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Comparative analysis of cement composition using x-ray fluorescence spectroscopy: A case study of cement commercialized in Burundi

Steve De Cliff [1]*, Sylvestre Ndayiragije[2], James Kubwimana[3], Samuel Nihorimbere[4], Jean Paul Simbavimbere[5] and Abel Désiré Nshimirimana[6]

[1,2,3,4] Department of Chemistry, Faculty of Sciences, University of Burundi, 2 Avenue de l'Unesco, P.O. Box 2700, Bujumbura, BURUNDI
[5,6] Office Burundais des Mines et Carrières (OBM), 6 Avenue du Treize Octobre, Kabondo, Bujumbura, BURUNDI
**Corresponding Author: e-mail: steve.decliff@ub.edu.bi - Tel: +257-68 00 00 69*
ORCIDs: https://orcid.org/0009-0003-6390-7152 (De Cliff)

Abstract

Cement is a key material in the construction industry, and its quality significantly influences the strength and durability of structures. This study aims to analyze the chemical composition of different brands of cement commercialized in Burundi using Wavelength Dispersive X-ray Fluorescence (WD-XRF). The results are compared with international standards (ASTM C150-12 and EAS 18-1:2001) to assess compliance and quality. The Congolese made GLC 42.5N cement appears to meet key quality parameters, including a balanced C_3S/C_2S ratio and acceptable AM and SM values. The results provide insights into the cement performance characteristics, particularly their early and long-term strength development. However, further mechanical testing is recommended to assess durability.

Keywords: Cement, BUCECO, XRF; GLC, BBN, SIMBA, Dangote

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1. Introduction

Cement is a hydraulic binder i.e, it is finely ground inorganic material which, when mixed with water, forms a paste which sets and hardens by means of hydration reactions and processes and which after hardening, retains its straight and stability even under water. It is one of the most commonly used materials in the global construction industry, playing a crucial role in the strength and durability of structures (Sultan *et al*., 2023). It serves as a binding agent, hardening from a plastic state when mixed with water to form a rigid mass. This process, known as hydration, develops the paste's rigidity and gradually increases compressive strength (Salah, 2019). The understanding of the behavior of cement can lead to design of high-performance concrete and mortar for making solid and long-life structure (Kosmatka *et al*, 2002).

Various properties of cement are considerably influenced by oxide compositions. A typical Portland cement clinker is produced by mixing limestone, clay, sand and iron oxide and heating them at elevated temperatures in rotary kilns were converted to major and minor oxides form (Almabrok, 2014). The major oxides include CaO, SiO_2, Al_2O_3, and Fe_2O_3 whereas the minor oxides also include MgO, SO_3, and some alkali oxides (K_2O and Na_2O) (Punmatharith, 2010). Thus, during the proportioning of raw materials, the content of each oxide must be in the right quantity (Huntzinger *et al*, 2009). During burning (900 ºC to 1450ºC) and blending of raw materials, the chemical reactions take place and four principal compounds which are normally referred to as minerals are resulted. These include tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C3A) and tetracalcium luminoferrite (C_4AF) (Tab.1). Silicates play the key role in gaining the strength compared to other components. C_3S easily reacts with water, results in more heat of hydration and responsible for early strength development, whereas, C_2S react slowly, produces less heat of hydration and responsible for later strength development (Bhamere, 2016).

Table 1: Comparative table of clinker mineral compositions with chemical reactions and the role of each in the cement

Compound	Chemical Formula	Chemical Name	Formation Reaction	Role in Cement
Alite	$3CaO \cdot SiO_2$ (C_3S)	Tricalcium Silicate	$3CaO + SiO_2 \rightarrow C_3S$	Provides early strength
Belite	$2CaO \cdot SiO_2$ (C_2S)	Dicalcium Silicate	$2CaO + SiO_2 \rightarrow C_2S$	Develops long-term strength
Aluminate	$3CaO \cdot Al_2O_3$ (C_3A)	Tricalcium Aluminate	$3CaO + Al_2O_3 \rightarrow C_3A$	Responsible for rapid setting
Ferrite	$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ (C_4AF)	Tetracalcium Aluminoferrite	$4CaO + Al_2O_3 + Fe_2O_3 \rightarrow C_4AF$	Influences color and setting time

