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Thermal Stability and Decomposition Behaviour of Bentonite Clay-Zirconium Silicate-Cow Dung Composites: A TGA/DTA Study

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ABSTRACT

The development of thermally stable and application specific composites from local raw materials is important for refractory, insulating, filtration, and structural ceramic industries. This study investigates the thermal stability and decomposition behavior of bentonite clay (C), zirconium silicate (Z), and cow dung (CD) composites using thermogravimetric and differential thermal analysis (TGA/DTA). Bentonite clay served as the aluminosilicate matrix, zirconium silicate as the refractory inorganic additive, and cow dung as a biomass-derived pore-forming agent. Pure samples (C100, Z100, CD100), binary, and ternary formulations were evaluated using a Thermal Stability Index (TSI) derived from phase transition temperatures, derivative weight loss, and residual weights. TSI performance was categorized into three regimes: high (TSI ≥ 220), moderate (175 ≤ TSI < 220), and low (TSI < 175). The composites underwent characteristic thermal events including moisture removal, organic burnout, dehydroxylation, and structural transformation. Pure Z100 exhibited the highest individual stability TSI of 255, as zirconium bearing phases effectively resisted degradation and stabilized the ceramic matrix. Conversely, cow dung rich compositions showed heightened decomposition due to organic burnout, promoting pore formation while reducing overall stability. Pure C100 and CD100 yielded TSI values of approximately 233 and 196, respectively. Crucially, specific ternary blending demonstrated strong synergistic behavior; the C40Z40CD20 formulation achieved a TSI of approximately 250, matching pure Z100 while incorporating functional porosity. Conversely, equal ratio binary (C50Z50) and high dung ternary (C20Z30CD50) blends showed low stability (TSI < 175). These findings demonstrate that targeted ternary formulation enables cost effective material design without sacrificing thermal endurance, providing composition selection pathways for refractory and porous ceramic manufacturing.

INTRODUCTION

Clay based ceramics remain essential in refractory, structural, insulating, filtration, and construction applications because clays are abundant, low-cost, and capable of forming consolidated ceramic bodies during firing (Murray2006, Sarkar2023, Bose2024). However, the direct use of natural clay in demanding industrial environments is often limited by moderate strength, shrinkage, porosity, and thermal instability. These limitations can be improved by incorporating suitable inorganic and organic additives that modify phase development, pore structure, and high temperature behaviour.

Bentonite clay is an aluminosilicate rich material with important industrial applications arising from its layered structure, plasticity, surface activity and thermal reactivity (Gupta et al., 2025; Kumari *et al.*, 2021, Yuan *et al.*, 2016). When fired, clay minerals undergo sequential processes such as moisture removal, dehydroxylation, phase rearrangement, vitrification and structural consolidation. The incorporation of suitable metal-oxide additives can modify phase formation, microstructure and the resulting properties of aluminosilicate-based materials (Tchakouté *et al.*, 2012). These transformations determine the final density, porosity, thermal conductivity and mechanical performance of the fired body.

Zirconium silicate is a refractory ceramic additive with high thermal resistance, chemical stability, hardness, and compatibility with high-temperature ceramic systems (Sengupta 2020, Sarkar 2023). Its incorporation into clay bodies can improve high temperature stability, enhance densification, and support the formation of mechanically robust ceramic composites. In contrast, cow dung is a biomass derived additive containing organic matter that decomposes during firing, similar to other biomass additives used to generate porosity in fired clay products (Akpomie 2025, Sorte 2020, Nathan 2020). Its burnout can generate controlled porosity, reduce thermal conductivity, and produce lightweight porous ceramic bodies. This makes cow dung attractive for sustainable insulation and filtration materials, especially where low heat transfer is more important than high mechanical strength.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) are powerful tools for examining the thermal behaviour of clay based composites (Galwey 1999, Saadatkhah 2020, Derkowski 2022). TGA measures mass change as a function of temperature, while DTA records thermal events such as endothermic and exothermic transformations. The combination of TGA and DTA provides insight into moisture loss, organic decomposition, dehydroxylation, phase transitions, and structural stability.

This study aims to investigate the thermal stability and decomposition behaviour of bentonite clay-zirconium silicate-cow dung composites using TGA/DTA. The specific objectives are to evaluate the phase transition temperature, derivative weight loss response, and residual weight of pure, binary, and ternary compositions; to identify the influence of zirconium silicate and cow dung on thermal degradation; and to assess the potential of these composites for refractory and porous ceramic applications.

