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Original Article

Thermal Evolution and Comparative Stability of Metakaolin-Based Geopolymer Matrices for High-Temperature Composite Applications

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Abstract - *Metakaolin-based geopolymers are being considered for applications requiring thermal resistance and structural stability at elevated temperatures. However, multiple formulations have been reported for comparative thermal evaluation studies; comparing formulations prepared under controlled activator conditions remains relatively limited. In this present study, twelve metakaolin-based geopolymer matrices were prepared by varying sodium hydroxide concentration, sodium silicate-to-hydroxide ratio, and precursor composition. Based on prior FTIR, XRD, and SEM characterization, six matrix formulations (C, D, E, F, G, and I) were selected for further detailed thermal investigation. Thermogravimetric Analysis (TGA–DTG) and Differential Scanning Calorimetry (DSC) were conducted over a temperature range of 30 °C to 1000 °C under an inert gas atmosphere to estimate mass-loss behaviour, thermal transitions, and structural stability of all the matrices. All the matrices exhibited thermal decomposition at various stages related to moisture release, dehydroxylation, and structural transformation due to high temperatures. Thermal analysis was observed among the six formulations. Sample I showed the lowest mass loss and the highest residual Mass (87.96%), representing greater thermal stability when compared to other samples. In comparison, Sample C exhibited higher mass loss and a highly reactive amorphous aluminosilicate network, which was identified through previous structural analyses. Based on TGA–DTG and DSC results, the overall thermal performance of the six geopolymer matrices was established as I > D > E > G > F > C. The findings show that the alkaline activator and precursor composition mixture strongly affect the thermal behaviour and microstructural development of the geopolymer matrix during heating. This study offers a simple method for evaluating thermal stability of geopolymer matrices and supports the selection of suitable matrices for the future development of fibre-reinforced geopolymer composites.*

Keywords - *Metakaolin-based geopolymers, Thermal stability, Thermogravimetric analysis (Tga–Dtg), Differential Scanning Calorimetry (Dsc), Alkali activation, High-temperature performance.*

1. Introduction

Metakaolin-based geopolymers have emerged as effective alkaline-activated binder materials due to their ability to form a rigid aluminosilicate network without the use of ordinary Portland cement [1-3, 19]. When aluminosilicate precursors and alkaline activators are mixed together, it reacts and form a three-dimensional network composed of Si–O–Al bonds, with chemical stability and resistance to elevated temperatures. These combined characteristics provide good thermal stability, chemical durability and resistance to high temperatures. These properties have supported the investigation on geopolymers for structural, refractory, and other high-temperature applications.

The geopolymer materials' behaviour at high temperature has become an important area of research area in recent years. Metakaolin-based geopolymers are better at maintaining their 3D structural network even after it is exposed to high temperature when compared to conventional ordinary Portland cement. OPC often undergoes dehydration and loses water, and experiences a loss of structural strength during heating at high temperatures [2]. Previous studies have shown that the thermal performance of the materials is strongly affected by their chemical composition and microstructural characteristics of the geopolymer matrix [3].

Similarly, other research has shown that geopolymers with better resistance to thermal degradation have a dense and homogeneous microstructure [6]. In another application area, Ke and Baki [13] demonstrated that metakaolin-based geopolymers are used for thermochemical energy storage and highlighted the importance of thermal durability and pore stability. These studies indicate that the thermal response of geopolymer is closely linked to the microstructure developed during the geopolymerisation process.

The alkaline activator plays a major role in controlling geopolymer performance. Sodium hydroxide concentration influences precursor dissolution and gel formation, while the sodium silicate content affects aluminosilicate network and structural development. Soares et al., [5] reported some noticeable changes in thermal decomposition behaviour when sodium silicate and sodium hydroxide proportions were modified in the entire geopolymer systems. Li et al., [14] also demonstrated that curing and processing conditions influence matrix densification and thermal resistance. Geopolymer materials exposed to long-term and prolonged heating plays an important factor in governing the thermal stability [23, 26]. The relationship between Alkaline activator, microstructure and thermal performance were observed from these observations.

Thermal analysis methods are commonly used to investigate the thermal effects of the material during heating. Thermogravimetric Analysis (TGA) is used to find the mass-loss behaviour during heating and allows identify the decomposition stages such as moisture removal, dehydroxylation, and microstructural changes in the geopolymer matrix. Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC) are used to measure and record thermal events related to different changes in the matrix structure and provides information on thermal stability and transformation that occurs in the alumino silicate network.

According to He et al., [2], TGA is used for tracking dehydration and decomposition processes in alkali-activated materials. Aversa et al., [7] demonstrated that DSC can identify the changes in thermal transitions and the modification of the geopolymer matrix. TGA and DSC are used together to evaluate the thermal stability of geopolymer materials [8, 18, 25]. Although many studies have investigated the thermal behaviour of geopolymers, comparison among different formulations remains difficult. Many studies focus only on activator concentration, curing conditions, or precursor composition [3, 5, 14].

Differences in raw materials and testing procedures further complicate comparison between published datasets. In addition, thermal analysis is often reported separately from structural characterization, making it difficult to relate thermal response to changes in chemical bonding, phase assemblage, and microstructure [29, 30]. As a result, the mechanisms linking formulation, geopolymer development, and thermal stability are not always discussed within a single experimental framework. Another point that receives comparatively little attention is the distinction between chemical reactivity and thermal resistance. A formulation that exhibits favourable geopolymerisation characteristics during synthesis does not necessarily provide the greatest resistance to thermal degradation after curing. This issue becomes particularly important when selecting materials for applications involving prolonged exposure to elevated temperatures. A comparative assessment of multiple formulations prepared and tested under identical conditions can therefore provide valuable insight into formulation-dependent behaviour.

In the present work, twelve metakaolin-based geopolymer matrices were produced using different combinations of sodium hydroxide concentration, sodium silicate-to-hydroxide ratio, and precursor composition. Preliminary FTIR, XRD, and SEM characterization was performed on all formulations. Based on those results, six representative matrices (C, D, E, F, G, and I) were selected for detailed thermal evaluation.

Thermogravimetric Analysis (TGA–DTG) and Differential Scanning Calorimetry (DSC) were employed to investigate decomposition behaviour, residual mass retention, thermal transitions, and overall stability between 30 and 1000 °C. The thermal results were subsequently considered alongside the previously obtained FTIR, XRD, and SEM observations in order to compare the behaviour of the selected matrices and identify formulations exhibiting favourable performance under elevated-temperature conditions.

2. Literature Review

Alkali-activated geopolymers prepared using metakaolin precursors have received considerable attention as alternatives to conventional binders because of their aluminosilicate network formation, thermal resistance, and reduced dependence on Portland cement-based systems [1-3, 19]. These alkaline-activated materials are mainly made of Si–O–Al networks formed by alkali activation. Previous research has revealed that geopolymers can have lower shrinkage, better thermal and heat resistance, and improved stability and structural properties at high temperatures compared with conventional ordinary Portland cement. The effects of different compositions and processing conditions on the thermal behavior of geopolymer conditions continues to be studied.

2.1. Thermal Behaviour of Metakaolin-based Geopolymers

The thermal behaviour of geopolymer binders depends on many factors, such as Si/Al ratio, alkaline activator composition, curing conditions, and internal microstructure [3, 14]. Thermogravimetric analysis is used to study the mass loss of geopolymer materials during heating, which shows several layers of decomposition. The initial stage, below 200 °C, is due to loss of physically adsorbed water. The second stage, between 200–400 °C, is related to dehydration of chemically bound water and dehydroxylation. Above 400 °C, additional mass loss is generally caused by structural changes and decomposition of carbonate materials [2, 7-9].

