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Thermal Degradation Behaviour and Residual Mass Characteristics of Cassava Cortex–Cow Bone-Reinforced Epoxy Composites

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Abstract

This study compares thermal degradation and residual-mass behaviour in three cassava cortex–cow bone particulate epoxy formulations using thermogravimetric (TG) and derivative thermogravimetric (DTG) curves. The specimens comprised a hybrid with 20 vol.% each of cow bone and cassava cortex (RDT4PS100), a 40 vol.% cow-bone composite (RBT0PS100) and a 40 vol.% cassava-cortex composite (RFT4PS500). At a programmed heating rate of 10 °C/min, the estimated principal DTG peaks were approximately 400, 415 and 400 °C, respectively; associated peak mass-loss-rate magnitudes were approximately 4.2, 2.5 and 5.0%/min. Residues at the plotted endpoints were approximately 16%, 16–17% and 5%, respectively. The cow-bone-only formulation showed the smallest principal degradation-rate magnitude, whereas the hybrid and cow-bone-only specimens retained substantially more mass than the cassava-cortex-only specimen. The results distinguish peak degradation temperature, degradation-rate intensity and non-volatile residue as separate descriptors. Because particle size and treatment also differed between formulations, the observations support a descriptive formulation-level comparison rather than an isolated hybridisation effect. The findings provide preliminary thermal-characterisation evidence for selecting formulations for further engineering assessment. The comparison uses a fixed total reinforcement fraction of 40 vol.% and separates thermogram-derived evidence from possible compositional explanations. The findings are relevant to screening waste-derived reinforcement systems, but they do not establish fire resistance, mechanical retention during heating or permissible automotive service temperatures. Further matched-control measurements and replicate-level analysis are needed before those engineering properties can be assessed.

Keywords: Cassava Cortex, Cow Bone, Epoxy Composites, Thermogravimetric Analysis, Derivative Thermogravimetry, Residual Mass

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1. INTRODUCTION

Agricultural and animal-processing residues can provide complementary reinforcement phases for epoxy composites. Cassava cortex is a lignocellulosic by-product, whereas cow bone contributes an organic–mineral mixture whose non-volatile constituents may remain after much of the polymeric fraction decomposes. Combining these constituents offers a materials-utilisation route, but the engineering value of a formulation depends on more than the quantity of residue remaining after heating. In particular, the onset of degradation, temperature of maximum degradation rate and ultimate residue represent different responses. These should not be used interchangeably to claim high-temperature service capability (Mahmud et al., 2025; Molla & Belay, 2024; Hiremath et al., 2024).

Thermogravimetric analysis (TGA) measures the mass of a specimen as a function of temperature or time under a specified heating programme. Its derivative (DTG) highlights the position and intensity of mass-loss events. The observed profiles can reflect moisture release, decomposition of organic filler fractions, epoxy network degradation and persistence or transformation of mineral components; assigning a specific mechanism requires additional evidence and a known test atmosphere. Surface treatment, particle size and filler loading can alter the profile by changing composition and interfacial conditions (Rahman et al., 2025; Soni & Sinha, 2022; Alsuwait et al., 2023). For comparisons involving bone and plant-derived fillers, the distinction between mass retention and functional thermal resistance is particularly important.

1.1 Recent research and knowledge gap

Natural-fibre/epoxy composite research has established that fibre chemistry, matrix formulation and interfacial processing influence the thermal response. Mahmud et al. (2025) reviewed the preparation and applications of natural-fibre epoxy systems, while Alsuwait et al. (2023) considered processing, physical performance and thermal behaviour across a broader group of epoxy biocomposites. The thermal-focused reviews of Hiremath et al. (2024) and Kumar et al. (2023) distinguish TG mass retention from the temperatures and rates associated with DTG events. These distinctions are important because a high residue at the end of a heating programme does not, by itself, establish a higher temperature of rapid decomposition. Related work by Daodu et al. (2025) reviewed the roles of reinforcement composition, particle characteristics and processing in cassava-cortex/cow-bone polymer composites, providing a direct materials-specific foundation for the present thermal comparison.