Theoretical Background

TGA Weight Loss Analysis

Thermogravimetric analysis measures the mass change of a sample as a function of temperature or time. The fractional weight loss can be expressed as:

$$W(T) = \frac{m(T)}{m_0} \times 100 \qquad (1)$$

where m(T) is the mass at temperature T and m_0 is the initial mass. The derivative weight loss response is given by:

$$\Delta(T) = \frac{m(T) - m_0}{m_0} \times 100 = 100 - W(T) \qquad (2)$$

For clay–zirconium silicate–cow dung composites, ΔW represents the cumulative contribution of physically adsorbed moisture removal, organic burnout, clay mineral dehydroxylation, and any additional decomposition or oxidation reactions occurring during heating.

The derivative thermogravimetric response is used to identify the rate at which mass is lost.

It is written as

$$\frac{dW}{dT} = \frac{1}{m_0}\frac{dm}{dT} \times 100 \qquad (3)$$

The temperature at which the absolute value of dW/dT becomes maximum is the peak decom-
position or phase transition temperature, expressed as

$$T_{phase} = T\left[max\left(\frac{dW}{dT}\right)\right] \qquad (4)$$

A higher T_{phase} generally indicates delayed decomposition and improved thermal resistance, whereas a more negative derivative response indicates a sharper and more intense mass loss process.

DTA Heat Flow Interpretation

Differential thermal analysis records the temperature difference between the sample and an inert reference material during heating. The DTA signal can be represented as:

$$\Delta T = T_s - T_r \qquad (5)$$

where T_s is the sample temperature and T_r is the reference temperature. Endothermic events such as moisture evaporation, dehydroxylation, and organic decomposition usually appear as downward thermal effects, while exothermic events may arise from oxidation, crystallization, or structural reorganization. In the present composites, the dominant endothermic features are associated with moisture loss, organic decomposition of cow dung, and clay mineral transformation.

Thermal Stability Index and Kinetic Consideration

For comparative interpretation, a simple thermal stability index can be defined from the transition region residual mass and transition temperature as:

$$TSI = \frac{W_{phase}T_{phase}}{100} \qquad (6)$$

where W_{phase} is the residual mass at the major transition region. A higher value suggests that a sample retains more mass at a higher transition temperature and is therefore relatively more stable. Although the present study focuses on comparative TGA/DTA interpretation rather than full kinetic modeling.

These relationships equation (1-6) provide the theoretical basis for interpreting the observed TGA/DTA peaks, derivative weight loss responses, and thermal stability differences among the composites.

MATERIALS AND METHODS

Raw Materials and Composite Design

The raw materials used were bentonite clay, zirconium silicate, and cow dung. Bentonite clay served as the aluminosilicate ceramic matrix. Zirconium silicate was incorporated as a refractory and thermally resistant inorganic additive. Cow dung was used as an organic pore forming additive. The materials were processed, blended, shaped, and prepared into pure, binary, and ternary compositions.

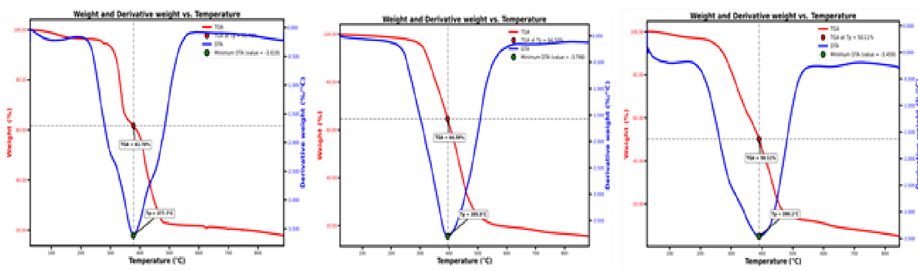
The sample codes represent the percentage composition of clay (C), zirconium silicate (Z), and cow dung (CD). The investigated samples included pure C100, Z100, and CD100; binary clay-zirconium silicate blends C80Z20, C60Z40, C50Z50, C40Z60, and C20Z80; binary clay-cow dung blends C80CD20, C60CD40, C50CD50, C40CD60, and C20CD80; and ternary composites C60Z20CD20, C50Z30CD20, C40Z40CD20, C33Z33CD33, C20Z40CD40, C20Z30CD50, and C20Z20CD60.

