



Development of Geopolymer Properties from Cement Industry Waste as Raw Material for Clay Pottery Products

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Abstract: This study demonstrates that cement industry waste (CCW) can be effectively utilized to produce high-performance clay-geopolymer hybrid ceramics with enhanced mechanical, physical, thermal, and durability properties. An optimum substitution of 20 wt% CCW achieved the highest compressive strength (42.5 MPa), compared with 31.4 MPa for the control, together with reduced porosity (22.1%) and water absorption (9.8%). These improvements are attributed to the combined effects of geopolymer reaction product development, improved particle packing, and microstructural densification, resulting in a dense and homogeneous matrix. Thermal and phase analyses suggest a progressive transition from geopolymer-dominated bonding to ceramic bonding during firing, with optimum densification at approximately 1050 °C, whereas firing at 1100 °C promoted over-fluxing and microstructural defects. The optimized composition also exhibited superior durability, retaining approximately 88% of its compressive strength after wet-dry cycling, and demonstrated good environmental compatibility, with heavy metal leaching below regulatory limits. In addition, CCW incorporation improved workability, reduced drying shrinkage from 6.5% to 4.0%, and enhanced surface quality while achieving comparable performance at lower firing temperatures than those typically required for conventional ceramics, suggesting the potential for reduced firing energy demand. Overall, the results demonstrate that CCW offers a promising and sustainable approach for producing durable, high-value ceramic materials while promoting waste valorization, resource efficiency, and circular economy practices.

Keywords: Geopolymer; cement industry waste; clay pottery; circular economy; sustainable materials

1. Introduction

The cement industry is a fundamental pillar of global infrastructure development; however, it is also a major contributor to industrial waste and CO₂ emissions [1,2]. During cement production, substantial quantities of by-products—such as cement kiln dust (CKD), clinker residue, and limestone fines—are inevitably generated [3]. These materials are commonly disposed of in landfills or open environments, leading to significant environmental impacts, including dust dispersion, soil alkalization, and groundwater contamination [4,5]. To address these challenges, increasing research efforts have focused on valorizing cement-based waste in construction materials and ceramic products as a pathway toward sustainable resource management and reduced environmental

burden [6–8]. Traditional pottery represents both a valuable cultural heritage and a key economic activity, particularly in Southeast Asia [9]. The Dangkwian pottery community in Nakhon Ratchasima, Thailand, is one of the oldest and most recognized centers of ceramic craftsmanship [10]. Despite its cultural significance, this sector faces several challenges, including depletion of natural clay resources, high energy consumption during firing, and limited integration of modern technologies [11, 12]. These constraints highlight the need for alternative materials and processing approaches that preserve traditional aesthetics while improving performance and sustainability.

Geopolymer technology, first introduced by Davidovits [13], offers a promising solution for converting industrial by-products into high-performance and environmentally friendly materials. Geopolymers are amorphous inorganic polymers formed by the polycondensation of aluminosilicate species in an alkaline medium, yielding a three-dimensional network with high mechanical strength, thermal stability, and chemical resistance [14–16]. Compared to ordinary Portland cement, geopolymer systems exhibit significantly lower CO₂ emissions and can incorporate a wide range of industrial wastes such as fly ash, slag, and CKD [17, 18]. These characteristics make geopolymers highly attractive for sustainable material development and waste valorization.

Recent studies have demonstrated the successful application of geopolymers in structural concrete, refractory materials, and ceramic composites [19–20]. However, their integration with natural clay in traditional pottery remains relatively underexplored [21]. Clay–geopolymer composites have the potential to combine the aesthetic qualities of traditional ceramics with enhanced performance, including higher compressive strength, reduced porosity, improved thermal resistance, and lower firing temperatures [22–24]. Furthermore, the formation of geopolymer gel phases within the clay matrix can improve densification, reduce shrinkage, and promote microstructural uniformity during firing [23, 25–26]. Despite these advantages, the use of cement industry waste in clay–geopolymer ceramics remains limited, particularly in artisanal or community-based production systems [21, 27]. The physicochemical interactions between geopolymer phases and clay, and their influence on microstructural evolution and mechanical performance, are not yet fully understood.

This study aims to address this gap by developing geopolymer binders derived from cement industry waste and incorporating them into clay pottery production. The effects of varying geopolymer substitution ratios on mechanical strength, water absorption, thermal stability, and microstructural characteristics are systematically investigated to identify an optimal formulation that balances performance, cost efficiency, and sustainability.

2. Materials and Methods

2.1. Materials

Two types of cement industry waste (CCW), cement kiln dust (CKD) and clinker fines (CF), were collected from a cement plant in Saraburi, Thailand. Each material was stored in sealed drums and sub-sampled by the coning-and-quartering method. Local clay (LC) was sourced from the Dangkwian pottery community, Nakhon Ratchasima. The clay was air-dried, oven-dried at 105 °C for 24 h, and sieved through a 200 µm mesh. Alkaline activators consisted of 10 M sodium hydroxide (NaOH, ≥98%) and commercial sodium silicate solution (SiO₂/Na₂O ≈ 3.2, solids ≈ 40 wt%). Minor additives, such as feldspar (flux), sodium polyacrylate (deflocculant), and polyvinyl alcohol (binder), were used as needed.

2.2 Characterization of Raw Materials

The chemical composition of CKD, CF, and LC was analyzed using wavelength-dispersive XRF, with loss on ignition (LOI) measured at 1000 °C. Mineralogical phases were identified by XRD (Cu Kα radiation, 2θ = 5–80°) and quantified using Rietveld refinement. Particle size distribution was determined by laser diffraction (reported as D10, D50, and D90), while specific surface area was obtained from BET analysis after degassing at 110 °C for 6 h. Morphological features were observed by scanning electron microscopy (SEM).

