was 7.6.

E. Characterization of Cement Samples Using X-ray Fluorescence Test Method

An X-ray fluorescence (XRF) test, a non-destructive analytical technique used to determine the elemental composition of cement samples, was carried out on the three samples of Portland limestone cement. To prepare the samples, each one was initially crushed with an electric crusher and then pulverized for 60 seconds using a Herzog Gyro-mill (Simatic C7-621). Pellets were prepared from the pulverized samples by first grinding 20g of each sample for 60 seconds. The Gyro-mill was cleaned after each grinding session to prevent contamination. An aluminum cup was then filled with 1g of stearic acid, serving as a binding agent, and subsequently filled to the level point with the sample. The cup was then subjected to 200KN of pressure for 60 seconds using Herzog pelletizing equipment to form pellets.

The XRF machine, Nitron 3000, was powered on and stabilized for 5 minutes before starting the analysis. The CU-Zn method was chosen for its ability to detect a wide range of elements and sesquioxides due to its high intensity. Each 2mm pellet was placed on the sample holder of the XRF equipment (Phillips PW-1800) for analysis. The ray point was positioned over the pellet, and the ray button was pressed to begin data collection. Data were collected in triplicates, and the average was automatically calculated. This procedure was done on all the XRF tests for the samples to determine the percentage chemical composition in both oxide and elemental forms.

The method used to determine the percentage of chemical composition in the three Portland limestone cement samples is based on the principles of atomic physics and quantum chemistry. During the process, the samples were exposed to the entire spectrum of photons emitted from a standard X-ray tube, which caused the elements in the samples to emit secondary fluorescence with characteristic X-ray line spectra.

The energy and intensities of the emitted lines were determined by the detection system, which consists of two units: the primary channel simultaneous wavelength dispersive spectrometer and a personal computer for control and data processing. The rapid detection system employs pre-positioned (analyzing) crystals around the specimen, causing dispersion of the wavelength of the secondary radiation. The intensity of the individual wavelengths was measured in a mass gas flow detector, allowing simultaneous measurements of up to ten elements at peak and background positions.

The output signals from the detector were fed into an analyzer, where the photon counts were stored in computer memory. The count rate for each element was calibrated by comparing it to the count rate from a standard of accurately pre-determined composition. The spectral line energies of the emitted lines were used in the quantitative analysis of the elements in the specimen, and the intensities of the emitted lines were related to their concentration for quantitative analysis.

The XRF tests on the samples were conducted at Allschoolabs Scientific, a research laboratory located at Suite C1, God's Promise Complex, Bells Drive, Ota, Ogun State, Nigeria.

F. *Analysis on Concrete Materials*

All the concrete materials used in this research were tested and analysed in accordance with the required standard specifications. The Portland limestone cement samples were tested for fineness in accordance with [16]. Sieve analysis and specific gravity tests were carried out on the fine and coarse aggregates to determine their suitability in concrete and right proportion to be used in mixing concrete and they proved adequate to be used in concrete.

G. *Concrete Mix*

Concrete mix design was calculated in accordance with [17]. The ratio of 1:1.83:3.39:0.522 was obtained for a concrete characteristic strength of 25N/mm^2. Concrete was batched, mixed, tested for slump, compacted in iron moulds and labelled A1, B1 and C1. A total of 36 cubes was produced, 12 cubes for each Portland limestone cement sample to be tested for compressive strength at 7, 14, 21 and 28days of curing. The average of three cubes was considered as the compressive strength for each curing age. Below is a plate showing the 36 cubes casted in moulds and totally immersed in water after 24 hours.