In Burundi, cement is sourced both domestically, from producers like BUCECO, and through imports from neighboring countries and other regions. The market includes a range of cements from various sources, such as Dangote Cement from Nigeria's Dangote Industries Limited, Simba Cement from Tanzania's Tanga Cement PLC, and GLC Cement from China's Great Lake Cement Company, which operates the former Kabimba cement plant in the Tanganyika province of the Democratic Republic of the Congo and acquired in October 2020. This diversity in supply ensures adequate availability but may pose challenges in quality monitoring. The Bureau Burundais de Normalisation et de Contrôle de la Qualité (BBN) oversees quality standards to prevent accidents and ensure product safety. In September 2023, 600 tons of GLC Cement were unloaded at Bujumbura port, but their sale was temporarily suspended by the BBN pending compliance analysis (ABP, 2023; IWACU, 2023). This incident raised questions about the standards used to take such a decision and the BBN's objectivity in issuing port clearance certificates.

It is in light of these issues that we have undertaken this study titled “Comparative Analysis of Cement Composition Using X-Ray Fluorescence Spectroscopy: A Case Study of Cements Commercialized in Burundi” in which we investigate key properties of above-mentioned Portland cements.

The properties of Portland cement are highly dependent on its clinker mineralogy. The Bogue equations allow for an estimation of the major clinker phases from the oxide composition (Taylor, 1997; Stutzman *et al*., 2014). This study applies these equations to cement available in Burundi to compare their potential performance characteristics. Furthermore, the computation of quality indices (LSF, SM, AM) provides an additional assessment of cement compliance with international standards (Mindess *et al*., 2003; Elbeyli *et al*., 2003; Huntzinger *et al*., 2009).

In the absence of a specific Burundian standard, results were compared against the EAS 18-1:2001 and ASTM C150-12 standards.

2. Materials and Methods

2.1. Sampling and Sample Preparation

All the cements concerned by this study were branded samples of cements purchased from the local market: BUCECO 32.5R, GLC 42.5N, Simba 42.5N, Dangote 42.5N and Dangote 42.5R. A specific quantity of cement sample was carefully ground into a fine powder. Eight grams of this powder were poured into a pre-cleaned pellet mold, which had been weighed using an analytical balance. Two grams of an organic binder (wax) were then added, and the mixture was homogenized for approximately five minutes using a Mixer/Mill 8000M. The homogenized sample was subsequently compressed under a pressure of 400 kN for about two minutes using a motorized hydraulic press (MP250, Maassen GmbH). The resulting fresh pellets had a diameter of 44 mm and a thickness of less than 10 mm. These pellets were then removed from the mold, ready for WD-XRF analysis of their chemical composition.

The samples analyzed in this study were solid pressed pellets, meeting all the necessary conditions for use with XRF equipment. These conditions aligned with the factory-configured SQX calibration library, made possible by the powerful Fundamental Parameters (FP) software. This software enables non-empirical calibration using a library of pure element standards, including metals, glass, oxides, and polymers, allowing for the quantitative analysis of unknown samples with near-empirical accuracy (Balasubramanian *et al*., 2016; Rousseau, 2013; Sherman, 1955; Spectroscopy, 2007). A key requirement of the FP method is that the pellet must be perfectly flat, highly smooth, and sufficiently thick to prevent incident X-ray beams from passing through. The preparation of the pressed pellets was conducted at the Burundian Office of Mines and Quarries (OBM) following the Rainner Schramm protocol (Schramm, 2012).

2.2. Analysis and Instrumental Specifications

In this research study, analyzes were carried out with a Supermini200, a sequential benchtop wavelength dispersive X-ray fluorescence (WD-XRF) spectrometer branded and manufactured by Rigaku Corporation (Figure 1). It operates under a Windows 10 environment with a built-in software based on the powerful of the Fundamental Parameters (FP) approach to calibration in XRF

(Sherman, 1955; Spectroscopy, 2007). Reliable quantitative analyses were achieved thanks to the performance of the following software features: an SQX software with the FP matching library, an SQX scatter FP method, a fused bead correction with a line overlap correction using theoretical intensities (LOCTI), and a Quant Scatter FP method. A Supermini200 is also equipped with the following technical operating built-in devices: a primary Zr-beam filter, a Pd-anode X-ray tube with a power of 200 W produced by a 50 kV and 4 mA generator. It also uses standard crystals of lithium fluoride LiF(200) and pentaerythritol PET(002). As for the detectors, the Supermini200 is equipped with a set of detectors based on the Pulsed Height Analyzer (PHA) with a scintillation counter (SC) for heavy metals, and a gas-flow proportional counter (F-PC) for light elements. All this ensures high sensitivity and high-speed counting.