Although this overall thermal behaviour is commonly observed, the amount of mass loss may differ depending on matrix composition and alkaline activator. Geopolymers with dense aluminosilicate networks generally show lower total mass loss and smoother DTG transitions, which indicates better thermal stability. In contrast, less dense matrices show greater decomposition and better thermal instability. Differential Scanning Calorimetry (DSC) is also used to study thermal changes occurring in geopolymer matrices during heating. Thermal events above approximately 300-400°C are related to structural changes, matrix densification, and continuous development of the geopolymer network [7, 12].

Previous studies, by Aversa et al., [7], have shown that the intensity and distribution of exothermic thermal events depend on the gel homogeneity and network development within the geopolymer. Differences in the temperature range and intensity of thermal events reported in studies indicate that geopolymer behavior is affected by compositional and processing conditions. Therefore, studies carried out under controlled conditions are important of better understanding of geopolymer thermal performance.

2.2. Influence of Activator Chemistry and Composition

Alkaline Activator plays an important role in the geopolymerisation process, microstructural development, and thermal performance of metakaolin-based geopolymer. The sodium hydroxide affects the dissolution of aluminosilicate in the precursor material and the formation of geopolymer gels. Kim et al., [3] reported Si/Al ratio affects thermal stability and network development during heating.

The sodium silicate-to-hydroxide ratio governs geopolymer network formation. Soares et al., [5] showed that combinations of silicate and hydroxide activators affect thermal decomposition and material stability. Previous studies showed that curing conditions also contribute to the development of geopolymer microstructure.

Li et al., [14] observed that curing conditions affect matrix densification and thermal resistance in metakaolin-based geopolymer composites. Also, Kljajević et al., [15] stated that activator concentration affects the thermal response of geopolymer during heat treatment. These findings indicate that geopolymer performance is governed by the effects of activator chemistry, precursor composition, and curing conditions.

2.3. Microstructural Correlation and Multi-Technique Analysis

Microstructural characterization is important for understanding the development and thermal behaviour of geopolymer materials. Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) are used to inspect chemical bonding, phase composition, and microstructure of alkali-activated aluminosilicate systems. FTIR analysis is used to identify geopolymerization through changes in Si–O–T bands, which reflect the formation and evolution of aluminosilicate gel networks [11]. XRD analysis provides information about the amorphous phase development and crystalline phases. Previous studies have reported that the formation of broad amorphous humps in XRD shows geopolymer network development during alkali activation [3, 11].

SEM investigations reported that the thermal and mechanical performance affects gel homogeneity, pore distribution, and matrix densification. Adjei et al., [6] reported that geopolymer with dense and uniform microstructures showed improved resistance to elevated temperatures. Thermally stable metakaolin-based geopolymer contributes to improved stability during heating [8, 14].

Recent studies highlighted the importance of combining FTIR, XRD, SEM, TGA, and DSC analysis to understand the relationship between geopolymer composition, microstructure and thermal behaviour [29, 30]. Therefore, the integration of these techniques provides a more comprehensive framework for evaluating geopolymer matrix development and detecting formulations suitable for high-temperature applications.

2.4. Benchmarking with Recent Studies

Recent studies have enhanced the understanding of the thermal behavior of metakaolin-based geopolymers. Recent studies have shown that features such as the Si/Al ratio, activator composition, and microstructure strongly effect thermal stability and high-temperature performance [3, 5, 6].

Thermal analysis techniques such as TGA and DSC for geopolymers are used to study dehydration, dehydroxylation, and structural changes during heating [7, 8]. Recent studies highlighted the importance of combining thermal, chemical, and microstructural analyses to better understand geopolymer behavior [29, 30]. However, studies comparing multiple geopolymer matrices prepared under the same conditions are still limited.

Table 1. Representative studies related to thermal characterization of metakaolin-based geopolymer systems

Study	Research Focus	Key Findings	Research Gap
Kim et al., [3]	Influence of Si/Al ratio on geopolymer behaviour	Thermal stability and phase evolution depend strongly on Si/Al ratio	Limited compositional scope
Soares et al., [5]	Effect of activator composition	Activator chemistry significantly affects thermal response	No comparative ranking framework

Adjei et al., [6]	Elevated-temperature microstructural behaviour	Dense microstructures improve thermal resistance	Limited thermal–microstructural integration
Aversa et al. [7]	DSC-based investigation of alkali-activated metakaolin systems	Structural rearrangement detected through calorimetric analysis	Focused on specific formulations
Bachirat et al. [8]	Combined structural and thermal characterization	Thermal behaviour linked to morphology and material structure	Limited multi-formulation comparison
Wu et al., [29]	Review of alkali-activated composites at elevated temperature	Need for integrated thermal and mechanical evaluation	Limited experimental validation
Provis [30]	Fundamental geopolymer science and activation mechanisms	Composition strongly influences geopolymer development	Does not provide a comparative thermal ranking

Collectively, the reviewed studies demonstrate substantial progress in understanding the thermal behaviour of metakaolin-based geopolymer systems. However, opportunities remain for systematic comparative investigations involving multiple formulations prepared under identical processing conditions and evaluated using integrated thermal and microstructural characterization approaches.

2.5. Competing Matrix Systems

The performance of metakaolin-based geopolymers is often evaluated in comparison with other inorganic binder systems used in thermally demanding environments. Conventional Portland cement materials are widely utilized in structural applications; however, exposure to elevated temperatures can lead to dehydration, microstructural degradation, and reductions in mechanical integrity [2]. These limitations have motivated the investigation of alternative binder systems capable of maintaining stability under thermal loading.

Among the available alternatives, calcium aluminate cements and phosphate-bonded materials have demonstrated favourable thermal resistance and high-temperature performance in specialized applications. However, their processing requirements, material characteristics, and application-specific limitations may influence their suitability for particular engineering uses. Consequently, no single binder system can be considered universally optimal for all thermal environments. Metakaolin-based geopolymers have emerged as attractive candidates because their properties can be tailored through modifications in precursor composition, activator chemistry, and curing conditions. Kim et al., [3] demonstrated that compositional parameters significantly influence thermal stability and structural evolution, while recent reviews have highlighted the adaptability of alkali-activated materials for applications requiring thermal resistance and long-term durability [2, 29].

In addition to thermal performance, geopolymer systems have attracted attention because they can be produced using a wide range of aluminosilicate precursors and can be engineered to achieve specific combinations of thermal, structural, and durability-related properties. These characteristics make metakaolin-based geopolymers promising materials for further investigation in elevated-temperature applications.

Although direct comparisons among Portland cement, calcium aluminate cement, phosphate-bonded materials, and geopolymers remain relatively limited, available studies indicate that activator-controlled geopolymer systems provide a flexible platform for tailoring high-temperature performance according to application requirements.

2.6. Sustainability Considerations

Sustainability is an important factor in the development of geopolymer materials. Geopolymers from aluminosilicate precursors are the alternatives to Portland cement, which allows the use of industrial by-products and natural resources [19, 33]. Studies have highlighted their potential for construction applications due to their durability and efficient material utilization [33]. However, the sustainability of geopolymers should be assessed carefully. The preparation of alkaline activators like sodium hydroxide and sodium silicate requires significant energy and contributes to environmental impacts [19]. Therefore, a complete assessment should consider both the benefits and environmental costs associated with production and use.