Plate 1: A Total of 36 cubes for Samples A, B and C casted in iron moulds ready for curing

IV. RESULTS

A. X-Ray Fluorescence Test Results

Table 1: Presents The key findings from the XRF test results for Samples A, B and C

No.	Chemical Component	Chemical Formula	Weight Percentage Concentration		
			Sample A	Sample B	Sample C
1.	Silicon Dioxide	SiO_2	8.694	12.418	9.516
2.	Aluminum Oxide	Al_2O_3	5.042	5.945	4.561
3.	Iron (III) Oxide	Fe_2O_3	3.676	3.735	2.836
4.	Calcium Oxide	CaO	77.171	70.326	74.178
5.	Sulfur Trioxide	SO_3	3.385	2.396	2.971
6.	Magnesium Oxide	MgO	0.00	2.229	3.124
7.	Potassium Oxide	K_2O	0.510	1.333	1.112
8.	Others	-	1.522	1.618	1.702

V. DISCUSSION

From the X-ray fluorescence test results, it was observed that Sample A exhibits higher concentration of Calcium Oxide (CaO = 77.171%) and Sulfur Trioxide (SO_3 = 3.385%). Sample B exhibits higher concentration of Silicon Dioxide (SiO_2 = 12.418%) and Aluminum Oxide (Al_2O_3 = 5.945%). Sample C exhibits higher concentration of Magnesium Oxide (MgO = 3.124%). All this differences in chemical composition can affect the reactivity of the Portland limestone cements in concrete in different ways. Higher concentration of SiO_2 and Al_2O_3 can impact the workability, setting time and contribute to overall durability and sulphate resistance of concrete. Higher CaO contribute effectively to the hydration process and early strength development of concrete while Fe_2O_3 influences color, setting time and early strength development of concrete. The results shows that different sources of raw materials for producing Portland limestone cements and the manufacturing processes of different Portland limestone cement factories have impact on the chemical composition of the final product. It can also affect its performance in the workability, strength and durability of concrete.

B. *Sieve Analysis Results*

The sieve analysis results show that the fine aggregate and coarse aggregate used in this study are suitable for making concrete. For the fine aggregate, the coefficient of uniformity Cu is 2.5, and coefficient of curvature Cc is 1.15. Physical observations showed that the fine aggregate is densely graded and has wide range of sizes, low void content, low permeability and grain to grain contact. Air gaps are filled with finer particles. For the coarse aggregates the Coefficient of uniformity Cu is 1.88 and coefficient of curvature Cc is 1.44 suggesting a uniformly graded aggregate with narrow range of sizes and predominantly 19mm. The aggregates were tested in accordance with [16] and met the specifications of [18]. Figure 1 and Figure 2 presents the cumulative percent finer against sieve sizes.

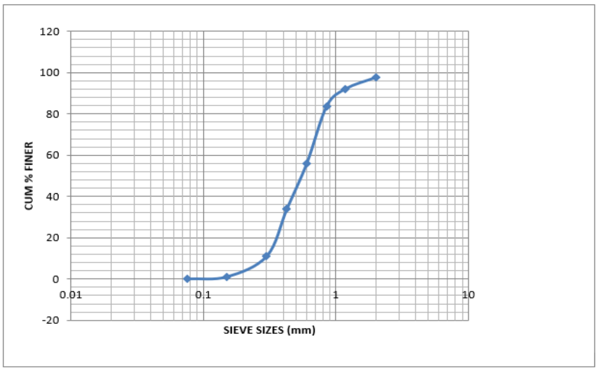

Figure 1: Gradation curve for Fine aggregate

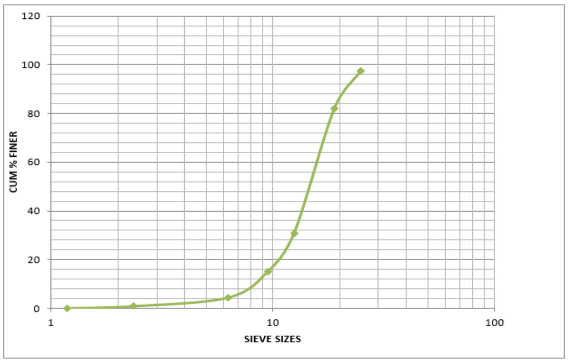

Figure 2: Gradation curve for Coarse aggregate

C. *Workability Test Result*

High workability in the concrete mix shows that the concrete is easier to handle. All the concrete mix had a moderate slump value. However, due to the variation in the chemical compositions of the Portland limestone cement Samples used in producing each of the concrete mix, some samples are more workable than others. This is a clear indication that the variations in their chemical compositions impacted the performance of the Samples. The slump values for Sample A, Sample B, and Sample C are; 46mm, 63mm, and 59mm respectively.