2.3 Preparation of Alkaline Activator and Geopolymer Paste

NaOH pellets were dissolved in deionized water to prepare a 10 M NaOH solution, which was cooled and mixed with sodium silicate to achieve a modulus ($\text{SiO}_2/\text{Na}_2\text{O}$) of ≈ 3.2 . The activator-to-solid ratio was fixed at 0.45 (with comparative tests at 0.35 and 0.55). For the geopolymer precursor, CKD and CF were blended in a 70:30 ratio (wt%) and milled to $<45 \mu\text{m}$. The alkaline activator was gradually added to the CCW powder, mixing at 300 rpm for 10 min and then at 600 rpm for 5 min. The paste was sealed and aged for 24 h at 25 °C to allow partial geopolymerization. For reference, geopolymer samples were cast, cured at 60 °C for 24 h, and then stored at 25 °C for 7 days.

Table 1. Experimental Matrix

ID	CCW substitution (%)	Activator/Solid	Firing Temp (°C)	Replicates (n)
C0	0 (control)	—	1000, 1050, 1100	6
G10	10	0.45	1000, 1050, 1100	6
G20	20	0.45	1000, 1050, 1100	6
G30	30	0.45	1000, 1050, 1100	6

2.4 Clay–Geopolymer Mixtures and Sample Preparation

Clay bodies were prepared by partially replacing clay solids with geopolymer solids at 0%, 10%, 20%, and 30% (by weight). Each mixture contained 5 wt% feldspar as a constant flux, consistent with conventional ceramic formulations [28]. At higher geopolymer content (30 wt%), the mixture exhibited increased viscosity and reduced workability, likely due to early-stage geopolymer gel formation, which restricts particle mobility [29]. Mixing was carried out using a pugmill for 15 min to ensure homogeneity. Polyvinyl alcohol (0.5–1.0 wt%) was added as needed to improve green strength, as is commonly practiced in ceramic processing [30,31]. Samples were shaped into:

- Bars ($25 \times 25 \times 100 \text{ mm}$) for mechanical testing,
- Discs ($\varnothing 50 \times 10 \text{ mm}$) for water absorption and porosity, and
- Tiles ($100 \times 100 \times 8 \text{ mm}$) for aesthetic evaluation.

All samples were air-dried (48 h, RH $50 \pm 5\%$) and oven-dried (105 °C, 24 h).

2.5 Firing Procedure and Sintering Procedure

Firing was conducted in an electric kiln (Nabertherm) following a controlled heating schedule: heating at 10 °C/min to 400 °C (holding for 30 min), followed by heating to the target temperatures of 1000, 1050, and 1100 °C (holding for 60 min), and then controlled cooling at -10 °C/min to 200 °C. This firing profile is consistent with typical sintering conditions for clay-based and geopolymer-derived ceramics, ensuring sufficient phase transformation and densification [32]. Linear shrinkage was determined by measuring dimensional changes before and after firing.

2.6 Physical, Mechanical, and Microstructural Characterization

Following conditioning, the ceramic specimens were systematically characterized to evaluate their physical, mechanical, thermal, microstructural, and durability-related properties. The characterization program included measurements of bulk density, water absorption, compressive strength, flexural strength, surface hardness, drying shrinkage, leaching behavior, thermal properties, microstructural features, and phase composition, as summarized in Table 2. All measurements were performed in accordance with internationally recognized ASTM, ISO, and EPA standard methods to ensure the reliability, reproducibility, and comparability of the experimental results. Bulk density and water absorption were determined using the Archimedes immersion method in accordance with ASTM C373. Compressive strength and flexural strength were measured using a universal testing machine (UTM) following ASTM C1424/ISO 8336 and ASTM C1161/ISO 178, respectively. Surface hardness was evaluated using the Vickers indentation method in accordance with ASTM E384, while drying shrinkage was determined according to ASTM C326. Thermal behavior was analyzed using thermogravimetric analysis and differential scanning calorimetry (TGA/DSC), which are widely employed to investigate dehydration behavior and phase evolution in alkali-activated and geopolymer materials [14]. Microstructural characteristics and elemental distributions were examined using scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS), while crystalline

phase composition was identified by X-ray diffraction (XRD). The environmental stability of the developed ceramic materials was further evaluated using the EPA Toxicity Characteristic Leaching Procedure (TCLP, Method 1311) to assess heavy metal immobilization. These complementary characterization techniques provide a comprehensive understanding of the relationships between processing, microstructure, phase evolution, and the resulting engineering performance of the developed geopolymer ceramic materials.

Table 2. Summary of characterization methods for physical, mechanical, thermal, and microstructural properties.

Property	Method / Standard	Description
Bulk density, water absorption	ASTM C373	Determined using the immersion and Archimedes weighing method
Compressive strength	ASTM C1424 / ISO 8336	Measured using a universal testing machine (UTM) at a loading rate of 1 mm/min
Flexural strength	ASTM C1161 / ISO 178	Measured using three-point bending under controlled loading conditions
Leaching test	EPA TCLP (Method 1311)	Evaluated heavy metal release to assess environmental safety
Surface hardness	Vickers (500 g, 10 s)	Measured under a 500 g load with a dwell time of 10 s; average of five indentations reported.
Thermal analysis	TGA/DSC	Conducted from 25 to 1200 °C at a heating rate of 10 °C/min
Drying Shrinkage	ASTM C326	Determined from dimensional changes before and after drying/firing
Microstructure	SEM–EDS	Used to examine morphology and elemental distribution
Phase composition	XRD	Identification of crystalline phases

2.7 Quality Control and Data Availability

All experimental procedures were conducted in accordance with standard laboratory safety and quality assurance protocols. Cement industry waste (CCW) samples were screened for heavy metals (Pb, Cr, Cd, As) using ICP-OES. When concentrations exceeded permissible limits, encapsulation or surface glazing was applied to ensure product safety. All raw and processed datasets—including XRF, XRD, SEM, mechanical tests, and LCA results—will be made publicly available on Figshare upon publication to promote transparency and data reproducibility.

