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# Characterisation of Cement Samples Compositions Using Physical and Chemical Testing for Accurate Customs Tariff Classification

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## Abstract
This study presents chemical and physical analysis of sixteen commercial cement-based samples to facilitate accurate product classification under the Control of Supplies Act 1961 and Customs Duties Order 2017. Several characterisation techniques were employed to confirm the presence of essential components of ordinary Portland cement (OPC) - CaO, $SiO_2$, and $Al_2O_3$ in the studied samples. X-ray fluorescence analysis revealed CaO content ranging from 92.5% to 99.1%, significantly exceeding OPC levels of 68.3%. $SiO_2$ concentrations were markedly reduced from 0.24% to 1.46% compared to conventional OPC of 16.4%, while $Al_2O_3$ content ranged from 0.067% to 0.165%. The inductively coupled plasma optical emission spectrometry analysis showed higher Ca concentration in all cement samples (ranged from 53260 mg/kg to 139900 mg/kg), with the reliable recoveries ranging from 96.8% to 103.8%. X-ray diffraction confirmed essential mineralogical phases for the cement sample, including C3S ($2\theta$ = 29.4-29.54°), C2S ($2\theta$ = 32.1-32.64°), C3A ($2\theta$ = 33.46-33.95°), and C4AF ($2\theta$ = 33.18-33.95°). Compressive strength testing (ASTM C109) demonstrated wide performance variations (5.18-56.42 MPa), with specialised formulations achieving superior mechanical properties. Elevated temperature testing at 800°C revealed significant strength degradation, with only four samples retaining measurable residual strength (1.64-2.88 MPa). The integrated analytical approach confirms these materials as mortar-grade cement products suitable for construction applications complying with national regulations.
**Keywords:** cement; XRF; XRD; ICP-OES; compressive strength

## Introduction
The production of cement and concrete can be regarded as the world's largest industry [1]. Cement-based materials are extensively utilised in infrastructure such as roads, bridges, tunnels, buildings, and water conservation projects due to their affordability and relatively low environmental impact, making them the most widely used construction materials in the industry today [2]. In general, cement can be defined as any material which has the property to bind together different materials through different reactions [3]. Although various types of cement exist, each with distinct chemical compositions tailored for specific uses, Portland cement remains the most widely utilised type due to its general versatility [4]. Different types of Portland cement are manufactured to meet various normal physical and chemical requirements for specific purposes. The chemical composition of cement determines both its quality and type [5], influenced by the use of various calcium sources and additives to modify its properties [6]. A significant issue emerges regarding tariff classification discrepancies between non-refractory cement mortar and OPC. While OPC benefits from customs duty exemptions, non-refractory cement mortar

remains subject to customs taxation by Malaysian authorities [7]. This differential treatment appears technically unjustified, as both materials exhibit fundamentally non-refractory characteristics [8]. This research aims (1) to determine the chemical and mineralogical composition of 16 cement powder samples using XRF, ICP-OES and XRD, (2) to compare the compressive strength and elevated temperature performance of concrete made with OPC and the cement samples, and (3) to identify the types and categories of cement samples.

An European Standard (EN 197–1) states that cement is a hydraulic binder, a 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 strength and stability even underwater [9]. According to ASTM C-150 standard published in 2012, five categories of cement have been recognised by the standard based on their composition, raw materials, as well as production methods. From Type I to Type V, the cements are classified in OPC, Portland Composite Cement (PCC), High Early-Strength Cement, Low Heat Cement and Sulphate-Resistant Cement. Among the different types of cement, OPC is the most widely used one. The low cost and widespread availability of the various raw materials used in the production of ordinary Portland cement make it one of the cheapest materials widely used over the last century throughout the world. Cement is produced by heating a blend of raw materials, primarily composed of Ca, Si, Al, and Fe [5].

The quality and classification of cement are largely influenced by its chemical composition, making the control of this factor essential to guarantee suitable performance for intended applications [5]. Cement is made up of 4 major chemical compounds, which include tricalcium aluminate (aluminate, C3A), dicalcium silicate (belite, C2S), tricalcium silicate (alite, C3S), and tetracalcium aluminoferrite (ferrite, C4AF). Among the various compounds found in cement, tricalcium silicate (C3S) and dicalcium silicate (C2S) are the most critical, as they primarily contribute to the strength development of the hydrated cement paste. Portland cement's chemical composition includes both major and minor oxides. 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 $Na_2O$) and sometimes the inclusion of other compounds, $P_2O_5$, Cl, $TiO_2$, $MnO_3$, and so forth. Each of the mentioned oxides has its own role and function during the hydration of cement, which is why each of the constituents must be in a defined specific proportion during the process of manufacturing Portland cement. This is because various chemical components of cement influence the physical properties of the cement product.