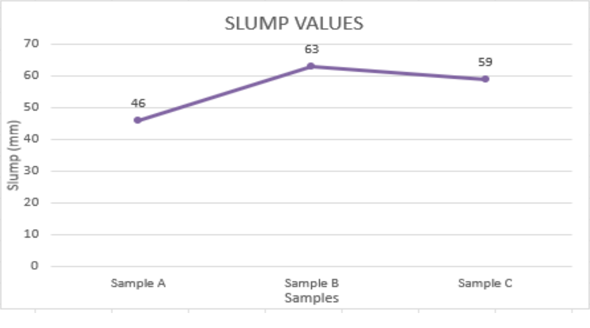

Figure 3: Slump Value Against Samples A, B, and C.

D. *Compressive Strength Test*

Compressive strength tests were conducted on the concrete cubes in conformity with the specifications of [19]. Three cubes were crushed for each sample at each curing age and the average was taken as the compressive strength, all the cubes gave desirable concrete strength. From the compressive strength result in Figure 4, it was observed that the compressive strength increases with curing age. This is a result of the ongoing hydration process of cement which results in strength development. Again, the impact of the varied chemical compositions of the samples cannot be undermined as the variations in the compressive strengths are a result of the variations in the chemical composition of the samples.

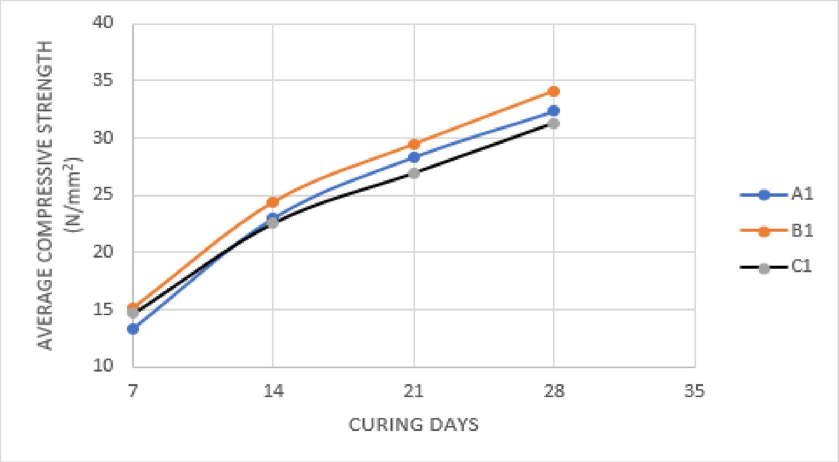

Figure 4: Average Compressive Strength against Curing Days

VI. CONCLUSION

This study highlights the critical influence of limestone sources on the chemical composition and performance of Portland limestone cement. The variations in chemical composition due to different limestone sources impact the workability and strength of the resulting concrete. Additionally, the development of green cement alternatives such as MOC and geopolymer concrete provides viable solutions to reduce the carbon footprint of the cement industry. The following conclusions are drawn from the results of the research;

1. CaO, SiO_2, Al_2O_3, Fe_2O_3, and SO_3 are the main chemical components of Portland limestone cement. They made up an average of 95.62% of the compositions. This conforms to the specifications of [9].

2. Different limestone sources lead to significant variations in the chemical composition of Portland limestone cement, affecting its workability and compressive strength. Sample B, with higher SiO_2, Al_2O_3, and Fe_2O_3 content, demonstrated superior performance.

3. The Concentration of SiO_2 in Portland limestone cement is significant in determining the strength of concrete made with the cement. The composition should range from 7% to 15% which conforms to standard specification.

4. The strength of concrete cubes increases with an increase in the curing age.

5. High silica Oxide (SiO_2), Alumina Oxide (Al_2O_3), Iron Oxide (Fe_2O_3), moderate Calcium oxide (CaO) above 70%, then low Magnesium Oxide (MgO) have proven to significantly impact the strength and durability of concrete.

6. The workability of concrete is also impacted by the chemical composition of Portland limestone cement.

7. Advances in MOC technology, including the addition of fly ash and silica fume, have improved its water resistance and compressive strength. MOC represents a promising green cement alternative that can significantly reduce construction industry emissions.