2.8 Wet–Dry Durability Test

The durability of the geopolymer-based ceramic samples was evaluated using a wet–dry cycling test adapted from ASTM D559/D559M (wetting–drying durability of compacted materials) and ASTM C67 (sampling and testing of brick and structural clay tile), with modifications appropriate for geopolymer-derived ceramics. Each cycle consisted of immersion in deionized water at room temperature (25 ± 2 °C) for 24 h, followed by oven drying at 105 ± 2 °C for 24 h. A total of 10 cycles were conducted. Before testing, all specimens were dried to constant mass using a laboratory drying oven (e.g., Memmert UF110 or equivalent) and weighed using an analytical balance with an accuracy of ± 0.001 g (e.g., Sartorius Entris II). After completion of the cycling test, the specimens were reweighed and tested for residual compressive strength using a universal testing machine (UTM) (e.g., Instron 5967) in accordance with ASTM C109/C109M.

3. Results and Discussion

3.1 Chemical and Mineralogical Composition of Raw Materials

The chemical composition of the raw materials analyzed by XRF is presented in Table 3. The cement industry waste (CCW) showed a high CaO content (38.6 wt.%), moderate SiO₂ (32.4 wt.%), and Al₂O₃ (11.8 wt.%), while the clay exhibited higher SiO₂ (56.1 wt.%) and Al₂O₃ (18.7 wt.%) with low CaO (1.2 wt.%). This compositional difference indicates complementary potential for geopolymerization CCW providing calcium for early strength, and the clay supplying reactive aluminosilicates for the geopolymer framework.

The presence of amorphous calcium silicate phases (C₂S and C₃S) identified by XRD further enhances the reactivity of CCW. This observation is consistent with previous studies [6–8], which reported that CaO-rich industrial wastes accelerate geopolymerization by facilitating the formation of calcium–aluminosilicate–

hydrate (C–A–S–H) gels alongside sodium–aluminosilicate–hydrate (N–A–S–H) networks. In addition, SEM micrographs (Figure 1) reveal the irregular and angular morphology of CCW particles, which contributes to mechanical interlocking and improved packing density, ultimately leading to enhanced densification of the geopolymer matrix.

Table 3. Chemical composition of raw materials (wt.%)

Oxide	CCW	Clay
SiO ₂	32.4	56.1
Al ₂ O ₃	11.8	18.7
CaO	38.6	1.2
Fe ₂ O ₃	3.4	5.0
MgO	1.2	0.6
Na ₂ O + K ₂ O	0.9	0.5
LOI	11.7	17.9

3.2 Physical Properties

The physical properties of the clay–geopolymer composites, including bulk density, water absorption, and apparent porosity, were strongly influenced by the geopolymer substitution ratio. As summarized in Table 4, the bulk density increased from 1.85 g/cm³ for the control sample to a maximum of 2.10 g/cm³ at 20 wt% geopolymer substitution, followed by a slight decrease to 2.02 g/cm³ at 30 wt%. The increase in density up to 20 wt% can be attributed to the combined effects of improved particle packing (filler effect) and geopolymer gel formation [6,7]. The local clay, characterized by a plate-like morphology and inherent porosity, typically exhibits a relatively low density (approximately 1.6–1.8 g/cm³). In contrast, the geopolymer paste derived from cement industry waste exhibits a higher density (approximately 2.1–2.3 g/cm³), owing to its calcium-rich composition and the formation of compact C–A–S–H and N–A–S–H gel phases [7,9]. When incorporated into the clay matrix, the finer geopolymer particles act as micro-fillers, effectively occupying interparticle voids and enhancing packing efficiency [6]. Concurrently, geopolymer gel formation during the initial curing stage strengthens interparticle bonding, while subsequent firing promotes further densification through sintering and pore reduction, resulting in a more compact and homogeneous microstructure [7,10]. At 20 wt% substitution, these mechanisms reach an optimal balance, resulting in maximum densification, as evidenced by the highest bulk density and the lowest apparent porosity (22.1%). This is consistent with the significant reduction in water absorption (9.8%), indicating limited pore connectivity and improved matrix compactness. However, increasing the geopolymer content to 30 wt% results in a slight decrease in density, accompanied by increased porosity (25.5%) and water absorption (11.2%). This behavior can be attributed to the excessive alkaline activator content, which may induce incomplete geopolymerization, gel inhomogeneity, and the formation of microcracks and entrapped gases during sintering [8,10]. These defects disrupt matrix continuity and reduce the filler effect, thereby limiting further densification. Overall, water absorption and apparent porosity exhibit a clear inverse relationship with bulk density, confirming that densification is primarily governed by pore reduction and improved microstructural packing. The results indicate that a moderate geopolymer substitution level (20 wt%) provides the most favorable balance between filler effect and geopolymerization, resulting in optimal physical performance. These findings are consistent with previous studies [6–8], which reported that optimal geopolymer content enhances packing efficiency and reduces porosity, whereas excessive alkali content adversely affects microstructural uniformity.

Table 4. Physical properties of clay–geopolymer composites (firing temperature 1050°C)

Geopolymer substitution (wt%)	Bulk density (g/cm ³)	Water absorption (%)	Apparent porosity (%)
0 (Control clay)	1.85 ± 0.03	18.6 ± 0.4	34.2 ± 0.6
10	1.95 ± 0.02	13.5 ± 0.3	27.9 ± 0.4
20	2.10 ± 0.04	9.8 ± 0.2	22.1 ± 0.5
30	2.02 ± 0.03	11.2 ± 0.3	25.5 ± 0.5