Figure 1: The sequential benchtop wavelength dispersive X-ray fluorescence (WD-XRF) spectrometer, a Supermini200 manufactured and branded by Rigaku Corporation.

2.3. Mineralogical Phases and Key Quality Parameters

Mineralogical phases involved in cement are the C_3S (Alite), which plays a key role for early strength; the C_2S (Belite), which contributes to long-term strength, the C3A (Aluminate) that affects setting time and sulfate resistance, and the C_4AF (Ferrite) that influences color and hydration kinetics. When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite shall be calculated from the chemical analysis by using the following Bogue equations:

- Tricalcium silicate (Alite):

$$C_3S = 4.071[CaO] - 7.600[SiO_2] -6.718[Al_2O_3] - 1.430[Fe_2O_3] \quad (1)$$

- Dicalcium silicate (Belite):

$$C_2S = 8.6024[SiO_2] + 1.0784[Fe_2O_3] - 5.0683[Al_2O_3] - 3.0710[CaO] \quad (2)$$

- Tricalcium aluminate (Aluminate):

$$C_3A = 2.650[Al_2O_3] - 1.692[Fe_2O_3] \quad (3)$$

- Tetracalcium aluminoferrite (Ferrite):

$$C_4AF = 3.043[Fe_2O_3] \quad (4)$$

In cement quality assessment, the following quantities are key parameters to decide on the quality of cement: the Lime Saturation Factor (LSF) determines clinker balance, the Silica Modulus (SM) usually affects strength and setting, and the Alumina Modulus (AM) in turn, indicates resistance to sulfates. These quality parameters were computed based on the following formulas:

- Lime Saturation Factor:

$$LSF = \frac{[CaO]}{2.8\,[SiO_2] + 1.2\,[Al_2O_3] + 0.65\,[Fe_2O_3]} \quad (5)$$

- Silica Modulus:

$$SM = \frac{[SiO_2]}{[Al_2O_3] + [Fe_2O_3]} \quad (6)$$

The silica modulus provides information on the ratio between the quantity of silicate (from SiO_2) and that of aluminate (from Al_2O_3 and Fe_2O_3) contained in the cement or clinker (Ahmed *et al*., 2022). Its value can have a significant impact on the properties of the final cement. Generally, a higher silica modulus indicates a higher silica content relative to alumina. This can lead to a cement with lower reactivity, slower setting, and lower early strength. Conversely, a lower silica modulus indicates a higher alumina content, which can improve the reactivity and strength of the cement (Elbeyli *et al*., 2003). In general, the SM ranges from 1.8 to 3.4, and for common Portland cements, from 2.2 to 2.5.

- Alumina Modulus:

$$AM = \frac{[Al_2O_3]}{[Fe_2O_3]} \tag{7}$$

This is the ratio between the amount of alumina and iron oxide. It particularly concerns the raw mix that will be fired. It provides information on the mix's suitability for firing, as iron oxide is a flux. Therefore, the higher the iron content of the mix, the easier it will be to fire, but the less resistant it will be. RA generally varies from 1.2 to 2.5, and from 1.5 to 2 for standard Portland cements.

These indices provide additional insights into clinker chemistry and its implications for cement quality (Elbeyli *et al*., 2003; Ahmed *et al*., 2022; Kosmatka *et al.*, 2002).

3. Results and Discussion

3.1. XRF Results of Major and Minor Oxides

The table below presents the comparative results of XRF analysis for major oxides, including standards from ASTM C150-12 and EAS 18-1:2001.

Table 2: XRF results of major and minor oxides present in the investigated cements (BUCECO 32.R, Dangote 42.5N and 42.5R, GLC 42.5N and Simba 42.5N).

Oxides (%)	BUCECO 32.5R	GLC 42.5N	Dangote 42.5N	Dangote 42.5R	Simba 42.5N	ASTM C150-12	EAS 18-1:2001
CaO	54.550	62.167	73.500	72.600	73.700	60.0 - 67.0	60.0 - 67.0
SiO_2	23.035	18.533	12.933	13.533	12.467	17.0 - 25.0	19.0 - 25.0
Al_2O_3	7.175	5.880	3.040	3.303	3.623	2.5 - 6.0	3.0 - 8.0
Fe_2O_3	7.575	4.693	4.667	5.217	6.113	≤ 6.0	0 - 6.0
MgO	1.920	1.400	0.821	0.652	0.357	≤ 5.0	< 6.0
SO_3	3.015	1.870	3.900	3.283	2.297	≤ 3.0	< 4.0
K_2O	0.735	0.916	0.696	0.633	0.175	< 1.0	
P_2O_5	0.340	1.700	0.113	0.121	0.358		
MnO	0.245	1.127	0.096	0.085	0.078		
TiO_2	0.720	0.419	0.456	0.485	0.640		
SrO	0.070	0.169	0.047	0.137	0.173		