8. Literature reviews have shown that utilizing fly ash and slag, geopolymer concrete can reduce carbon emissions by up to 50% and address industrial waste disposal issues. Increased adoption of geopolymer concrete can contribute to a more sustainable construction industry.

Future Research

Further research should focus on optimizing the mix designs of Portland limestone cement to enhance its performance. Additionally, exploring the practical applications and scalability of green cement technologies like MOC and geopolymer concrete is essential for their widespread adoption in the construction industry. Addressing challenges such as the corrosion of steel reinforcement in MOC and integrating geopolymer concrete into existing supply chains will be crucial

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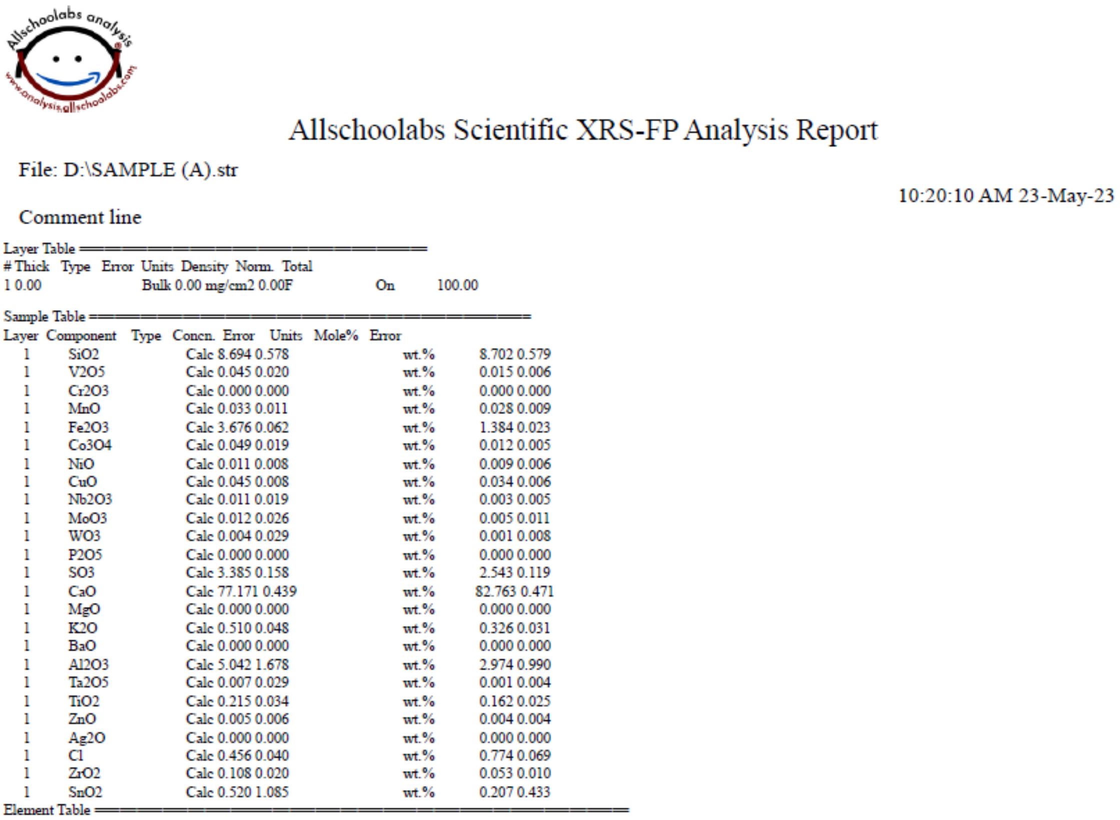
APPENDIX