3.3 Mechanical Properties

Compressive and flexural strengths of the clay–geopolymer composites increased markedly with geopolymer substitution up to 20 wt%, as shown in Table 5. The specimen containing 20 wt% geopolymer exhibited the highest compressive strength (42.5 MPa), representing a 35% improvement over the control (31.4 MPa). A similar trend was observed for flexural strength, which increased from 8.2 MPa to 11.0 MPa, corresponding to a 34% enhancement. The improvement in mechanical performance may be attributed to the combined effects of chemical bonding and microstructural densification. The interaction between aluminosilicate phases in clay and calcium-rich cement industry waste is likely to promote the coexistence of reaction products, including sodium aluminosilicate hydrate (N–A–S–H) and calcium aluminosilicate hydrate (C–A–S–H) gels, thereby enhancing interfacial bonding and improving load transfer within the matrix [7–8, 14]. The coexistence of these reaction products has been reported in calcium-containing alkali-activated systems and is considered an important mechanism contributing to strength development [8, 14–15]. In addition, physical densification plays an important role in improving mechanical properties. Fine reaction products produced during geopolymerization act as micro-fillers, effectively occupying interparticle voids and improving particle packing, thereby reducing porosity and increasing the measured bulk density [6–7]. The combined effects of micro-filler action and reaction-product development contribute to a denser and more homogeneous microstructure, which is consistent with the observed improvements in compressive and flexural strengths [7, 8]. This interpretation is consistent with the increase in bulk density and the corresponding reduction in apparent porosity discussed in Section 3.2. However, when the geopolymer content exceeded 20 wt%, a slight reduction in strength was observed. This decrease may be attributed to excessive alkaline activator content and incomplete geopolymerization, which could result in a less homogeneous reaction matrix and the formation of microstructural defects, such as microcracks and entrapped pores [8, 15]. These defects may disrupt matrix continuity, thereby reducing the mechanical strength of the composites. Overall, the observed trend is in good agreement with previous studies reporting that optimum mechanical performance in calcium-containing alkali-activated systems is generally achieved at moderate geopolymer contents because of the balanced effects of reaction-product development and microstructural densification [6–8, 14]. The proposed reaction mechanisms should be regarded as plausible explanations based on previous studies, as no direct spectroscopic characterization (e.g., FTIR or solid-state NMR) was performed in the present study to verify the nature of the reaction products.

Table 5. Compressive and flexural strengths of clay–geopolymer composites at different substitution levels (firing temperature 1050 °C)

Geopolymer substitution (wt%)	Compressive strength (MPa)	% Increase vs. Control	Flexural strength (MPa)	% Increase vs. Control
0 (Control clay)	31.4 ± 0.8	—	8.2 ± 0.2	—
10	36.7 ± 0.9	+17 %	9.4 ± 0.3	+15 %
20	42.5 ± 1.0	+35 %	11.0 ± 0.4	+34 %
30	38.6 ± 0.7	+23 %	9.7 ± 0.3	+18 %

3.4 Microstructural Analysis

The SEM micrographs (Figure 1) show a composition-dependent microstructural evolution that is closely related to the densification behavior and mechanical performance of the CCW-modified clay system. At low CCW content, the matrix exhibits a relatively compact but discontinuous aluminosilicate framework containing residual interparticle voids, indicating incomplete densification primarily governed by solid-state diffusion. The initial incorporation of CCW modifies this framework, resulting in a more heterogeneous morphology characterized by localized particle agglomeration and pore formation, which may be associated with carbonate decomposition and gas evolution during firing [1–2]. Although Ca-rich regions may facilitate the onset of liquid-phase sintering, their non-uniform distribution appears to limit effective particle rearrangement and consolidation, resulting in only partial densification [2–3]. At an intermediate composition (approximately 20 wt% CCW), the microstructure becomes denser and more homogeneous, accompanied by

a noticeable reduction in pore size and pore connectivity. This microstructural refinement is consistent with the improved densification behavior observed in the physical property measurements and the enhanced mechanical performance discussed in Sections 3.2 and 3.3. Based on the XRD results presented in Section 3.1, the refinement of the matrix may also be associated with the development of calcium-containing geopolymer reaction products. Previous studies have reported that calcium-containing alkali-activated systems may contain reaction products, including calcium–aluminosilicate–hydrate (C–A–S–H) and sodium–aluminosilicate–hydrate (N–A–S–H) gels, which contribute to matrix densification and improved interparticle bonding [8, 14–15]. However, SEM observations alone cannot directly identify the nature of these reaction products. Therefore, the proposed mechanism is inferred from the combined interpretation of the SEM observations, XRD analysis, and previous studies rather than direct experimental confirmation. Similar synergistic effects between geopolymer reaction-product development and liquid-phase sintering have also been reported for calcium-modified alkali-activated systems [6–8]. When the CCW content exceeded 20 wt%, the microstructure exhibited increased microcracking and interconnected porosity, indicating progressive microstructural deterioration. This behavior may be attributed to excessive CaO, which can promote over-fluxing and excessive liquid-phase formation during firing [2, 8]. Upon cooling, thermal expansion mismatch between coexisting crystalline and amorphous phases may generate internal stresses that facilitate crack initiation and propagation. In addition, the formation of calcium-rich crystalline phases may reduce matrix continuity, thereby limiting the beneficial effects of densification and contributing to the observed reduction in mechanical strength [7, 8]. Overall, the SEM observations indicate a non-linear microstructural evolution with increasing CCW content, progressing from incomplete densification at low replacement levels to a dense and homogeneous matrix at the optimum composition, followed by defect development at higher CCW contents. These observations are consistent with the trends observed for bulk density, apparent porosity, compressive strength, and flexural strength. Collectively, the results suggest that approximately 20 wt% CCW provides an appropriate balance between matrix densification and microstructural stability. The proposed mechanisms should be regarded as plausible explanations based on the combined interpretation of SEM observations, XRD results, and previous studies, since no direct spectroscopic characterization (e.g., FTIR or solid-state NMR) was performed to verify the specific gel phases formed during geopolymerization.