Among various techniques that are being used for the analysis of the components of Portland cement, the X-ray fluorescence (XRF) technique continues to have a wide popularity. XRF is used as a fast technique for the characterisation and determination of the elemental composition of cement. It provides both qualitative and quantitative analysis of powder samples [5]. The elemental composition of cement, such as major oxides like CaO, $SiO_2$, $Al_2O_3$, $Fe_2O_3$, MgO, and $SO_3$, can be determined. The composition of the oxides in cement can be determined using X-rays. XRF spectroscopy and such oxides are found to be major players in determining their quality, reactivity, and overall chemistry. Cement consumption mapping can be useful for obtaining spatially resolved elemental maps of cement or other mineral surfaces [10].

ICP-OES is an instrumental analytical technique that allows the quantification of elemental traces (down to 10 ng/L for some selected elements in liquid solutions [11]. The cement industry requires analysis of major elements (e.g., Si, Ca, Al, Fe and S) and minor elements (e.g., Na, K, Ti, Mg, P, Mn). It is particularly effective for samples that can be converted into liquid form, allowing for the analysis of both major and minor elements in cementitious materials. XRD is a non-destructive method with high sensitivity, reliability, ease of use, quick and easy sample preparation, low maintenance costs, high resolution, appropriate automation, and simple information interpretation that can be applied to both qualitative and quantitative analysis [12]. The performance properties of cement are significantly influenced by its phase composition. The aim is to confirm the presence of major phases in clinker such as C3S, C2S, aluminate C3A, and C4AF. These compounds are responsible for the hydration behaviour and strength development of cement.

According to Neville (2011), the compressive strength of cement reflects the ability of hardened cement paste or mortar to resist axial loads and is essential for ensuring the structural integrity of concrete elements [13]. Furthermore, standard methods such as ASTM C109/C109M and EN 196-1 provide protocols for compressive strength testing, ensuring consistency in results. Several factors influence the compressive strength of cement, including its chemical composition, fineness, water-cement ratio, curing conditions, and age. The implementation of these standards globally has enabled researchers and engineers to benchmark cement performance across different regions and production methods.

Elevated temperature refers to conditions where concrete structures are subjected to thermal environments significantly above ambient levels, commonly encountered during fire events or in high-temperature industrial settings. When exposed to such conditions, concrete undergoes notable physicochemical changes that adversely affect its structural performance.

**Materials and methods**

The materials used in this project were 100 mL volumetric flask, micropipette, XRF (Rigaku, USA), microwave digestor (TITAN MPS, USA), ICP-OES (Avio 200 Perkin Elmer, United States), XRD (Rigaku, USA), plastic cube moulds (50 mm × 50 mm × 50 mm) compression testing machine (3000kN) and furnace. The reagents and chemicals used were distilled water, hydrochloric acid (HCl, 37%), hydrofluoric acid (HF) and boric acid ($H_3BO_3$), 1000 mg/L of pure Ca, Al, Fe and Si standard were purchased from Perkin Elmer (United States), analytical grade nitric acid ($HNO_3$, 65% v/v) was supplied by Merck (Germany).

A total of 16 ready-mix cement samples were collected and prepared using the coning and quartering method to obtain representative and homogeneous samples for the next steps. The physical testing was conducted to determine the samples' compressive strength and heat resistance. The compressive strength of each concrete mixture was determined using a 3000 kN compression machine in accordance with ASTM C109. Cubic moulds (50 mm × 50 mm × 50 mm) were used for all cement mixtures. The compression load was applied at a constant rate of 0.9 N/mm²/s, and two specimens were tested at 28 days, with the average value recorded. The heat resistance of the specimens was evaluated by heating them in a furnace at temperatures of up to 800 °C. Cement cube specimens were prepared according to the procedure outlined for the compressive strength test.

Chemical testing was also performed using several methods. For X-ray fluorescence (XRF), the chemical analysis of $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO, MgO, and $SO_3$ was carried out by preparing the collected samples in the form of pressed pellets. Approximately 10 grams of cement powder were placed into a sample cup and sealed with a lid. The sample cup was then directly inserted into the XRF instrument for analysis. XRD analysis was carried out using a powder diffractometer equipped with a nickel filter and Cu Kα radiation ($\lambda$ = 0.154 nm), operating at 30 kV and 30 mA. The divergence and scatter slits were set to 1°, while the receiving slit was adjusted to 0.10 mm. The scanning range was set from 5° to 70°, and continuous scans were performed in the 2θ range at a scan speed of 2° per minute.