A. X-Ray Fluorescence Test Result for Sample A



Allschoolabs Scientific XRS-FP Analysis Report

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Comment line

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Layer Table ==================================
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Sample Table ========================================
Layer  Component   Type   Concn.  Error   Units   Mole%   Error
  1      SiO2         Calc 8.694 0.578              wt.%     8.702 0.579
  1      V2O5         Calc 0.045 0.020              wt.%     0.015 0.006
  1      Cr2O3        Calc 0.000 0.000              wt.%     0.000 0.000
  1      MnO          Calc 0.033 0.011              wt.%     0.028 0.009
  1      Fe2O3        Calc 3.676 0.062              wt.%     1.384 0.023
  1      Co3O4        Calc 0.049 0.019              wt.%     0.012 0.005
  1      NiO          Calc 0.011 0.008              wt.%     0.009 0.006
  1      CuO          Calc 0.045 0.008              wt.%     0.034 0.006
  1      Nb2O3        Calc 0.011 0.019              wt.%     0.003 0.005
  1      MoO3         Calc 0.012 0.026              wt.%     0.005 0.011
  1      WO3          Calc 0.004 0.029              wt.%     0.001 0.008
  1      P2O5         Calc 0.000 0.000              wt.%     0.000 0.000
  1      SO3          Calc 3.385 0.158              wt.%     2.543 0.119
  1      CaO          Calc 77.171 0.439             wt.%    82.763 0.471
  1      MgO          Calc 0.000 0.000              wt.%     0.000 0.000
  1      K2O          Calc 0.510 0.048              wt.%     0.326 0.031
  1      BaO          Calc 0.000 0.000              wt.%     0.000 0.000
  1      Al2O3        Calc 5.042 1.678              wt.%     2.974 0.990
  1      Ta2O5        Calc 0.007 0.029              wt.%     0.001 0.004
  1      TiO2         Calc 0.215 0.034              wt.%     0.162 0.025
  1      ZnO          Calc 0.005 0.006              wt.%     0.004 0.004
  1      Ag2O         Calc 0.000 0.000              wt.%     0.000 0.000
  1      Cl           Calc 0.456 0.040              wt.%     0.774 0.069
  1      ZrO2         Calc 0.108 0.020              wt.%     0.053 0.010
  1      SnO2         Calc 0.520 1.085              wt.%     0.207 0.433
Element Table ==================================================
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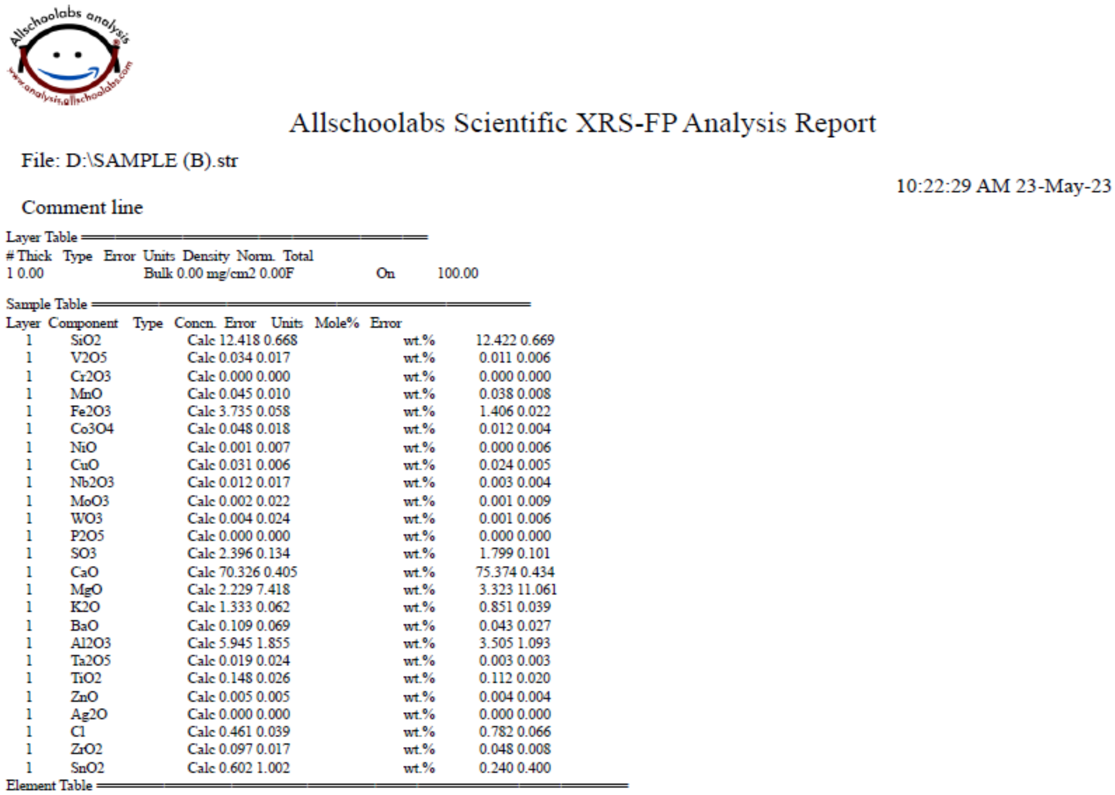
B. X-Ray Fluorescence Test Result for Sample B