2.7. Critical Research Gaps and Need for Study

Studies have enhanced the understanding of metakaolin-based geopolymers, but some gaps still remain. Most research focuses on a limited number of formulations, making direct comparisons difficult [3, 5, 14]. Thermal analyses, TGA and DSC, are often reported separately from FTIR, XRD, and SEM results, limiting the understanding of the relationship between thermal behaviour and microstructure [29, 30]. Comparative studies that rank many formulations under the same testing conditions are also limited. Therefore, integrated studies combining FTIR, XRD, SEM, TGA, and DSC are needed to evaluate geopolymer development, thermal stability, and performance.

2.8. Scope of the Present Study

The aim of this study to assess the thermal performance of metakaolin-based geopolymer matrices prepared under controlled activator conditions. Twelve formulations (A–L) were produced by varying sodium hydroxide concentration, sodium silicate-to-hydroxide ratio, and precursor composition. Based on previous FTIR, XRD, and SEM results, out of twelve, six matrices (C, D, E, F, G, and I) were selected for thermal analysis. TGA–DTG and DSC were used to study mass loss, thermal transitions, and thermal stability from 30 to 1000 °C. The thermal results were combined with FTIR, XRD, and SEM findings to understand how the composition and microstructure affect the thermal behaviour, and to identify suitable matrices with good thermal stability for high-temperature applications.

3. Materials and Methods

3.1. Research Design and Study Framework

Twelve metakaolin-based geopolymer matrices (A–L) were prepared to study the effects of alkaline activator chemistry and precursor composition on thermal performance. The formulations were produced by varying sodium hydroxide concentration (8 M, 10 M, and 12 M), geopolymer grade sodium silicate-to-sodium hydroxide ratio (1.5 and 2.0), and metakaolin-to-aluminosilicate ratio (60:40 and 70:30). The study included slurry matrix preparation, geopolymer synthesis, curing, and thermal characterization. Based on previous FTIR, XRD, and SEM results, six representative formulations were selected for further thermal analysis using TGA–DTG and DSC to compare their thermal stability.

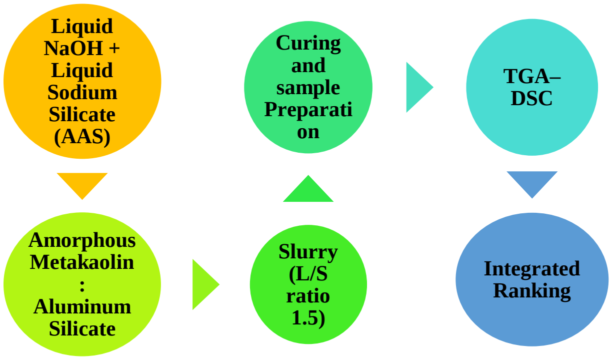


Fig. 1 Step-by-step process used to prepare and thermally analyze the metakaolin-based geopolymer matrices

3.2. Inclusion and Exclusion Criteria

All geopolymer specimens were prepared using the same precursors as raw materials and alkaline activators to ensure consistency. Mixing, casting, curing, and storage conditions were kept the same for all formulations. Specimens with visible defects, poor curing, or excessive pores were excluded. Only defectless specimens were selected for microstructural and thermal testing.

3.3. Materials

Metakaolin (MK) and Aluminum Silicate (AS) were used as precursor materials at different ratios. Sodium hydroxide solutions of 8 M, 10 M, and 12 M and geopolymer-grade sodium silicate solution were used as alkaline activators. The activator was prepared by mixing sodium hydroxide and sodium silicate at $Na_2SiO_3/NaOH$ ratios of 1.5 and 2.0. The activator was then combined with the precursor powders to produce geopolymer slurries with a liquid-to-solid ratio of 1.5. The formulation details are presented in Section 3.4.

Table 2. Chemical composition of raw materials used in geopolymer matrix synthesis

Oxide / Property	Metakaolin (MK)	Aluminum Silicate (AS)	Sodium Silicate Solution
SiO_2 (%)	45.17	82	28–33
Al_2O_3 (%)	37.59	9.5	NA
Fe_2O_3 (%)	0.50	0.03	NA
CaO (%)	0.22	NA	NA
MgO (%)	0.12	NA	NA
Na_2O (%)	0.31	8	13.7–14.3
K_2O (%)	0.07	NA	NA
TiO_2 (%)	0.50	NA	NA
SO_3 (%)	NA	≤1.0	NA
Loss on Ignition (LOI) (%)	≤14	NA	NA
Moisture (%)	≤1	NA	NA
Specific Gravity	2.63	NA	1.47–1.56
pH	4.5–6.5	9.5–10.3	11.0
Viscosity (cPs)	NA	NA	150–200
SiO_2/Na_2O Molar Ratio	NA	NA	2.1–2.3

3.4. Formulation of Geopolymer Matrices

Initially, twelve geopolymer matrices were prepared by systematically varying the three key parameters:

- NaOH molarity: 8 M, 10 M, and 12 M
- $Na_2SiO_3/NaOH$ ratio: 1.5 and 2.0
- Precursor ratio (MK: AS): 60:40 and 70:30
- From these 12 samples, six representative matrices, viz., C, D, E, F, G, and I, were selected for detailed thermal evaluation, based on prior FTIR, SEM, and XRD screening

Table 3. Composition of selected geopolymer matrices

Sample	NaOH (M)	Na_2SiO_3 / NaOH	MK:AS	Si/Al ratio
C	8M	2:1	60:40	1.92
D	8M	2:1	70:30	2.05
E	10M	1.5:1	60:40	1.88
F	10M	1.5:1	70:30	2.01
G	10M	2:1	60:40	2.15
I	12M	1.5:1	60:40	1.90

3.5. Preparation of Alkaline Activator Solutions

Sodium hydroxide pellets were gradually dissolved in distilled water under controlled conditions to prevent excessive heat from the highly exothermic reaction, which releases a significant amount of energy. The prepared NaOH solutions were left to settle for 24 hours to ensure they were fully dissolved and had reached thermal stability. Geopolymer-grade sodium silicate was then added to the NaOH solution to prepare the alkaline activator solution. Liquid sodium silicate is added to the sodium hydroxide solution before mixing with the precursors to avoid early gel formation. Later, the alkaline activator solution is added to the precursors.

3.6. Synthesis of Geopolymer Matrices

The precursor powders were first blended using a mechanical mixer to obtain a uniform dry mixture. The alkaline activator solution was then added slowly while mixing continued. Mixing was carried out until a homogeneous slurry was obtained. The slurry was poured into moulds and compacted by vibration to reduce entrapped air. All specimens remained at room temperature for 24 h before oven curing at 80 °C for an additional 24 h. After curing, the samples were demoulded and stored under laboratory conditions until testing.

3.7. Thermal Analysis

Thermal analysis of the geopolymer matrices was performed using thermogravimetric analysis (TGA–DTG) and differential scanning calorimetry (DSC). These techniques were used to study mass loss and thermal changes in the geopolymer matrices during heating at a selected temperature range.

3.7.1. Thermogravimetric Analysis (TGA–DTG)

TGA–DTG was performed using a NETZSCH STA 2500 Regulus system. Powdered specimens were heated from 30 °C to 1000 °C at a heating rate of 10 °C /min under a nitrogen gas atmosphere. The results were used to evaluate and study the changes in mass loss of samples during heating and to identify thermal decomposition stages during heating.