3.5 Thermal Stability and Firing Behavior

The thermal history of the samples was systematically controlled, with maximum firing temperatures of 1000, 1050, and 1100°C, as described in Section 2.5. TGA/DSC analysis (Figure 3) revealed two distinct mass-loss regions. The first, occurring below 150°C, is attributed to the evaporation of physically adsorbed water. The second, within the range of 400–700°C, corresponds to the removal of chemically bound water and the dehydroxylation of geopolymer gel structures [11–12]. The total mass loss (~5 wt%) is consistent with previously reported values for stabilized geopolymeric systems, indicating a relatively low bound-water content and a thermally stable aluminosilicate network [11]. These thermal characteristics correlate closely with the phase evolution observed in the XRD patterns (Figure 2). At temperatures above approximately 600°C, a critical transformation occurs in which the geopolymer gel structure—primarily sodium–aluminosilicate–hydrate (N–A–S–H) and calcium–aluminosilicate–hydrate (C–A–S–H)—undergoes progressive degradation due to structural dehydration and collapse of the amorphous network [3, 12]. This transition is evidenced by the reduction of the amorphous hump and the simultaneous increase in the intensity of the crystalline phases, including quartz (Q), anorthite (A), gehlenite (G), and cristobalite (C). At 1000°C, the XRD patterns indicate the coexistence of residual quartz and newly formed crystalline phases, suggesting the onset of solid-state reactions and phase transformation. Increasing the temperature to 1050°C results in further peak sharpening and increased intensity, reflecting enhanced crystallinity and improved structural ordering, consistent with geopolymer-to-ceramic transformation pathways reported in the literature [12, 30–31].

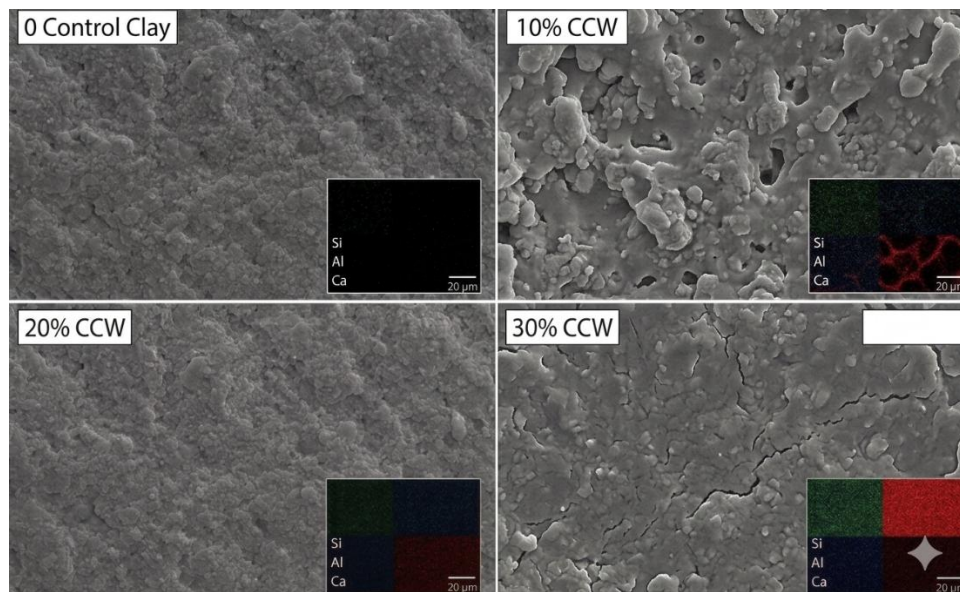


Figure 1. SEM–EDS images of ceramics with 0–30 wt% CCW, showing microstructural evolution from porous to dense and subsequent degradation at high calcium content.

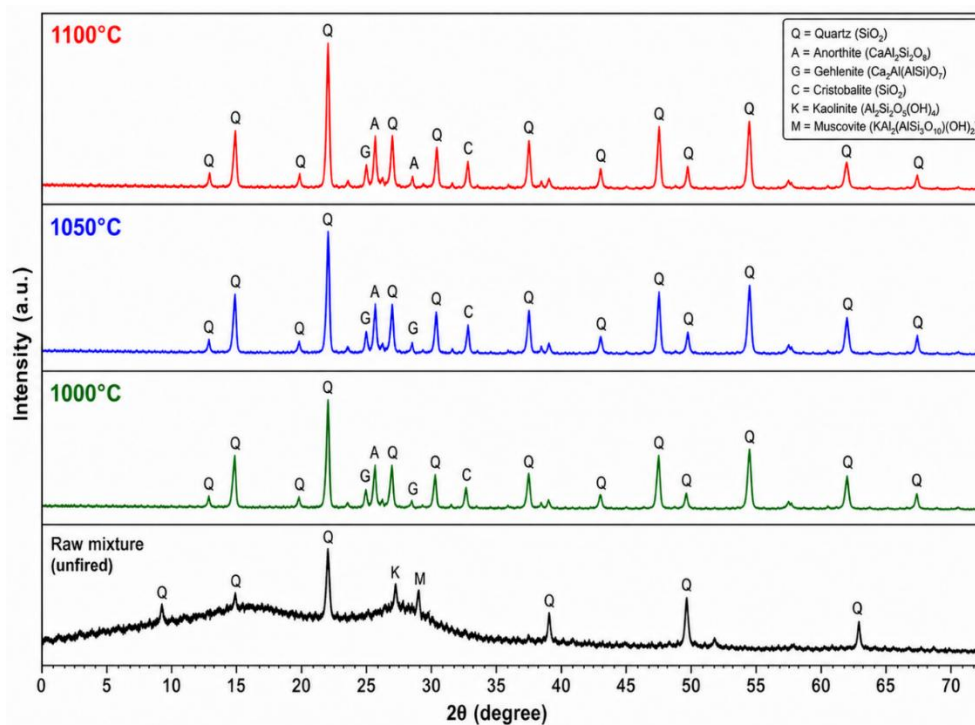


Figure 2. XRD patterns of raw mixture and fired samples at 1000, 1050, and 1100°C, showing phase evolution from amorphous geopolymer to crystalline phases (quartz, anorthite, gehlenite, and cristobalite).