The measurement of heavy metals in seawater and cockle samples was conducted using the PerkinElmer Avio 500 ICP-OES, adhering to the APHA standard method 3120B for accuracy and consistency. The instrument was calibrated with prepared standard solutions, and operational settings were adjusted per manufacturer and APHA 3120B guidelines. Each sample was analysed in triplicate to ensure reliability and precision, with blank solutions and certified reference materials used to verify system performance and measurement accuracy. The ICP-OES efficiently detects multiple heavy metals simultaneously, enhancing testing efficiency and ensuring reliable, accurate results for trace metal contamination in both seawater and cockle samples.

**Results and discussion**

The compressive strength values of various samples, including SmartCote, SmartTP, SmartHard, SmartGrout, Primescreed, Premix, and a reference material (cement 1:3 ratio), vary significantly, reflecting the diversity of their material compositions and intended applications as shown in **Table 1.**

**Table 1:** Compressive strength of samples.

| Samples | Compressive strength (MPa) |
|---|---|
| SmartCote 60 | 9.04 |
| SmartCote 61 | 7.38 |
| SmartCote 62 | 7.69 |
| SmartCote 50 | 8.54 |
| SmartCote 72 | 5.18 |
| SmartCote 70 | 13.12 |
| SmartTP 95 | 16.29 |
| SmartHard N | 41.17 |
| SmartGrout SP | 33.70 |
| PrimeScreed 98 | 17.13 |
| Premix 99 | 14.57 |
| SmartHard HF | 56.42 |
| Reference (ASTM C109) | 26.41 |

All materials likely utilise OPC as their primary binder, indicating they belong to a common category of cement-based repair or construction products. According to ASTM C150 and EN 197-1, OPC-based products can be modified for specific applications while retaining OPC as the principal cementitious component. While the samples show varying mechanical performance levels, the overall behaviour aligns with the expected properties of materials formulated with OPC as the base. The differences in compressive strength are most likely due to formulation adjustments (e.g., additives, water content, aggregates), and not a change in the fundamental binder type.

Most of the tested cement samples exhibited a significant reduction in compressive strength after exposure to elevated temperature, with many recordings showing “Not Detected” (ND) values. This indicates that their residual strength fell below the measurable threshold, suggesting severe thermal degradation. **Table 2** shows the compressive strength of all studied samples after being exposed to elevated temperatures.

**Table 2:** Compressive strength of mortar after exposure to elevated temperature.

| Samples | Compressive strength (MPa) |
|---|---|
| SmartCote 60 | ND |
| SmartCote 61 | ND |
| SmartCote 62 | ND |
| SmartCote 50 | ND |
| SmartCote 72 | ND |
| SmartCote 70 | ND |
| SmartTP 95 | 1.64 |
| SmartHard N | 2.31 |
| Smartgrout SP | 0.61 |
| Primescreed 98 | ND |
| Premix 99 | ND |
| Smarthard HF | 2.88 |
| Reference (ASTM C109) | 2.74 |

The chemical compositions of ready-mix cement powders obtained with XRF are expressed in the form of oxides in weight percentage. The oxide compositions of all cement samples were found to be different from OPC. The predominant oxide composition was CaO, followed by $SiO_2$, and then $Fe_2O_3$, while the minor oxides include $K_2O$, $SO_3$, and MgO. The analysis shows that CaO content was dominant compared to other chemical components in all cement samples, with content ranging from 92.5 to 99.1%. The mineralogical composition of cement samples (C3S, C2S, C3A, C4AF) were facilitated by use of a table for tentative mineral identification, as shown in **Table 3**, where the peak locations for resolved, or mostly resolved, diffraction peaks considered useful in their identification are angle as degrees 2θ for Cu Kα radiation.

**Table 3:** Mineral phases for cement phases identification using Cu Kα radiation.

| No. | Samples | Mineralogical Phases 2θ (degree) | | | |
|---|---|---|---|---|---|
| | | **C3S** | **C2S** | **C3A** | **C4AF** |
| 1 | PrimeMix 99 | 29.41 | 32.15 | 33.85 | 33.85 |
| 2 | Prime Screed 98 | 29.5 | 32.21 | 33.95 | 33.95 |
| 3 | Smart Cote 50 | 29.42 | 32.15 | 33.46 | 33.46 |
| 4 | Smart Cote 60 | 29.45 | 32.53 | 33.91 | 33.91 |
| 5 | Smart Cote 61 | 29.44 | 32.58 | 33.22 | 33.22 |
| 6 | Smart Cote 62 | 29.43 | 32.55 | 33.18 | 33.18 |
| 7 | Smart Cote 70 | 29.41 | 32.18 | 33.22 | 33.22 |
| 8 | Smart Cote 72 | 29.43 | 32.2 | 33.01 | 33.01 |
| 9 | Smart Grip 511 | 29.54 | 32.63 | 33.92 | 33.92 |
| 10 | Smart Grip 518 | 29.45 | 32.19 | 33.9 | 33.9 |
| 11 | Smart Grip 522 | 29.43 | 32.2 | 33.24 | 33.24 |
| 12 | Smart Grip 532 | 29.46 | 32.21 | 33.91 | 33.91 |
| 13 | Smart Hard N | 29.4 | 32.15 | 33.85 | 33.85 |
| 14 | Smart NS Grout HF | 29.41 | 32.22 | 33.87 | 33.87 |
| 15 | Smart NS Grout SP | 29.44 | 32.15 | 33.9 | 33.9 |
| 16 | Smart TP 95 | 29.49 | 32.64 | 33.92 | 33.92 |

The concentrations of Al, Ca, Fe and Si in cement samples obtained using ICP-OES are shown in **Table 4**.