3.7.2. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) was used to assess the thermal stability and thermal transitions of the geopolymer matrices. DSC analysis was performed using a NETZSCH DSC 214 Polyma instrument. All the samples were heated from room temperature to 1000 °C at a rate of 10 °C/min under a nitrogen atmosphere. The results were used to study heat-flow behaviour and thermal changes during heating.

3.8. Data Collection and Statistical Analysis

TGA and DSC were used to study the thermal behaviour of the geopolymer matrices. TGA measured mass loss during heating, and DSC identified thermal transitions and heat-flow changes. Thermal data were studied using NETZSCH Proteus software, which includes baseline correction, peak identification, and curve generation.

3.9. Instruments and Software

The following instruments were used in this study.

1. NETZSCH STA 2500 Regulus for Thermogravimetric analysis and Derivative Thermogravimetry
2. NETZSCH DSC 214 Polyma for Differential Scanning Calorimetry (DSC)
3. Mechanical mixer for Homogeneous mixing of metakaolin, Aluminum silicate precursors and liquid alkaline activators solution.
4. Hot air oven for curing the matrices

3.10. Summary of Methodological Workflow and Limitations

The study was conducted in two stages:

1. Geopolymer matrices were prepared under controlled conditions.
2. Thermal characterization was evaluated using TGA–DTG and DSC analyses. The same raw material precursors, preparation procedures, and testing conditions were used for all samples to ensure a good and consistent valuation. The thermal analysis focused on the thermal behaviour of the matrices during continuous heating. The thermal performance was supported by the previously analyzed FTIR, XRD, and SEM results. This approach provided a basis for comparing the thermal performance and stability of the selected geopolymer matrices.

4. Results

Thermal analysis was employed to evaluate the decomposition behaviour, mass loss, and thermal stability of all the geopolymer matrices. Though twelve formulations (A–L) were prepared under controlled conditions, detailed thermal analyses were conducted on all the six representative matrices (C, D, E, F, G, and I). All the examined samples showed similar decomposition stages during heating, but notable differences were observed in mass loss, position and DTG peak position and intensity, and residual Mass at the end of the run. These variations indicate that the thermal behaviour of the geopolymer matrices is strongly influenced by alkaline activator composition and the ratio of the precursors.

4.1. Thermogravimetric Analysis (TGA–DTG): Mass-Loss Behaviour

The TGA results showed that all geopolymer matrices exhibited a similar multi-stage mass-loss during heating. Mass loss occurred in four different temperature regions: moisture removal, dehydroxylation, and high-temperature structural changes. However, the amount of mass loss varied among the matrices.

DTG analysis revealed a major peak between 120–180 °C, related to the loss of adsorbed moisture, and a second peak between 350–450 °C, associated with dehydroxylation and partial decomposition. The mass-loss values for each temperature region are summarized in Table 4.

Table 4. Mass-loss distribution of geopolymer matrices across characteristic temperature regions derived from TGA analysis

Sample	30–200 °C (%)	200–400 °C (%)	400–800 °C (%)	800–1000 °C (%)	Residual Mass (%)
C	0.09	10.84	3.02	–	86.05
D	0.07	10.28	2.59	–	87.03
E	0.08	9.51	3.8	–	86.56
F	0.11	8.19	2.77	4.06	84.89
G	0.1	9.93	3.5	–	86.42
I	0.05	8.87	3.12	–	87.96

4.1.1. Region I (30–200 °C)

Physically Adsorbed Moisture (30–200 °C). All matrices displayed an initial mass loss due to the removal of free and weakly bound water in the samples. Sample I exhibited the lowest mass loss, whereas Sample C showed a relatively higher mass loss, indicating greater moisture content. This difference may be due to variations in microstructure and gel development.

4.1.2. Region II (200–400 °C)

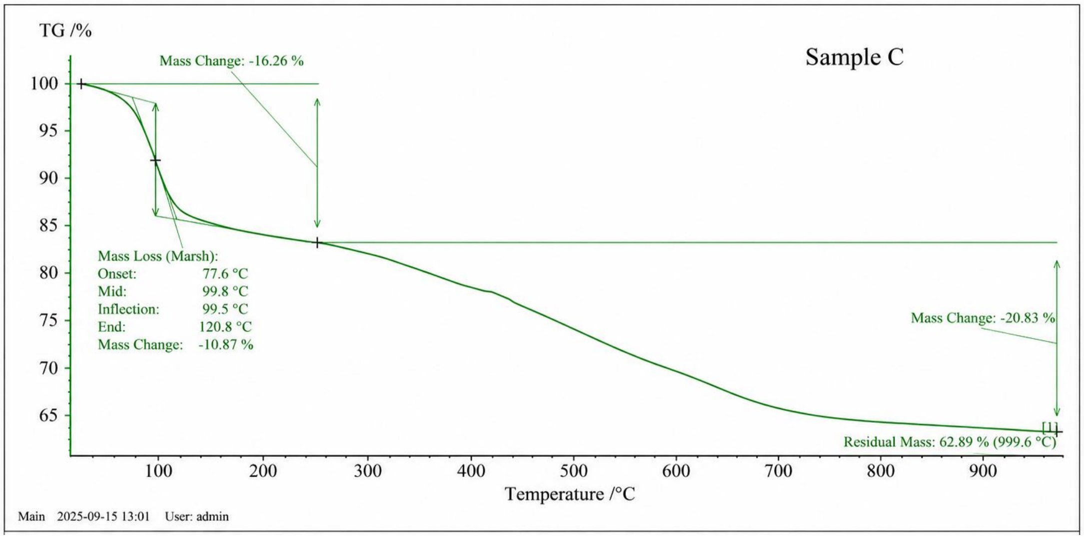
Release of bound water and dehydroxylation. This stage is related with the removal of bound water from the geopolymer during heating. Samples I, D, and E showed smooth and low-intensity DTG peaks, indicating gradual dehydroxylation and good thermal stability. Sample C showed a sharper DTG peak, indicating the presence of a higher amount of chemically bound water in the geopolymer matrix.

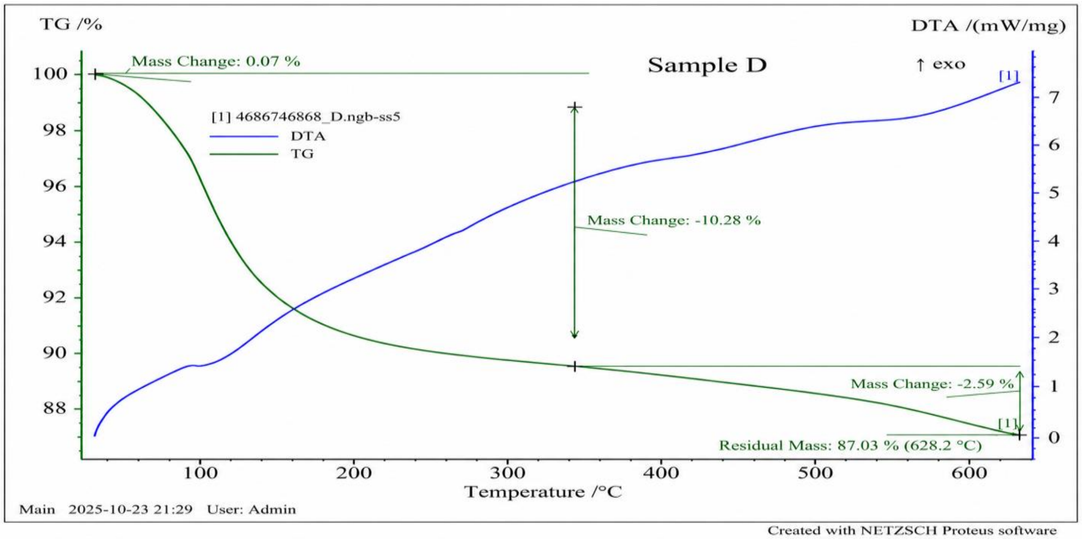
4.1.3. Region III (400–800 °C)

Major dehydroxylation and carbonate decomposition occurred at 400–600 °C This stage represents the important thermal changes in the geopolymer matrix. Samples I, D, and G showed low mass loss and broader DTG peaks, indicating good thermal stability. In contrast, Sample C exhibited sharper DTG peaks and higher mass loss, suggesting the greater structural changes in the matrix during heating.