TGA/DTA Characterization

The thermal stability and decomposition behavior of each composite sample were determined by measuring their weight loss as a function of temperature under a controlled heating program. The experiment was carried out on the composites between room temperature and 1000 ^{0}C at a heating rate of 10 ^{0}C minute in an air tight atmosphere. A standard TGA/DTA system which comprises a high precision microbalance, a programmable furnace capable of reaching temperatures up to 1600 ^{0}C, a sample holder made of platinum, and an integrated gas flow system for maintaining inert or reactive atmospheres of nitrogen was use (Iyamu *et al.*, 2026) The system is connected to software that allows real time data acquisition and analysis of the thermogram. The TGA/DTA machine now provides detailed insight into the thermal transitions associated with moisture loss and structural transformations for the various composites. The various events that happen at each temperature (weight loss and the derivative weight) were all recorded. All the thermal events in the various composites at heating rates, 10 ^{0}C /minute were recorded and plotted against temperature.

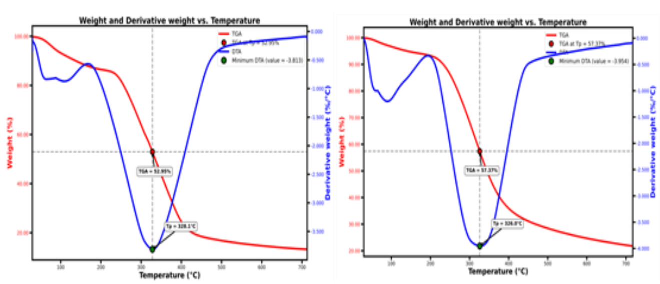
RESULTS AND DISCUSSION

The major TGA/DTA plots selected from the full experimental set are presented in Figures 1–4. These plots represent the pure raw material systems, the strongest clay–zirconium silicate blends, the pore forming clay-cow dung blends, and the balanced ternary compositions. They provide visual confirmation of the numerical thermal parameters in Table 1.



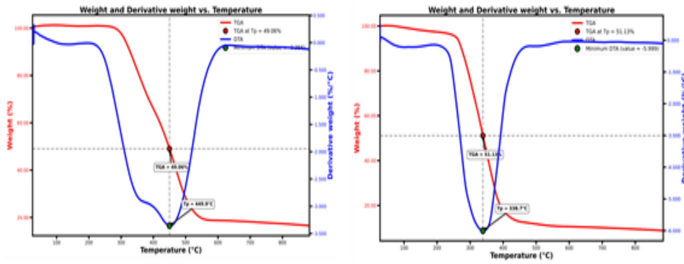
(a) C100 (b) Z100 (c) CD100

Figure 1: Representative TGA/DTA Plots of Pure Bentonite Clay, Zirconium Silicate, and Cow Dung Samples



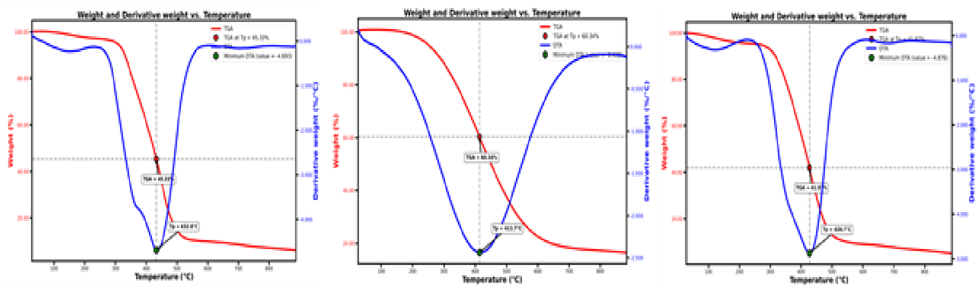
(a) C50Z50 (b) C20Z80

Figure 2: Major TGA/DTA Plots of Clay–Zirconium Silicate Composites Showing Refractory Rich Binary Behavior



(a) C40CD60 (b) C40CD60

Figure 3: Major TGA/DTA Plots of Clay–Cow Dung Composites Showing Refractory Rich Binary Behaviour



(a) C60Z20CD20 (b) C40Z40CD20 (c) C20Z20CD60

Figure 4: Major TGA/DTA Plots of Ternary Clay–Zirconium Silicate–Cow Dung Composites Showing Combined Refractory Stabilization and Organic Burnout Effects

TGA/DTA Parameters of the Composites

The phase transition temperatures, T_{phase}, minimum derivative weight, W^{I}_{min} and the percentage weight loss at phase transition W_{phase} were extracted from the various TGA/DTA plots for all the prepared samples and the results presented in Table 1.