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Sample Table ========================================
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  1      V2O5         Calc 0.034 0.017              wt.%     0.011 0.006
  1      Cr2O3        Calc 0.000 0.000              wt.%     0.000 0.000
  1      MnO          Calc 0.045 0.010              wt.%     0.038 0.008
  1      Fe2O3        Calc 3.735 0.058              wt.%     1.406 0.022
  1      Co3O4        Calc 0.048 0.018              wt.%     0.012 0.004
  1      NiO          Calc 0.001 0.007              wt.%     0.000 0.006
  1      CuO          Calc 0.031 0.006              wt.%     0.024 0.005
  1      Nb2O3        Calc 0.012 0.017              wt.%     0.003 0.004
  1      MoO3         Calc 0.002 0.022              wt.%     0.001 0.009
  1      WO3          Calc 0.004 0.024              wt.%     0.001 0.006
  1      P2O5         Calc 0.000 0.000              wt.%     0.000 0.000
  1      SO3          Calc 2.396 0.134              wt.%     1.799 0.101
  1      CaO          Calc 70.326 0.405             wt.%    75.374 0.434
  1      MgO          Calc 2.229 7.418              wt.%     3.323 11.061
  1      K2O          Calc 1.333 0.062              wt.%     0.851 0.039
  1      BaO          Calc 0.109 0.069              wt.%     0.043 0.027
  1      Al2O3        Calc 5.945 1.855              wt.%     3.505 1.093
  1      Ta2O5        Calc 0.019 0.024              wt.%     0.003 0.003
  1      TiO2         Calc 0.148 0.026              wt.%     0.112 0.020
  1      ZnO          Calc 0.005 0.005              wt.%     0.004 0.004
  1      Ag2O         Calc 0.000 0.000              wt.%     0.000 0.000
  1      Cl           Calc 0.461 0.039              wt.%     0.782 0.066
  1      ZrO2         Calc 0.097 0.017              wt.%     0.048 0.008
  1      SnO2         Calc 0.602 1.002              wt.%     0.240 0.400
Element Table ==================================================
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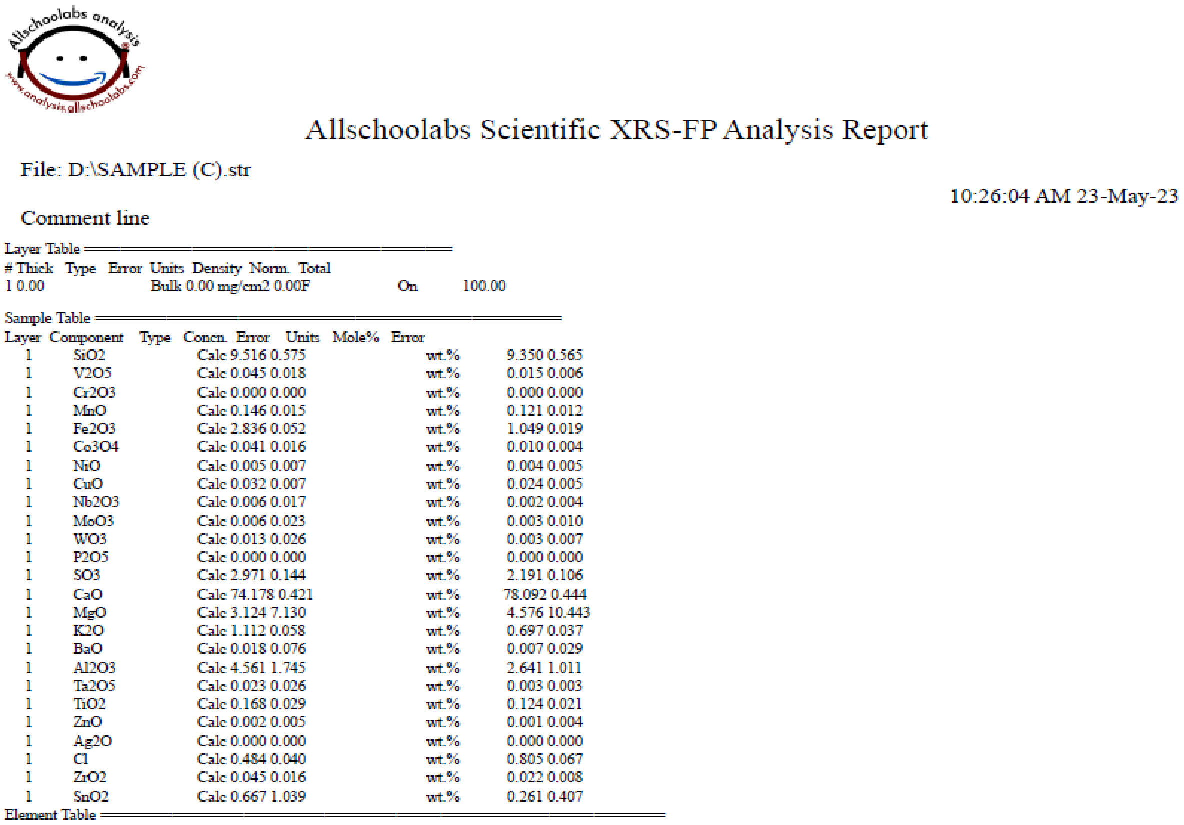
C. X-Ray Fluorescence Test Result for Sample C