At temperatures above $\sim 900^\circ\text{C}$, sintering and vitrification processes become dominant. These processes facilitate the formation of strong ceramic bonds (Si–O–Si, Al–O–Si, and Ca–O–Si linkages) through particle rearrangement, neck formation, and solid-state diffusion [12, 30–31]. Concurrently, the formation of a viscous glassy phase enhances mass transport and accelerates densification. As a result, the dominant bonding mechanism transitions from geopolymeric gel bonding to ceramic bonding. The CCW-based samples exhibited optimal performance within this temperature range, characterized by high density and minimal cracking, which is consistent with prior studies on kaolin-based and calcium-modified geopolymer ceramics [12]. However, further increasing the firing temperature to 1100°C results in excessive liquid-phase formation,

leading to microstructural instability. This is manifested by localized melting and the formation of microcracks, primarily due to over-fluxing associated with high CaO content [10, 13]. While calcium promotes liquid-phase sintering, excessive amounts can induce differential shrinkage, thermal mismatch, and internal stress during cooling, ultimately leading to structural defects [10]. Such behavior has been widely reported in calcium-rich geopolymer and ceramic systems subjected to high-temperature firing [10, 13].

Overall, the combined TGA/DSC and XRD analyses confirm that geopolymerization governs early-stage bonding before high-temperature exposure, while temperatures exceeding $\sim 600^{\circ}\text{C}$ progressively transform the material into a ceramic system dominated by sintering mechanisms [3, 12]. The identification of an optimal CCW substitution level (~ 20 wt%) aligns with the densification and strength results discussed in previous sections, highlighting the critical role of balanced calcium content in controlling phase evolution, densification, and thermomechanical stability [6, 10]. While firing at 900 – 1050°C yields dense, mechanically robust ceramics, excessive temperatures (e.g., 1100°C) promote over-fluxing and defect formation. These findings demonstrate the strong potential of cement industry waste as a sustainable precursor for high-performance ceramic materials, supporting both resource efficiency and circular economy strategies.

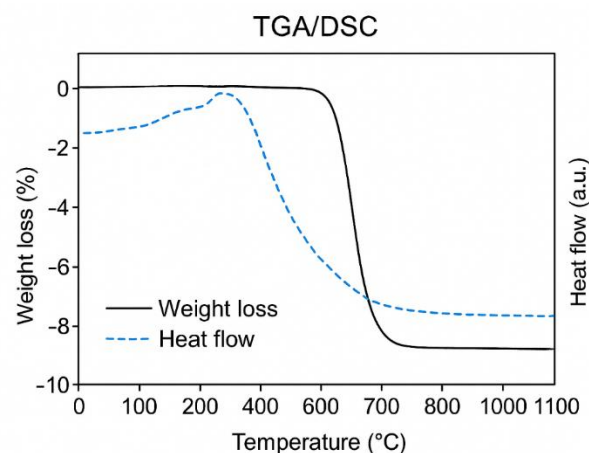


Figure 3. TGA–DSC curves of clay–geopolymer composites showing two major weight-loss regions.

3.6 Leaching Performance

The leaching results (Table 6) indicate that the concentrations of all analyzed heavy metals in the CCW-based geopolymer ceramics were well below the US EPA regulatory limits, indicating compliance with the environmental requirements under the present test conditions. These results suggest that the geopolymer ceramic matrix was effective in restricting the release of hazardous elements. The low concentrations of leached metals may be associated with the dense and relatively continuous matrix developed during geopolymerization and subsequent firing. The denser microstructure observed in the SEM images, together with the increase in bulk density and the reduction in apparent porosity discussed in Sections 3.2 and 3.4, is likely to limit liquid penetration and reduce the mobility of dissolved ions [6, 7]. Consequently, the refined microstructure is considered to contribute to the reduced release of heavy metals observed in the leaching test. It should be noted that the present study did not directly investigate the mechanisms responsible for heavy metal immobilization. Therefore, whether the metals were physically encapsulated within the geopolymer matrix or chemically incorporated into geopolymer reaction products cannot be verified based on the available experimental evidence. Confirmation of these mechanisms would require additional characterization techniques, such as sequential extraction, X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), or synchrotron-based analyses. Overall, the results demonstrate that the CCW-based geopolymer ceramics satisfy the US EPA leaching requirements while maintaining favorable physical, mechanical, and microstructural properties. These findings support the potential use of CCW as a sustainable raw material for geopolymer ceramic production and provide additional evidence that calcium-rich industrial waste can be beneficially utilized in environmentally compatible construction materials.

Table 6. Leaching test results (mg/L)

Metal	Control	20% CCW	EPA Limit
Pb	0.12	0.06	5.0
Cr	0.09	0.04	5.0
Cd	<0.01	<0.01	1.0
Ni	0.05	0.03	5.0