4.1.4. Region IV (800–1000 °C)

This region shows the high-temperature Stabilization range of 600–1000 °C. In this region, Sample I showed the highest residual Mass with small changes in the Mass, indicating the best thermal stability. Samples D, E, and G remained stable with only minor mass loss, and Sample F showed slightly higher mass loss, suggesting a comparatively lower thermal stability at elevated temperatures.

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Residual Mass: 87.03 % (628.2 °C)
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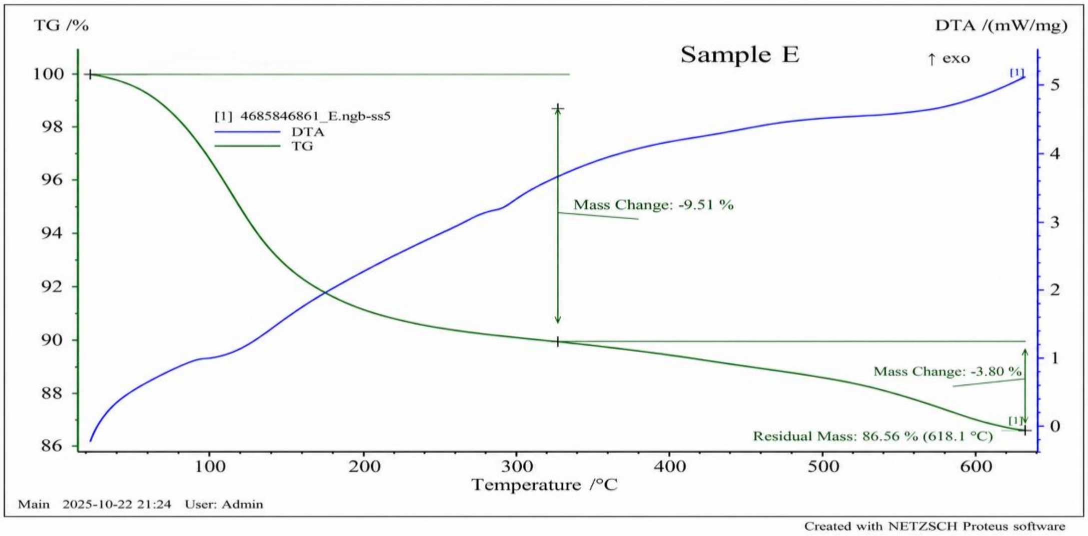
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Mass Change: -3.80 %
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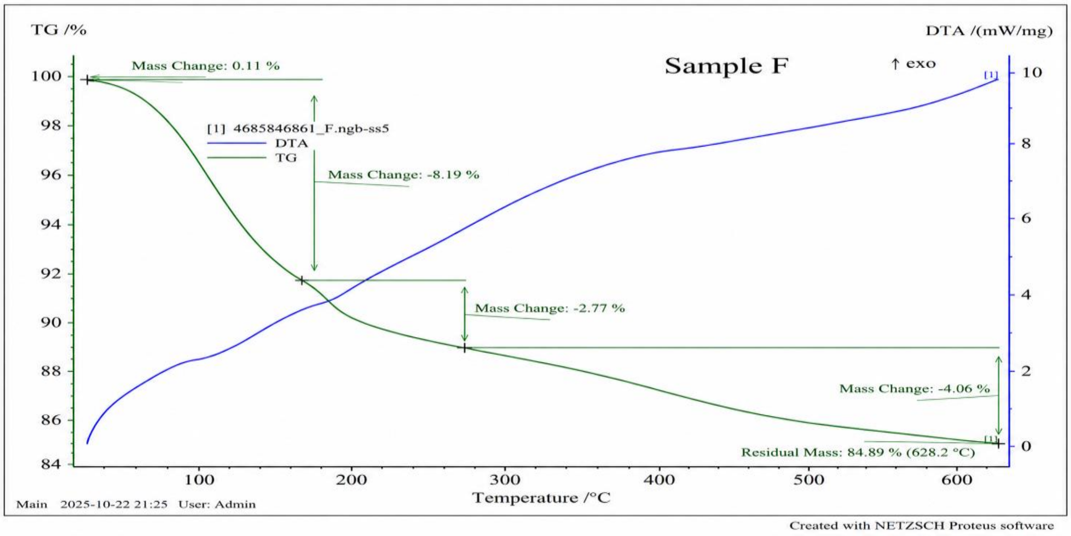
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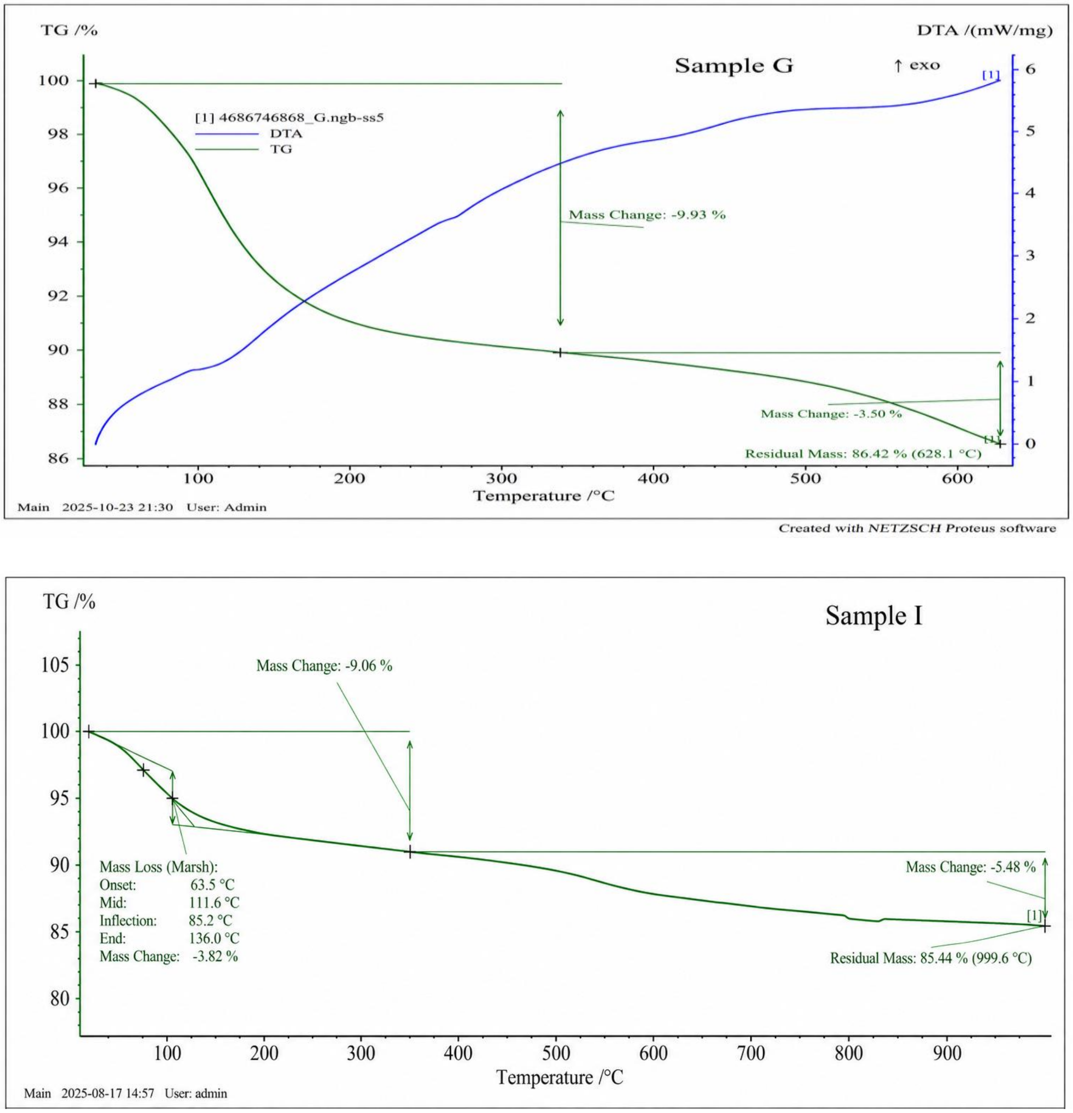