These results clearly show that lime content (CaO) for Dangote and Simba cements have higher CaO content, exceeding ASTM/EAS limits. This may affect setting time and soundness for that cement. The observed lower silica content (SiO_2) in all imported cements may impact long-term strength development. In turn, BUCECO cement has lower lime content and a higher alumina (Al_2O_3) and iron oxide (Fe_2O_3) content, potentially influencing setting time for that cement.

All the major oxides in the GLC cement brand were in the recommended ranges and limits (ASTM 2012, EAS 2001).

Apart from the major oxides, the presence of minor oxides such as K_2O, P_2O_5, MnO, TiO_2, and SrO also influences the properties of cement. All these minor oxides have been detected in all cement samples investigated. Potassium Oxide (K_2O) is present in low concentrations (0.175–0.916%) ; K_2O is one of the alkali oxides that can contribute to alkali-silica reactions (ASR), potentially causing expansion and cracking in concrete when reactive aggregates are present (Ahmed *et al*., 2022; Huntzinger *et al*., 2009). Phosphorus Pentoxide (P_2O_5) is detected at levels between 0.113% and 1.700% ; P_2O_5 can influence clinker formation and, when present in excess, may retard hydration, affecting cement strength development (Taylor, 1997; Stutzman *et al*., 2014). Manganese Oxide (MnO) was found in small amounts (0.078–1.127%) ; MnO can affect the coloration of cement and may improve its mechanical properties when adequately incorporated into the clinker (Kosmatka *et al*., 2002). Titanium Dioxide (TiO_2) is present in the range of 0.419–0.720% ; TiO_2 acts as a phase stabilizer in the clinker and may influence the hydration kinetics of cement (Elbeyli *et al*., 2003; Neville, 2011). Strontium Oxide (SrO) has also been detected in minimal quantities (0.047 - 0.173%), SrO is known to enhance the durability and resistance of cement to sulfate attacks, though its impact at these levels remains marginal (Kosmatka *et al*., 2002; Mindess *et al*., 2003).

The inclusion of these minor oxides, though present in small amounts, plays a role in modifying the chemical and physical behavior of cement. Their presence should be monitored to ensure they remain within acceptable limits for optimal cement performance.

3.2. Mineral Composition

The mineral composition of the cement is more useful than its oxide composition regarding determination of cement performance (Winter, 2016). The mineral composition of cement samples was computed based on Bogue's equations (1 to 4) and is presented in Table 3.

Our attempts of applying Bogues formulas yields negative values for C_2S and C_3A for some cements, suggesting that the Bogues formulas are not fully adapted to the specific compositions of these cements, which can occur if the composition does not meet the assumptions of these equations (e.g., excess of certain oxides or other phases not considered). Furthermore, the C_3S is very high in some cases, indicating a highly reactive clinker, favorable to rapid setting. To correct for outliers, one has to force negative values to zero to avoid physical inconsistencies. For this, two approaches were envisaged: the first approach involved re-evaluating the available CaO by considering the portion that could be combined in the form of $CaSO_4$ (anhydrite/gypsum) or free CaO. This approach effectively eliminates negative values, but other values such as those of C_3S, remain too high. The better approach involves recalculating the available CaO. Indeed, the problem with negative values may well stem from an incorrect estimation of free CaO, which can be combined in the form of $CaSO_4$ (calcium sulfate, from gypsum) and free CaO remaining after reaction with SiO_2, Al_2O_3, and Fe_2O_3.

With this approach, it then becomes possible to adjust the available CaO by subtracting a portion of the SO_3 (since SO_3 comes mainly from gypsum, which fixes some of the CaO in the form of $CaSO_4$). The estimate follows the relationship:

$$[CaO]_{Available} = [CaO]_{Total} - 0.7[SO_3] \quad (8)$$

Calculating the minerals using this approach, still using Bogue's formulas with this adjusted CaO, yields the results shown in Table 3.