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Sample Table ============================================
Layer Component  Type  Concn. Error  Units  Mole%  Error
  1     SiO2           Calc 9.516 0.575          wt.%     9.350 0.565
  1     V2O5           Calc 0.045 0.018          wt.%     0.015 0.006
  1     Cr2O3          Calc 0.000 0.000          wt.%     0.000 0.000
  1     MnO            Calc 0.146 0.015          wt.%     0.121 0.012
  1     Fe2O3          Calc 2.836 0.052          wt.%     1.049 0.019
  1     Co3O4          Calc 0.041 0.016          wt.%     0.010 0.004
  1     NiO            Calc 0.005 0.007          wt.%     0.004 0.005
  1     CuO            Calc 0.032 0.007          wt.%     0.024 0.005
  1     Nb2O3          Calc 0.006 0.017          wt.%     0.002 0.004
  1     MoO3           Calc 0.006 0.023          wt.%     0.003 0.010
  1     WO3            Calc 0.013 0.026          wt.%     0.003 0.007
  1     P2O5           Calc 0.000 0.000          wt.%     0.000 0.000
  1     SO3            Calc 2.971 0.144          wt.%     2.191 0.106
  1     CaO            Calc 74.178 0.421         wt.%    78.092 0.444
  1     MgO            Calc 3.124 7.130          wt.%     4.576 10.443
  1     K2O            Calc 1.112 0.058          wt.%     0.697 0.037
  1     BaO            Calc 0.018 0.076          wt.%     0.007 0.029
  1     Al2O3          Calc 4.561 1.745          wt.%     2.641 1.011
  1     Ta2O5          Calc 0.023 0.026          wt.%     0.003 0.003
  1     TiO2           Calc 0.168 0.029          wt.%     0.124 0.021
  1     ZnO            Calc 0.002 0.005          wt.%     0.001 0.004
  1     Ag2O           Calc 0.000 0.000          wt.%     0.000 0.000
  1     Cl             Calc 0.484 0.040          wt.%     0.805 0.067
  1     ZrO2           Calc 0.045 0.016          wt.%     0.022 0.008
  1     SnO2           Calc 0.667 1.039          wt.%     0.261 0.407
Element Table ====================================================
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