Fig. 2 Derivative thermogravimetric (DTG) curves of geopolymer matrices C, D, E, F, G, and I were obtained using the NETZSCH STA 2500 Regulus from 30–1000 °C under nitrogen flow (10 °C/min)

4.2. Differential Scanning Calorimetry (DSC): Thermal Transitions

4.2.1. Glass Transition (Tg)

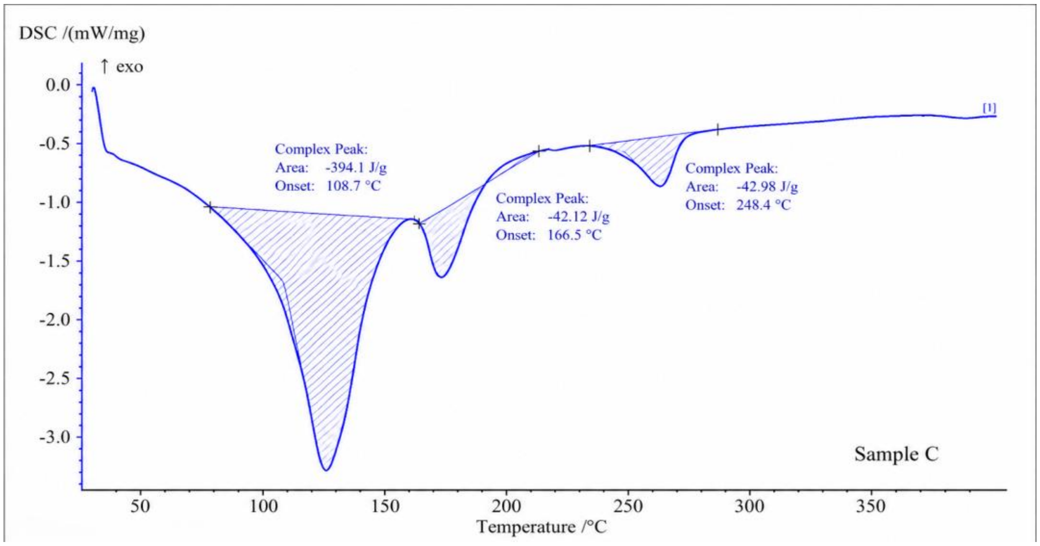
The glass transition temperature (Tg), which was measured at a slightly higher than the room-temperature in all the matrices. Sample I exhibited a slightly higher Tg, indicating a more stable and rigid geopolymer network, whereas sample C exhibited lower Tg, which is consistent with the increased moisture content and lower network rigidity.

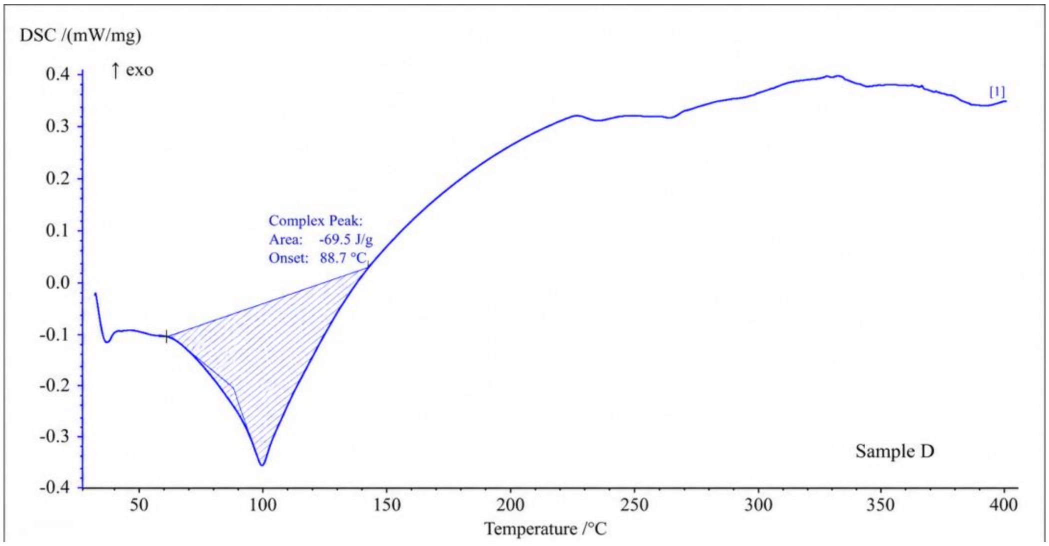
4.2.2. Exothermic Rearrangement (~380–382 °C)

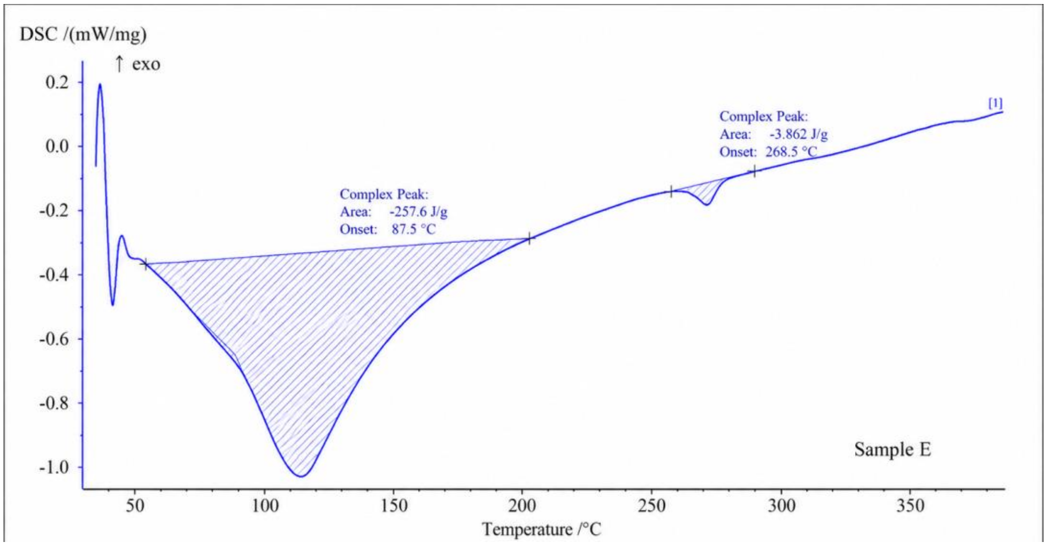
All the samples exhibited a distinct exothermic peak during heating. Sample E exhibited the highest reaction enthalpy. Samples I and D showed moderate enthalpy values, and Sample C displayed a sharper but lower intensity exothermic peak. Sample I recorded the highest glass transition (Tg) of 128 °C, indicating a more condensed and cross-linked geopolymer network. Samples G and D also showed relatively high and elevated glass transition values. The exothermic peak temperature observed for all the matrix formulations suggests a similar structural rearrangement mechanism. The DSC parameters are summarised in Table 5.