Table 3: The computed clinker phase composition and quality parameters computed for each cement sample

Cement	Clinker phase composition			
	C_3S (%)	C_2S (%)	C_3A (%)	C_4AF (%)
BUCECO 32.5R	0.00	81.59	6.20	23.05
GLC 42.5N	60.69	7.37	7.64	14.28
Dangote 42.5N	162.72	0.00	0.16	14.20
Dangote 42.5R	153.70	0.00	0.00	15.88
Simba 42.5N	165.66	0.00	0.00	18.60
ASTM C152-12 EAS EAS 18 2001	45 - 65	7 - 32	8 - 12	10 – 11

From results of Table 3, it appears that the correction applied to the available CaO by accounting for SO_3 provided a more realistic estimation of the clinker phases, mitigating inconsistencies observed in initial calculation attempts (not shown) (Elbeyli *et al*., 2003). However, the locally produced cement BUCECO 32.5R exhibits a dominant C_2S phase (81.59%), characteristic of slow hydration and long-term strength development (Kosmatka *et al*., 2002).. The absence of C_3S suggests a lower early strength. The cement brands Dangote 42.5N, 42.5R, and Simba 42.5N show exceptionally high C_3S values, indicative of rapid strength gain, making them suitable for applications requiring high early strength. The low or absent C_2S phase suggests limited late strength gain compared to other cements (Taylor, 1997).

Against all expectations, it is GLC 42.5N cement, which had been hit by a marketing refusal measure by the standards authority, that presents a balanced C_3S/C_2S ratio, indicating good early and late strength performance (Neville, 2011). Fortuntely, the C_3A content is relatively low across all samples, which may contribute to improved sulfate resistance, particularly for Dangote 42.5R and Simba 42.5N, where C_3A is nearly absent (Mindess *et al*., 2003).

The results provide insights into the cement performance characteristics, particularly their early and long-term strength development (Kosmatka *et al.*, 2002; Neville, 2011; Taylor, 1997; Stutzman *et al*., 2014).

3.3. Quality Parameters Assessment

The Lime saturated factor (LSF), the silica modulus (SM) and the Alumina modulus (AM) were also determined. Each of these parameters influences performance of cements and is often used for control purposes (Taylor, 2014; Mengistu, 2010). These quality parameters were computed by using equations 5 to 8 are presented in Table 4 and Figure 2 for cements investigated in this study.

Table 4: The computed clinker phase composition for five most popular cement brands commercialized in Burundi.

Cement	Quality parameters		
	LSF	SM	AM
BUCECO 32.5R	0.86	2.02	0.95
GLC 42.5N	0.92	2.16	1.25
Dangote 42.5N	1.12	2.45	0.65
Dangote 42.5R	1.08	2.38	0.63
Simba 42.5N	1.10	2.32	0.59
ASTM C152-12	0.92 - 0.98	2.3 - 2.7	1.3 -2.5

A critical review of the quality parameters reveals that the LSF values for Dangote and Simba cements exceed the standard range (0.92 - 0.98), indicating an excess of CaO, which may have significant implications for cement performance (soundness and setting time). For the SM parameter, all samples fall within the standard range (2.3 - 2.7) except for BUCECO 32.5R, which has a slightly lower SM, potentially affecting its reactivity. However, the lower AM values for Dangote and Simba indicate a higher Fe_2O_3 content, making these cements easier to burn but less resistant to sulfate attacks. High LSF values indicate an excess of CaO relative to the available SiO_2, Al_2O_3, and Fe_2O_3, which can lead to the formation of free lime (CaO). The presence of excess free lime increases the risk of unsoundness and delayed expansion due to hydration after setting (Taylor, 1997).

Among all the cement samples analyzed, only GLC 42.5N has quality parameter values within acceptable limits, indicating a cement of good overall quality.