Table 1: TGA/DTA Thermal Parameters

Sample ID	T_{phase} (^{0}C)	W^{I}_{min} ($\%/^{0}C$)	W_{phase} (%)	TSI
C100	377.70	-3.619	61.70	233.04
Z100	395.00	-3.798	64.59	255.13
CD100	390.10	-3.459	50.11	195.48
C80Z20	307.00	-3.605	64.80	198.94
C60Z40	314.80	-2.771	63.70	200.53
C50Z50	328.10	-3.813	52.95	173.73
C40Z60	335.60	-4.021	50.34	168.94
C20Z80	326.00	-3.954	57.37	187.03
C80CD20	329.60	-3.631	63.57	209.53
C60CD40	311.50	-5.249	59.25	184.56
C50CD50	305.30	-5.554	51.49	157.20
C40CD60	449.90	-3.356	49.06	220.72
C20CD80	338.70	-5.999	51.13	173.18
C60Z20CD20	432.80	-4.693	45.33	196.19
C50Z30CD20	377.40	-3.507	51.91	195.91
C40Z40CD20	413.70	-2.438	60.34	249.63
C33Z33CD33	334.80	-6.656	63.05	211.09
C20Z40CD40	383.60	-4.839	58.25	223.45
C20Z30CD50	339.00	-5.936	46.97	159.23
C20Z20CD60	426.70	-4.876	41.97	179.09

The thermal degradation profile of the bentonite clay-zirconium silicate-cow dung (C-Z-CD) matrix is fundamentally governed by three distinct thermodynamic metrics (Li and Stoliarov, 2014). The phase transition temperature T_{phase}, the peak derivative weight loss rate W'_{min}, and the transition mass retention W_{phase}. Together, these parameters dictate the Thermal Stability Index (TSI), which serves as the definitive indicator for advanced material selection (Granados *et al.*, 2025, Goodarzi, and Zendehboudi, 2019)

The underlying pyrolytic breakdown of these composites progresses through four distinct operational regimes (Lu and Gu, 2022). At lower temperatures 150 ºC, desorption dominates via the evaporation of physically adsorbed moisture and interlayer water. This is followed between 150 ºC and 400 ºC by biomass pyrolysis, characterized by the oxidative decomposition and volatilization of hemicellulose, cellulose, and lignin fractions originating from the cow dung dopant. As heating continues into the 400 – 600 ºC window, dehydroxylation occurs, triggering the structural collapse and crystalline rearrangement of the aluminosilicate clay mineral framework (Heller, 2013). Finally, at temperatures exceeding 600 ºC, sintering and phase stabilization take place, driving high temperature solid state reactions and ceramic residue consolidation that are heavily reinforced by the refractory zirconium bearing phases (Dunn, 2000). The complex interaction between the rigid refractory agent (Z) and the volatile biomass pore-former (CD) within the clay matrix (C) yields highly non-linear thermal trends across the distinct composition suites (Kumari et al., 2025, Shearer *et al.*, 2024)

Pure Material Baselines (C100, Z100, CD100)

Establishing the upper boundary for baseline performance, pure Z100 boasts the highest comprehensive stability with a TSI of 255.13 ºC and a notable mass retention of 64.59 % at T_{phase} of 395.00 ºC. This behavior is directly attributable to the intrinsic thermodynamic resistance of zirconium silicate. Conversely, CD100 experiences rapid organic burnout that actively drives volatile outgassing, while the pure clay baseline C100, T_{phase} of 377.70 ºC occupies the intermediate thermal ground, reflecting typical aluminosilicate dehydroxylation dynamics.