Table 5. DSC parameters of representative geopolymer matrices

Sample	Tg (°C)	Exothermic Peak (°C)
C	112	381
D	121	380
E	118	382
F	115	381
G	124	380
I	128	381


DSC /(mW/mg)
↑ exo
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
-3.0
[1]
Complex Peak:
Area: -394.1 J/g
Onset: 108.7 °C
Complex Peak:
Area: -42.12 J/g
Onset: 166.5 °C
Complex Peak:
Area: -42.98 J/g
Onset: 248.4 °C
Sample C
50
100
150
200
250
300
350
Temperature /°C


DSC /(mW/mg)
↑ exo
0.4
0.3
0.2
0.1
0.0
-0.1
-0.2
-0.3
-0.4
[1]
Complex Peak:
Area: -69.5 J/g
Onset: 88.7 °C
Sample D
50
100
150
200
250
300
350
400
Temperature /°C


DSC /(mW/mg)
↑ exo
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
[1]
Complex Peak:
Area: -3.862 J/g
Onset: 268.5 °C
Complex Peak:
Area: -257.6 J/g
Onset: 87.5 °C
Sample E
50
100
150
200
250
300
350
Temperature /°C

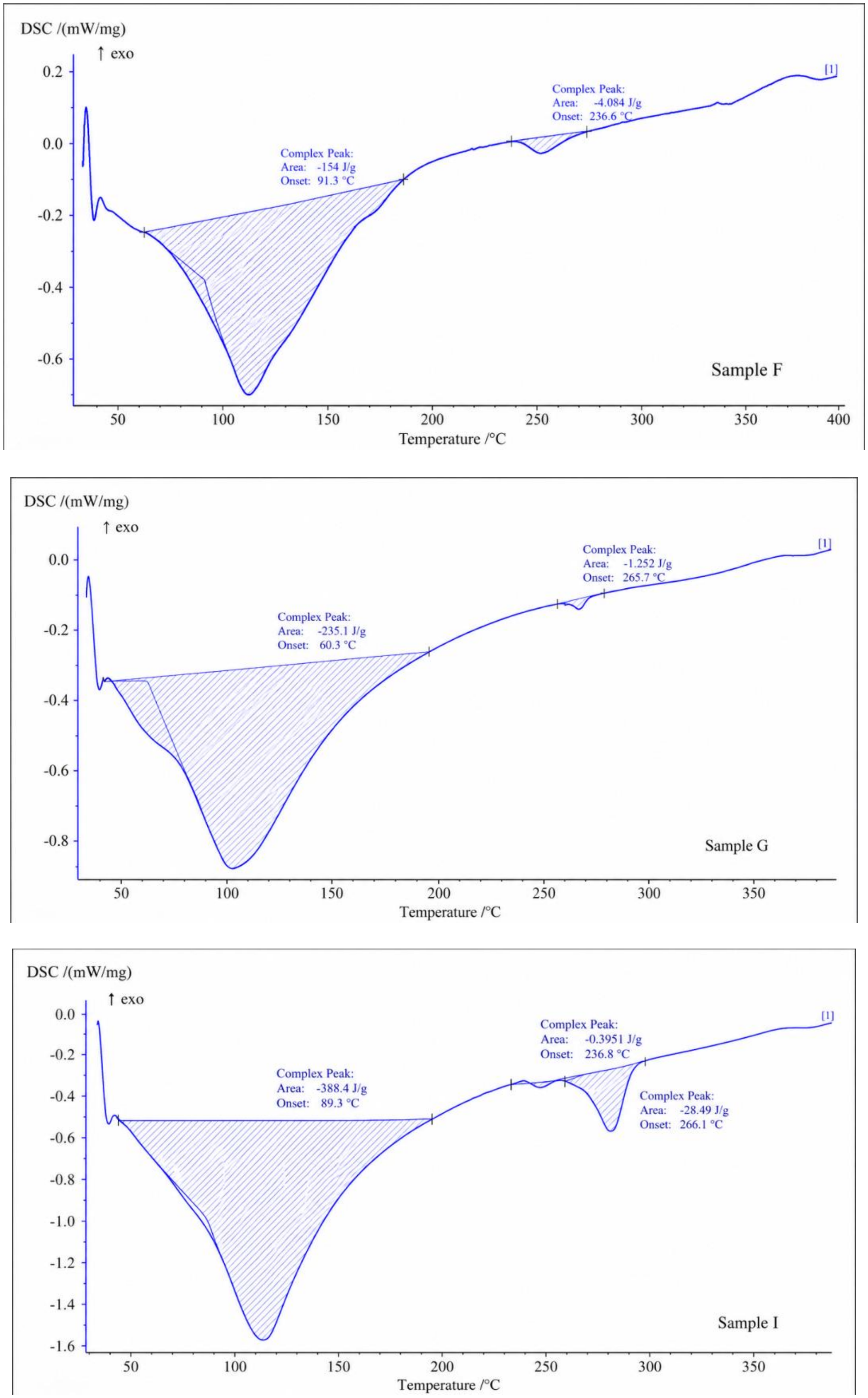


Fig. 3 Differential scanning calorimetry (DSC) thermograms of geopolymer matrices C, D, E, F, G, and I were recorded from 30–1000 °C using the NETZSCH DSC 214 Polyma under a nitrogen atmosphere (10 °C/min)

The glass-transition temperatures (Tg) varied among the investigated matrix formulations. The highest glass transitions were measured for Sample I at 128 °C, followed by Samples G and D, indicating a better thermal stability. The relatively small variation in exothermic peak suggests that similar structural changes occurred within the geopolymer network across all formulations.

4.3. Comparative Thermal Stability and Matrix Ranking

Based on the combined of TGA and DSC results, the thermal stability ranking was: I > D > E > G > F > C. where Sample I showed the highest thermal stability, with the lowest mass loss, the highest residual Mass, and steady thermal transitions. Samples D, E, and G exhibited moderate thermal stability, while Sample C showed the highest mass loss with the lowest thermal stability. The ranking was consistent across repeated tests and agree with previous FTIR and XRD microstructural analyses, indicating that thermal stability and chemical reactivity are not always directly related.