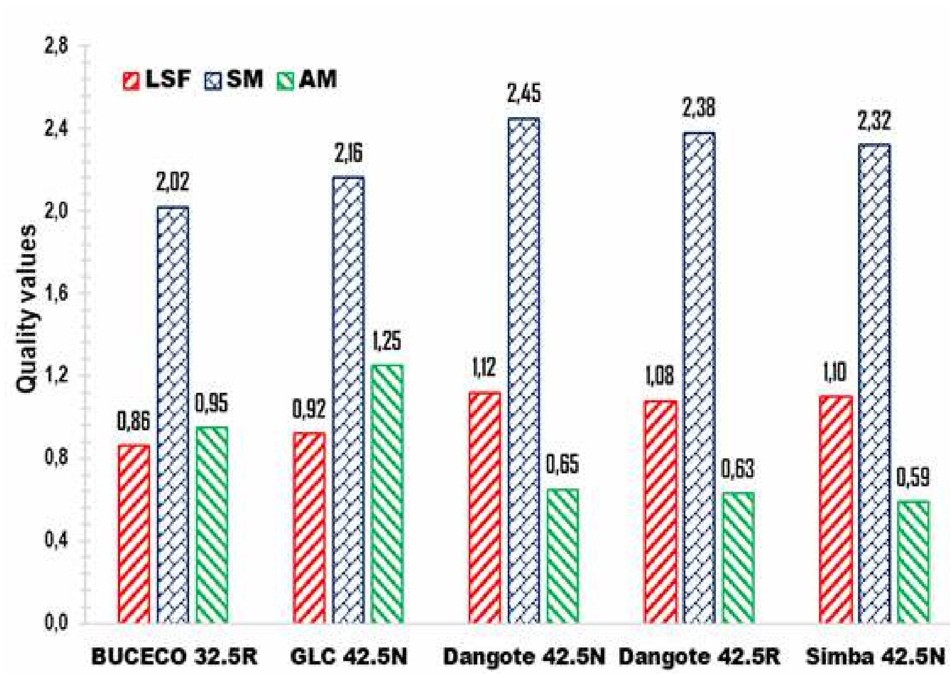

Figure 2: Lime saturated factor (LSF), silica modulus (SM) and Alumina modulus (AM) for five most popular cement brands commercialized in Burundi

Additionally, high LSF values can affect the clinker phase composition by promoting an overabundance of C_3S while reducing the proportion of C_2S. While this favors early strength development, it may lead to brittleness and reduced long-term durability. The lower levels of C_2S observed in Dangote and Simba cements confirm this shift towards a more reactive but potentially less durable cement (Kosmatka *et al*., 2002). From a practical standpoint, cement with a high LSF requires strict quality control during manufacturing to ensure complete clinkerization and avoid excessive free lime content (Neville, 2011). Further mechanical testing is recommended to assess whether the high LSF values negatively impact cement durability in real-world applications.

3.4. Recent Research Insights

Recent studies have provided further understanding of the limitations of Bogue calculations and the role of minor oxides in cement performance. Variability in clinker phases has been shown to impact cement performance significantly, highlighting the need for new predictive models beyond Bogue equations (Sciencedirect, 2024). Moreover, thermodynamic simulations have been used to assess the role of minor oxides such as MnO and TiO_2 in clinker formation, reinforcing the importance of their controlled presence (Springer, 2020). The effect of alkali oxides, particularly K_2O, on durability and ASR susceptibility has also been examined (Ahmed *et al*., 2022; Huntzinger *et al*., 2009).

Advancements in neural networks have been applied to optimize sulfate content in cement, demonstrating significant improvements in compressive strength predictions (MDPI, 2024). Additionally, graphene oxide has been identified as a promising additive to enhance cementitious composites (De Gruyter, 2021). These insights confirm that a more comprehensive approach is required to evaluate cement quality beyond oxide composition alone.

4. Conclusion

This study provides an in-depth assessment of the chemical and mineralogical composition of various cements available in Burundi. The results reveal significant variations among brands, particularly in the clinker phase composition and quality indices. Contrary to regulatory concerns, GLC 42.5N appears to meet key quality parameters, including a balanced C_3S/C_2S ratio and quality parameter values within acceptable limits, indicating a cement of good overall quality. The decision to suspend its commercialization may require further mechanical testing for validation.

The interpretation of cement chemical analysis data in relation to a given standard is often complex and cannot, on its own, determine cement quality. A few out-of-range values for a major oxide do not necessarily indicate that the cement is of poor quality and should therefore be rejected. A more holistic approach, incorporating mechanical and durability testing, is necessary to make a comprehensive assessment of cement performance.

Future research should focus on mechanical strength tests and durability studies to complement the chemical analysis.

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Biographical notes

Steve De Cliff and **Sylvestre Ndayiragije** are both of the Department of Chemistry, University of Burundi.

James Kubwimana is now at the Ecole Normale Supérieure (ENS), Bujumbura, Burundi.

Samuel Nihorimbere is now at the Bureau Burundais de Normalisation et de Contrôle de la Qualité (BBN).

Jean Paul Simbavimbere and **Abel Désiré Nshimirimana** are both of the Office Burundais des Mines et Carrières (OBM), Bujumbura, Burundi.