Surface modification can alter fibre chemistry and fibre–matrix adhesion, but its thermal effects are not universal. Soni and Sinha (2022) investigated alkali and silane treatments in hemp/epoxy composites, and Rahman et al. (2025) examined treated Borassus husk fibres in heat-resistant epoxy. Ghori et al. (2024) studied alkali-treated date-palm/kenaf hybrid epoxy composites, while Kar et al. (2026) reported thermal characterisation of NaOH-treated Calamus tenuis epoxy composites. These studies support interpreting alkali treatment alongside reinforcement content and fibre type rather than treating it as an independent guarantee of higher degradation temperature.

Mineral-rich and mixed-filler systems offer a complementary comparison. Molla and Belay (2024) fabricated ox-bone-powder/bamboo-fibre epoxy composites, principally assessing mechanical properties and moisture uptake. Kabave Kilincarslan et al. (2023) evaluated surface-modified cow-bone-powder polymer composites and reported changes in thermal-related material properties. Karagöz et al. (2025) investigated walnut-shell/talc epoxy composites, and Kanaginahal et al. (2024) examined eggshell-particle epoxy systems. Taken together, these studies motivate separate reporting of non-volatile residue, DTG peak temperature and mass-loss rate when an organic filler is combined with a mineral-bearing reinforcement.

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Additional work illustrates the diversity of biomass-based composite responses. Mohammad Taib (2025) studied oil-palm-trunk-derived fillers in epoxy using thermal and mechanical characterisation; Haque and Islam (2024) examined waste jute and sawdust in epoxy, including thermal analysis. Tasgin et al. (2024) compared the thermal transport and other properties of cotton-, sisal-, coir- and wool-reinforced epoxy materials; their thermal conductivity measurements should not be confused with TG degradation parameters. Oladele et al. (2025) investigated cyclic thermal treatment of bagasse-particle epoxy composites, illustrating how reinforcement processing can influence composite performance. Mylsamy et al. (2025) reviewed the wider consequences of fibre hybridisation, including its limits and processing challenges. In a separate cassava-cortex-based biocomposite system, Daodu and Agbonko (2026) found that sorbitol content altered degradation thresholds, DTG peak temperatures and high-temperature residues in a non-monotonic manner. Although that study used different formulations, its findings reinforce the need to interpret these thermal indicators separately.

Quantitative interpretation also depends on measurement quality. Xie et al. (2025) examined error analysis in thermogravimetric measurements, reinforcing the need to distinguish raw instrument-export values from estimates read off plotted traces. For the present study, original numerical TGA files are unavailable. Accordingly, the analysis is confined to the three reproduced TG/DTG records, with peak temperatures, peak mass-loss-rate magnitudes and terminal residues explicitly described as approximate readings.

Previous studies have addressed natural-fibre epoxy composites, bone-derived mineral fillers and thermal characterisation, but direct comparisons among cassava-cortex-only, cow-bone-only and hybrid particulate epoxy formulations remain limited. Daodu et al. (2025) reviewed the characterisation and optimisation of cassava-cortex and cow-bone particulate polymer composites, establishing the materials context for this investigation. In a related study, Daodu et al. (2025) examined the tribological behaviour of cow-bone–cassava-cortex epoxy composites under different loads and sliding speeds. The present study extends the characterisation of this material system to thermal degradation and residual mass. The three selected formulations also differ in particle size and chemical treatment; consequently, the comparison describes their overall thermal responses rather than isolating the effect of hybridisation. The authors' related tribological investigation (Daodu et al., 2025a) addressed wear performance rather than thermal decomposition. The present work therefore adds a distinct TG/DTG perspective without implying that wear behaviour predicts thermal stability. The research question is whether the three selected formulations differ consistently across three distinct TG/DTG descriptors, and whether the apparent residue advantage of cow-bone-containing composites is accompanied by a corresponding change in the temperature and intensity of their principal mass-loss event. This question is answered using the available plotted records without inferring causal effects from unmatched processing conditions.