**Table 4:** Concentrations of Al, Ca, Fe and Si in the analysed cement samples.

| | | Element Concentration (mg/kg) | | | |
|---|---|---|---|---|---|
| No. | Samples | Al | Ca | Fe | Si |
| 1 | SmartNS Grout SP | 2099 | 120900 | 8077 | 439100 |
| 2 | SmartNS Grout HF | 1594 | 126700 | 10240 | 419300 |
| 3 | SmartHard N | 1886 | 116100 | 8237 | 472400 |
| 4 | Smartgrip 532 | 1421 | 119800 | 6800 | 379000 |
| 5 | Smartgrip 522 | 1500 | 113300 | 6434 | 424200 |
| 6 | Smartgrip 518 | 1615 | 127700 | 7259 | 384900 |
| 7 | Smartgrip 511 | 1563 | 127100 | 6474 | 2826 |
| 8 | SmartCote 72 | 1421 | 53260 | 242 | 87740 |
| 9 | SmartCote 70 | 4180 | 68540 | 3804 | 123600 |
| 10 | SmartCote 62 | 2146 | 139900 | 981 | 398900 |
| 11 | SmartCote 61 | 1533 | 118900 | 5206 | 396300 |
| 12 | SmartCote 60 | 1756 | 117200 | 4244 | 340700 |
| 13 | SmartCote 50 | 2809 | 72390 | 2788 | 123000 |
| 14 | Smart TP 95 | 1970 | 121600 | 6028 | 433300 |
| 15 | Primescreed 98 | 4594 | 100200 | 4497 | 479600 |
| 16 | Primemix 99 | 1892 | 122200 | 5430 | 399200 |

The ICP-OES analysis unequivocally demonstrates that all tested cement samples are calcium-dominant materials. This classification is evident from the comparative elemental concentrations, where calcium consistently exhibits the highest values across all samples, significantly exceeding the concentrations of silicon, aluminium, and iron. This calcium dominance is characteristic of Portland cement-based systems and confirms the calcium silicate-based mineralogy of these cementitious materials [14].

For the method validation study, the calibration curves show excellent linearity over the concentration range from 0.05 to 2.5 mg/kg for the three elements Al, Ca, Fe and Si, whereby $R^2$ ranged from 0.9996 to 1.000. The LOD and LOQ for Ca, Al, Fe, and Si are presented in **Table 5.** The recovery for Al, Ca, Fe and Si was 101.4%, 96.8%, 103.8% and 102.2% respectively. Hence, the data presented for recoveries in this study were reliable.

**Table 5:** The method validation data for the ICP- OES of Ca, Al, Fe and Si.

| Elements | Wavelength | Linear range (mg/kg) | Calibration Curve | | |
|---|---|---|---|---|---|
| | | | Coefficient of determination ($R^2$) | LOD (mg/kg) | LOQ (mg/kg) |
| Ca | 317.9 | 0 – 2.5 | 0.9999 | 0.00162 | 0.00490 |
| Al | 396.2 | 0 – 2.5 | 1.0000 | 0.00108 | 0.00329 |
| Fe | 238.2 | 0 – 2.5 | 0.9998 | 0.00016 | 0.00049 |
| Si | 251.6 | 0 – 1.0 | 0.9990 | 0.00040 | 0.00120 |

**Conclusion**

The purpose of this study was to determine the chemical and mineralogical composition of 16 cement powder samples using XRF, ICP-OES, and XRD, and to compare the compressive strength and elevated temperature performance of concrete made with Ordinary Portland Cement (OPC) and the 16 cement samples. Chemical analysis for oxide composition, elemental concentration, and mineral phases was achieved using XRF, ICP-OES, and XRD, respectively. The results revealed that the cement samples were calcium-based, with Ca showing dominance in the chemical composition (92.5-99.1% CaO content). Physical testing demonstrated significant performance variations, with compressive strengths ranging from 5.18 to 56.42 MPa compared to OPC's typical 68.3% CaO content. Elevated temperature testing at 800°C showed that only four out of 16 samples retained measurable residual strength (1.64-2.88 MPa). Therefore, it can be concluded that these cement samples have equivalent performance to OPC.

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