3.7 Wet–Dry durability

The durability results (Figure 4) indicate a progressive reduction in strength retention with increasing wet–dry cycles for all specimens. The control specimen (0 wt% CCW) exhibited the greatest deterioration, retaining only approximately 65% of its initial strength after 10 cycles. This degradation is primarily associated with repeated moisture ingress and evaporation, which generate internal stresses, promote microcrack propagation, and gradually weaken the matrix [6,21]. The relatively porous microstructure and higher apparent porosity of the control specimen further facilitate water penetration, thereby accelerating damage accumulation during cyclic exposure [6]. The incorporation of CCW significantly improved resistance to wet–dry cycling. Among all compositions, the specimen containing 20 wt% CCW exhibited the highest strength retention (approximately 88%), followed by the 30 wt% (approximately 76%) and 10 wt% (approximately 74%) specimens. This improvement may be associated with the denser microstructure and lower apparent porosity developed through the combined effects of geopolymerization and CaO-assisted densification. Previous studies have reported that calcium-containing alkali-activated systems may contain reaction products, including calcium–aluminosilicate–hydrate (C–A–S–H) and sodium–aluminosilicate–hydrate (N–A–S–H) gels, which contribute to matrix refinement and improved durability [7–8, 14]. Although the present study did not directly identify these reaction products, the denser microstructure observed in the SEM images, together with the increased bulk density and reduced apparent porosity, is consistent with the improved durability observed in this study. Similar relationships between microstructural refinement, reduced porosity, and enhanced durability have also been reported for calcium-containing alkali-activated materials [7–8, 21]. The superior durability observed at 20 wt% CCW suggests that this composition provides an appropriate balance between matrix densification and microstructural stability. This observation is consistent with the highest bulk density, the lowest apparent porosity, and the greatest mechanical strength obtained in the present study. Moderate calcium content may promote the development of calcium-containing geopolymer reaction products while simultaneously enhancing liquid-phase sintering, resulting in a refined and relatively homogeneous microstructure [8, 14–15]. However, these mechanisms should be regarded as plausible explanations based on the combined interpretation of the experimental results and previous studies, rather than direct experimental confirmation. When the CCW content was increased to 30 wt%, a slight reduction in durability was observed. This behavior may be attributed to microstructural heterogeneity, localized pore development, and microcrack formation caused by excessive CaO and non-uniform densification during firing [2, 8]. These microstructural features are likely to increase water penetration and facilitate crack propagation during repeated wetting and drying, thereby reducing long-term durability. Overall, the results suggest that incorporating approximately 20 wt% CCW provides the most favorable balance between matrix densification and microstructural stability, resulting in improved resistance to wet–dry cycling. This observation is consistent with the physical and mechanical property results presented in this study and agrees with previous studies on calcium-containing alkali-activated materials [7–8, 14]. The close relationship between durability, bulk density, apparent porosity, and mechanical strength suggests that microstructural refinement is an important factor influencing the long-term performance of CCW-modified geopolymer ceramics.

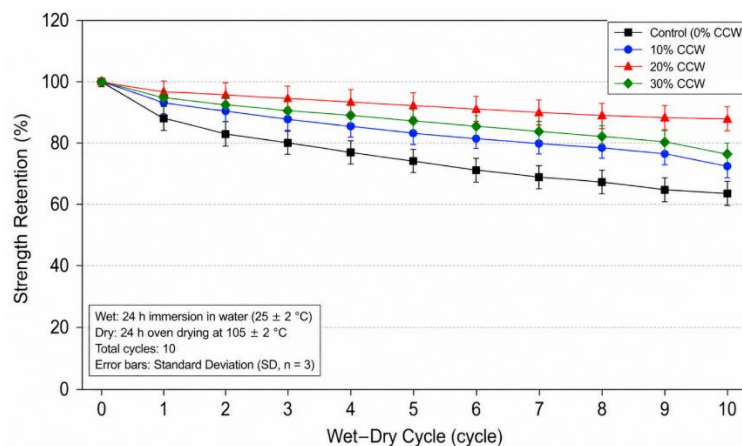


Figure 4. Strength retention of geopolymer-based ceramics incorporating CCW under wet–dry cycling (24 h immersion at 25 ± 2 °C and 24 h drying at 105 ± 2 °C, 10 cycles).

3.8 Pottery-Relevant Characteristics

The incorporation of cement industry waste-based geopolymer (CCW) significantly improved both the processing behavior and the aesthetic–functional properties of the clay ceramics. In terms of workability, the plasticity of the mixtures gradually decreased with increasing CCW content. The control and the mixtures containing 10–20 wt% CCW exhibited good moldability and were readily shaped during forming, whereas the 30 wt% CCW mixture became more viscous and less plastic, requiring greater effort during shaping. This behavior is likely associated with the increased solid loading introduced by the CCW addition, which reduces the plasticity and deformability of the mixtures during forming. Nevertheless, satisfactory workability was maintained with only minor adjustments in water content, indicating that all compositions remained practically processable. As shown in Table 8, drying shrinkage decreased systematically from 6.5% for the control specimen to 4.0% at 20 wt% CCW and then increased slightly to 4.3% at 30 wt% CCW, indicating improved dimensional stability with moderate CCW incorporation, consistent with previous studies on geopolymer drying behavior [23]. This trend is consistent with the evolution of the microstructure observed in the SEM analysis. At moderate CCW contents, improved matrix compactness together with more efficient particle packing contributes to reduced moisture-induced deformation and enhanced dimensional stability [6–7]. However, at higher CCW contents (30 wt%), microstructural heterogeneity and localized pore development may partially offset these beneficial effects, resulting in a slight increase in drying shrinkage [2, 8]. These observations agree well with previous studies reporting that improved matrix densification and reduced porosity enhance the dimensional stability of geopolymer-modified ceramic materials [6–7]. The surface characteristics exhibited a similar composition-dependent trend. The specimen containing 20 wt% CCW exhibited the most homogeneous and compact surface morphology and showed a 20–25% increase in Vickers hardness compared with the control specimen. This improvement is consistent with the enhanced densification and reduced porosity observed in the physical property measurements. Previous studies have shown that calcium-containing alkali-activated systems may develop reaction products that contribute to matrix refinement and improved mechanical performance [8, 14–15]. However, the present study did not directly identify these reaction products; therefore, their presence cannot be verified based solely on the SEM and XRD results. The smoother surface morphology and increased hardness observed in this study are more appropriately attributed to improved densification and microstructural refinement during firing.