2. MATERIALS AND METHODS

2.1 Materials and composite preparation

Cassava cortex and cow bone were processed as particulate reinforcements for a diglycidyl ether of bisphenol A (DGEBA) epoxy matrix. The cassava cortex was cleaned to remove adhering soil, dried by sun exposure or low-temperature oven drying, milled and sieved. Alkali treatment was applied to the cassava cortex in treated formulations, with 0, 2 and 4 wt.% NaOH levels considered in the experimental design. Cow bones were cleaned and boiled to remove residual tissue, dried at 105 °C for 24 h, and calcined at 600–800 °C for 2–3 h before milling and sieving. The prepared reinforcements were stored dry before composite fabrication.

The epoxy resin and hardener were combined at a ratio of 2:1 and mixed mechanically. Reinforcement powders were added gradually under continuous stirring, with cow-bone and cassava-cortex powders

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premixed for the hybrid formulation. Each composite contained 60 vol.% epoxy system and 40 vol.% total reinforcement (Table 1). The mixtures were cast into moulds, vacuum-degassed and cured initially for 24 h, followed by post-curing; the post-curing temperature and duration were not specified in the available record. The investigated formulations were RDT4PS100 (20 vol.% cow bone and 20 vol.% cassava cortex, 4 wt.% NaOH treatment, 100 µm), RBT0PS100 (40 vol.% cow bone, no alkali treatment, 100 µm) and RFT4PS500 (40 vol.% cassava cortex, 4 wt.% NaOH treatment, 500 µm).

Table 1 Composition and processing conditions of the composites evaluated by TGA.

Formulation	Cow bone (vol.%)	Cassava cortex (vol.%)	NaOH treatment	Particle size (µm)
RDT4PS100	20	20	4 wt.%	100
RBT0PS100	40	0	None	100
RFT4PS500	0	40	4 wt.%	500

2.2 Thermogravimetric measurements and analytical approach

Thermogravimetric analysis of the composites was performed using a PerkinElmer thermogravimetric system under nitrogen at a heating rate of 10 °C/min. The programmed temperature range was 30–950 °C; the thermograms presented here extend to approximately 884–888 °C. The initial masses displayed for RDT4PS100, RBT0PS100 and RFT4PS500 were 16.103, 16.859 and 16.617 mg, respectively. Three specimens were reportedly tested per formulation, and the available plots were presented as mean responses; the individual replicate records were not available for independent confirmation. The nitrogen purge-flow rate was not recorded in the available thesis or manuscript materials. Separate measurements of reinforcement powders were conducted from room temperature to 800 °C at 10 °C/min.

The temperature of the principal DTG peak was identified from the dominant mass-loss-rate extremum. Peak mass-loss rates were estimated from the DTG ordinates, and residual mass was determined at the endpoint of each plotted TG curve. Accordingly, the values read from the thermograms are reported as approximate. Residual fractions correspond to the plotted endpoints, rather than the programmed maximum temperature of 950 °C. Dispersion among the three specimen measurements is not reported because individual replicate values are not available. The instrument plots are treated as the available evidence; raw time–mass–temperature exports were not provided for independent reprocessing.

2.3 Comparative analysis

The formulations were compared descriptively using the principal DTG peak temperature, magnitude of the dominant mass-loss rate and plotted-endpoint residue. Differences in mass retention were expressed in percentage points, and ratios were used only as descriptive comparisons. Because reinforcement type, treatment status and particle size are not held constant across all specimens, differences cannot be causally assigned to any one processing or composition variable. Although three specimens per formulation were tested and mean results reported.

3. RESULTS AND DISCUSSION

3.1 Thermal mass-loss profiles

Figures 1–3 present the TG/DTG profiles of the selected formulations. The main DTG extrema occur at approximately 400 °C for RDT4PS100, 415 °C for RBT0PS100 and 400 °C for RFT4PS500. The respective peak-rate magnitudes are estimated at 4.2, 2.5 and 5.0%/min. The cow-bone-only formulation thus has the least intense dominant mass-loss-rate peak, whereas the cassava-cortex-only formulation has

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the most intense peak. The approximately 15 °C difference between the cow-bone-only peak and the other two peaks is a plot-based estimate, not an experimentally resolved or statistically established shift. Because the plotted DTG axes have different scales, peak-rate comparisons must be based on the numerical ordinates rather than the visual depth of each curve.