Clay-Zirconium Silicate (C-Z) Binary Composites

Incorporating zirconium silicate maintains excellent structural char yields, as evidenced by C80Z20 and C60Z40 retaining 64.80 % and 63.70 % of their mass, respectively. However, the binary interaction introduces an unexpected downward shift in initial phase transition temperatures 307.0 ºC – 335.6 ºC relative to pure Z100. This phenomenon implies that the clay-zirconium interfacial zone alters early-stage reaction kinetics, triggering premature structural rearrangements within the mixed matrix even though high-temperature structural integrity is ultimately preserved. (Ma and Atzmon, 1995)

Clay-Cow Dung (C-CD) Binary Composites

The addition of cow dung shifts the binary system toward rapid mass loss behavior, characterized by sharp derivative weight loss slopes that reach a highly volatile -5.999 %/ºC in C20CD80. This trend underscores the role of biomass as an aggressive pore forming agent (Ashraf *et al.*, 2021). A compelling anomaly occurs in C40CD60, which displays the highest overall T_{phase} 449.90 ºC paired with low mass retention. This suggests the formation of a temporary, complex clay organic intermediate phase that successfully delays macromolecular collapse until higher temperatures are reached (Tian *et al.*, 2023).

Ternary System Synergies (C-Z-CD)

The ternary framework successfully balances the antagonistic effects of Z and CD, offering a highly tunable compromise between refractory stability and porosity development. Composition selection is critical here; C40Z40CD20 emerges as the premier configuration with TSI of 249.63, anchoring a high transition ceiling 413.70 ºC with an exceptionally controlled, slow degradation velocity W'_{min} of -2.438 %\ºC. Conversely, an less thermally stable composition ternary ratio like C33Z33CD33 results in chaotic, aggressive outgassing W'_{min} of -6.656 %\ºC, highlighting how critical compositional balance is to preventing structural failure (Lee *et al.*, 2026)

The Industrial Material Selection Framework

To guide industrial application, the experimental results can be partitioned into three distinct application paradigms based on their thermal stability index profiles (Barra *et al.*, 2023; Sun *et al.*, 2025).

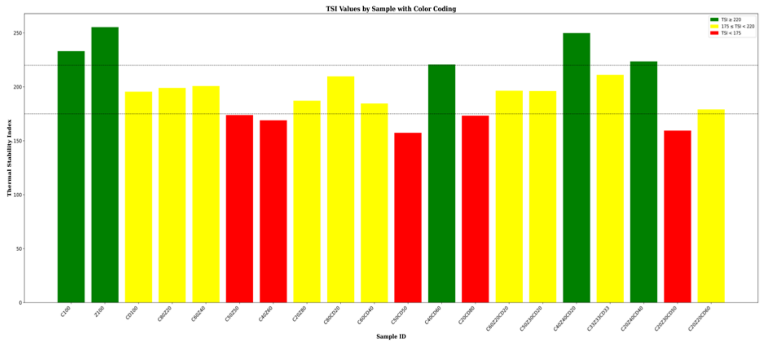


Figure 5: Thermal Stability Index (TSI) Values Across Pure and Blended Sample Compositions, Categorized by Performance Thresholds

CONCLUSION

The Thermal Stability Index (TSI) classified the sample formulations into three performance categories according to their resistance to thermal degradation. The high-performance category (TSI $\geq$ 220) comprised Z100, C40Z40CD20, C100, C20Z40CD40 and C40CD60, with Z100 recording the highest TSI of approximately 255, followed by C40Z40CD20 at about 250. These formulations demonstrated strong thermal stability and may therefore be suitable for demanding high-temperature applications, including refractory coatings, automotive heat shields, aerospace components and high-power semiconductor substrates. The moderate performance category (175 $\leq$ TSI < 220) included C80CD20, C33Z33CD33, C60Z40, C80Z20, CD100, C60Z20CD20, C50Z30CD20, C20Z80, C60CD40 and C20Z20CD60, with CD100 recording a TSI of approximately 196. These formulations provide sufficient thermal resistance for general purpose industrial applications such as electrical insulation, wire jacketing, conduit piping, automotive interior components, commercial piping and fluid storage tanks. Conversely, C50Z50, C40Z60, C50CD50, C20CD80 and C20Z30CD50 fell within the lower performance category (TSI < 175), indicating comparatively poor thermal stability and greater susceptibility to heat induced degradation. Consequently, these formulations can be more appropriate for lower temperature applications, non-structural fillers, sacrificial layers, single use packaging, agricultural films and transient devices, rather than structural or high-temperature industrial applications.

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CONFLICT OF INTEREST

The author declares no conflict of interest.

DATA AVAILABILITY

The data used in this article were derived from the thesis experimental results and are summarized in the TGA/DTA table included in this manuscript.

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