Overall, the composition containing 20 wt% CCW provided the most favorable balance between workability, dimensional stability, mechanical performance, and surface quality. At this composition, the combined effects of geopolymerization and CaO-assisted densification produced a dense and relatively homogeneous microstructure with minimal defects. Furthermore, the CCW-modified ceramics achieved favorable densification and surface quality after firing at approximately 1050 °C, suggesting that satisfactory ceramic properties can be obtained at a lower firing temperature than is commonly required for conventional clay ceramics [30–31]. This observation indicates the potential for reduced energy consumption during ceramic

production; however, the present study did not directly evaluate energy savings. Therefore, the results demonstrate that partial replacement of clay with 20 wt% CCW represents a promising approach for producing high-performance and environmentally sustainable geopolymer-modified ceramics while promoting the beneficial utilization of cement industry waste.

Table 7. Effect of Cement Industry Waste–Based Geopolymer (CCW) on Drying Shrinkage and Surface Properties of Clay Ceramics

Sample	Drying shrinkage (%)	Surface hardness (VHN)	Surface observation
Control (0% CCW)	6.5 ± 0.2	420 ± 15	Slightly rough, microcracks visible
10% CCW	5.1 ± 0.2	470 ± 12	Denser, fewer cracks
20% CCW	4.0 ± 0.1	520 ± 10	Smooth, compact surface
30% CCW	4.3 ± 0.2	505 ± 11	Slight vitrification, minor pores

3.9 Comparison with Previous Studies

The mechanical performance obtained in the present study compares favorably with those reported for other calcium-assisted geopolymer and geopolymer–ceramic systems, as summarized in Table 8. Heah et al. [12] demonstrated that kaolin–metakaolin-based geopolymer ceramics achieved compressive strengths of approximately 20–25 MPa after firing, with the improvement primarily attributed to phase transformation and ceramic densification during thermal treatment. Likewise, Somna et al. [33] reported compressive strengths of approximately 20–23 MPa for alkali-activated high-calcium fly ash, demonstrating that calcium plays a significant role in accelerating geopolymerization and enhancing early-age strength development. More recently, Huang et al. [8] achieved a compressive strength of 32.78 MPa in calcium-rich geopolymer ceramics prepared from burnt coal cinder and CaO. Their study revealed that the coexistence of C-(A)-S-H and (N,C)-A-S-H gel phases produced a dense reaction matrix, leading to improved mechanical performance. The present study achieved a substantially higher compressive strength of 42.5 MPa at an optimum replacement level of 20 wt% cement industry waste (CCW), exceeding the values reported in the above studies. This superior performance can be attributed to the synergistic interaction between geopolymerization and ceramic sintering. The incorporation of calcium-rich CCW into the aluminosilicate-rich clay matrix promoted the simultaneous formation of N-A-S-H and C-A-S-H binding gels, resulting in a hybrid reaction network with enhanced interparticle bonding and load-transfer capability [6,10]. In addition, the fine CCW particles acted as micro-fillers, improving particle packing, reducing pore connectivity, and producing a denser and more homogeneous microstructure, consistent with previous studies on calcium-modified geopolymer systems [6–8]. Furthermore, the firing process provided an additional strengthening mechanism through partial vitrification and ceramic bonding. Unlike conventional geopolymer materials that rely primarily on gel formation, the hybrid geopolymer–ceramic system developed in this study benefited from both chemical bonding during alkali activation and microstructural consolidation during firing. The combined effects of hybrid gel formation, pore refinement, and ceramic densification resulted in superior mechanical performance compared with previously reported geopolymer and geopolymer–ceramic materials. Overall, these findings indicate that the incorporation of calcium-rich cement industry waste into traditional clay ceramics represents an effective approach for developing high-performance and sustainable hybrid ceramic materials. The results also demonstrate that optimizing the interaction among precursor chemistry, calcium availability, particle packing, and thermal processing is more critical than simply increasing calcium content, providing valuable guidance for the future design of geopolymer–ceramic composites.

Table 8. Comparison of the present study with previous research

Study	Raw Materials	Optimum Condition	Compressive Strength (MPa)	Key Observation
Heah et al. (2012)	Kaolin + Metakaolin	Fired at 1000–1100 °C	20–25	Phase transformation and ceramic densification during firing enhanced strength.
Somna et al. (2011)	Fly ash + NaOH activator	Alkali activation	20–23	Calcium accelerated geopolymerization and promoted early strength development.
Huang et al. (2023)	Burnt coal cinder + CaO	BCC:CaO:NaAlO ₂ :SS = 18:3:2:4; L/S = 0.46	32.78	Coexisting C-(A)-S-H and (N,C)-A-S-H gels produced a dense microstructure and improved mechanical performance.
Present study	Clay + CCW	20 wt% CCW	42.5	Synergistic clay–CCW interaction promoted hybrid gel formation, ceramic densification, and superior compressive strength.

4. Conclusions

This study demonstrates that integrating cement industry waste (CCW) with clay provides an effective route for developing high-performance hybrid ceramics through the combined effects of geopolymerization and ceramic processing. The results indicate that material performance is governed not only by CaO content but, more importantly, by the balance among geopolymer reaction product development, particle packing, and thermally induced microstructural evolution during firing. An optimum composition of 20 wt% CCW defines a critical processing window in which these mechanisms act synergistically to produce a dense, homogeneous, and defect-minimized microstructure. Below this level, densification is limited by insufficient reaction products and incomplete particle bonding, whereas excessive CCW promotes over-fluxing, microstructural heterogeneity, and defect formation. The experimental observations further suggest a transition from geopolymer-dominated bonding to ceramic bonding during firing, enabling hybrid materials to integrate low-temperature chemical bonding with conventional ceramic sintering. This synergistic behavior improves mechanical performance, durability, and chemical stability while allowing reduced firing temperatures. Overall, this study provides a practical framework for designing sustainable clay–geopolymer hybrid ceramics using cement industry waste. The findings highlight the potential of this approach to improve resource efficiency, reduce industrial waste disposal, and produce high value ceramic materials with enhanced engineering performance. Future research should focus on optimizing compositional design and processing conditions, validating the proposed reaction mechanisms using advanced characterization techniques, and evaluating long-term durability under realistic service conditions.

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