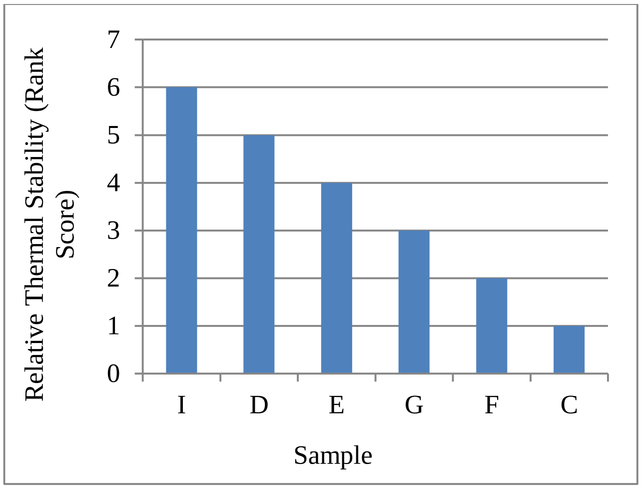


Fig. 4 Thermal stability ranking of geopolymer matrices based on TGA-DTG and DSC analyses

5. Discussion

The TGA, DTG, and DSC analyses provided valuable information on the thermal behaviour of the geopolymer matrices. Differences in mass loss, thermal transitions, and residual Mass showed that the samples had variations in thermal stability. Overall, the observed results were similar to those reported for other alkali-activated geopolymer materials.

5.1. Summary of Key Findings

The geopolymer matrices showed multi-stage thermal behaviour during heating, including moisture loss, dehydroxylation, and structural changes. However, differences were observed in mass loss, residual Mass, and thermal transitions. Sample I showed the best thermal stability with lower mass loss, smoother DTG peaks, and higher residual Mass. Sample C showed greater mass loss and lower thermal stability. DSC analysis revealed similar thermal transition temperatures for all formulations, but variations in heat-flow behaviour indicated differences in geopolymer development and structural consolidation.

5.2. Interpretation of Thermal Behaviour

Sample C showed higher mass loss, which indicates a greater amount of water content and thermally removable compounds in the geopolymer matrix. Samples I and D exhibited lower mass loss, which suggests a denser and more thermally stable aluminosilicate network. DSC results exhibited similar thermal transitions for all matrices, which indicates structural changes due to high temperature. However, differences in mass loss and heat-flow behaviour suggest variations in geopolymer network development. Samples I and G showed higher glass-transition temperatures, indicating better structural stability. These findings support the findings obtained from previous FTIR and XRD results.

5.3. Study Limitations

This study focused on the thermal characterization of six metakaolin-based geopolymer matrices selected after FTIR, XRD, and SEM microstructural analyses. Thermal behaviour was related to microstructural observations; factors such as pore size, pore distribution, and gel densification were not measured.

In addition, the study considered only controlled laboratory heating conditions. The effects of thermal cycling, thermal shock, and long-term exposure to high temperatures were not investigated. Future studies should examine these factors to better understand geopolymer performance.

5.4. Implications for Material Selection

These results show that material selection should depend on the specific application. Sample I showed good thermal stability, lower mass loss, high residual Mass, and thermally stable behaviour during heating. Sample C showed lower thermal stability but demonstrated good geopolymerisation characteristics from the previous FTIR and XRD analyses.

This shows that thermal stability and chemical reactivity are not always directly related. Therefore, both thermal performance and geopolymerization behaviour should be considered when selecting geopolymer formulations for specific applications.

5.5. Comparison with Previous Studies

The thermal behaviour observed in this study was similar to the previously studied metakaolin-based geopolymers. All samples showed moisture loss, dehydroxylation, and structural changes during heating. The results confirmed that the alkaline activator composition affects the thermal stability of the matrix. Sample I showed good thermal performance, while Sample C showed the lowest. DSC transition temperature results indicated similar thermal transition temperatures for all matrices, suggesting comparable thermal transformation. The thermal stability ranking (I > D > E > G > F > C) offers a useful comparison for selecting geopolymer matrices for high-temperature applications.

5.6. Environmental Impact and Sustainability

Metakaolin-based geopolymers have been investigated as alternatives to conventional cementitious systems because of their reduced use of clinker and potential thermal durability. The reduced clinker dependence may contribute to lower environmental impact compared with conventional Portland cement, as reported in previous sustainability valuations of alkali-activated binder [19, 33].

In addition, improved thermal resistance may contribute to longer service performance under elevated-temperature conditions. However, the production of alkaline activators such as sodium hydroxide and sodium silicate still involves energy-intensive processing. Therefore, environmental assessment of geopolymer systems should consider both reduced clinker dependence and the energy requirements associated with alkaline activator production.

5.7. Advanced Characterization Perspective

The thermal behaviour of the geopolymer matrices was related to their chemical structure and microstructure. FTIR and XRD analyses showed the differences in geopolymer network development and amorphous phase among the formulations.

The combined FTIR, XRD, TGA–DTG, and DSC results improved the understanding of the relationship between precursor composition, microstructure, and thermal performance. Future studies should include detailed microstructural analysis to further explain thermal stability.

5.8. Long-Term and Cyclic Thermal Stability

The current analysis evaluated thermal behavior under continuous heating. But, in practical use, geopolymer may be exposed to repeated heating and cooling cycles. Cyclic heating of geopolymer materials can result in microcracking, structural fatigue and degradation. Future studies investigate cyclic thermal loading and thermal resistance of geopolymer materials for long-term applications at high temperatures.

6. Conclusion

This study evaluated the thermal behaviour of six metakaolin-based geopolymer matrices using TGA-DTG and DSC. Thermogravimetric Analysis (TGA–DTG) and Differential Scanning Calorimetry (DSC) revealed that all matrices exhibited characteristic multi-stage thermal decomposition related to moisture loss, dehydroxylation, and high-temperature structural transformation.

Sample I exhibited the best thermal performance, with the lowest mass loss and highest residual Mass (87.96%). Sample C showed higher mass loss but demonstrated good geopolymerisation characteristics in previous FTIR and XRD analyses. The thermal stability ranking was: I > D > E > G > F > C. Overall, the results confirm that alkaline activator composition and precursor ratio strongly influence thermal behaviour of the geopolymer matrices and provide guidance for high-temperature applications.

6.1. Future Work

The findings of this study provide a foundation for the development of fibre-reinforced geopolymer composites for structural and high-temperature applications. Future investigations will focus on incorporating natural and hybrid fibre reinforcements into the selected geopolymer matrices identified in the study.

- Fabrication of Fibre-reinforced geopolymer composites using the thermally stable and structurally enhanced matrices identified in this study.
- Assessment of mechanical properties like tensile, flexural, impact, compression, and interlaminar shear properties of fibre-reinforced geopolymer composite laminates.
- Study of fibre–matrix interfacial behaviour through microstructural characterization using SEM-EDS.
- Evaluation of long-term durability under cyclic thermal exposure and thermal shock.
- Development and fabrication of Fibre-reinforced geopolymer composite for applications requiring thermal resistance, structural integrity, and sustainable composite material.

These future studies will improve the understanding of the relationship between composition, structure and performance in geopolymer composite materials and support their usage in advanced engineering applications.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethics Statement

This research did not involve human participants or animals. Therefore, ethical approval and informed consent were not required.

Data Availability

The datasets generated and/or analysed during the current study are available from the corresponding author on reasonable request.

Author Contributions

Iynthezhuthon K.: Conceptualization, Methodology, Investigation, Data curation, Visualization, Writing – original draft, Writing – review & editing.

Ganapathy Subramanian L. R.: Supervision, Project administration, technical guidance, Validation, Writing – review & editing.

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