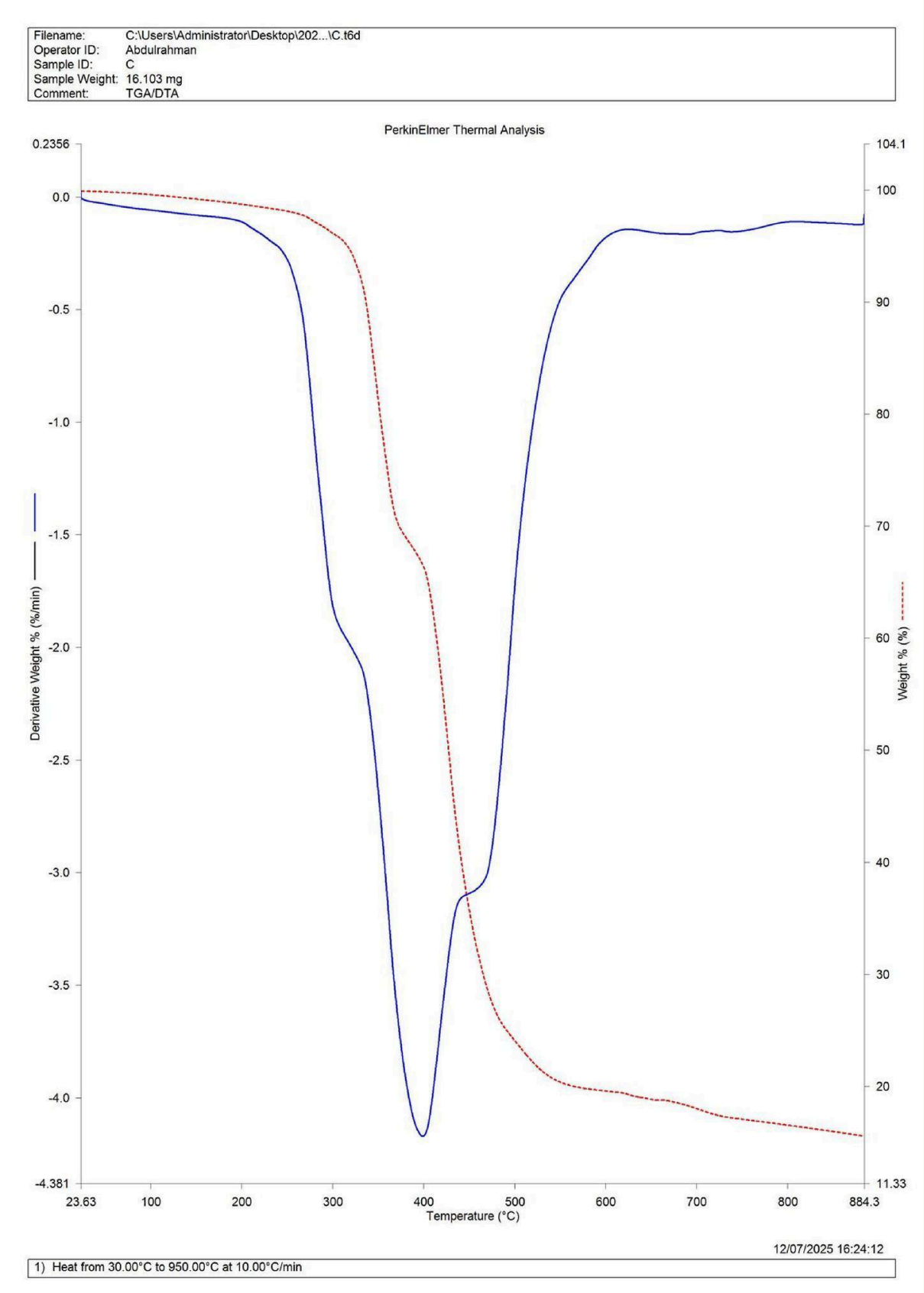


Figure 1 – TG/DTG thermogram of the hybrid composite RDT4PS100.

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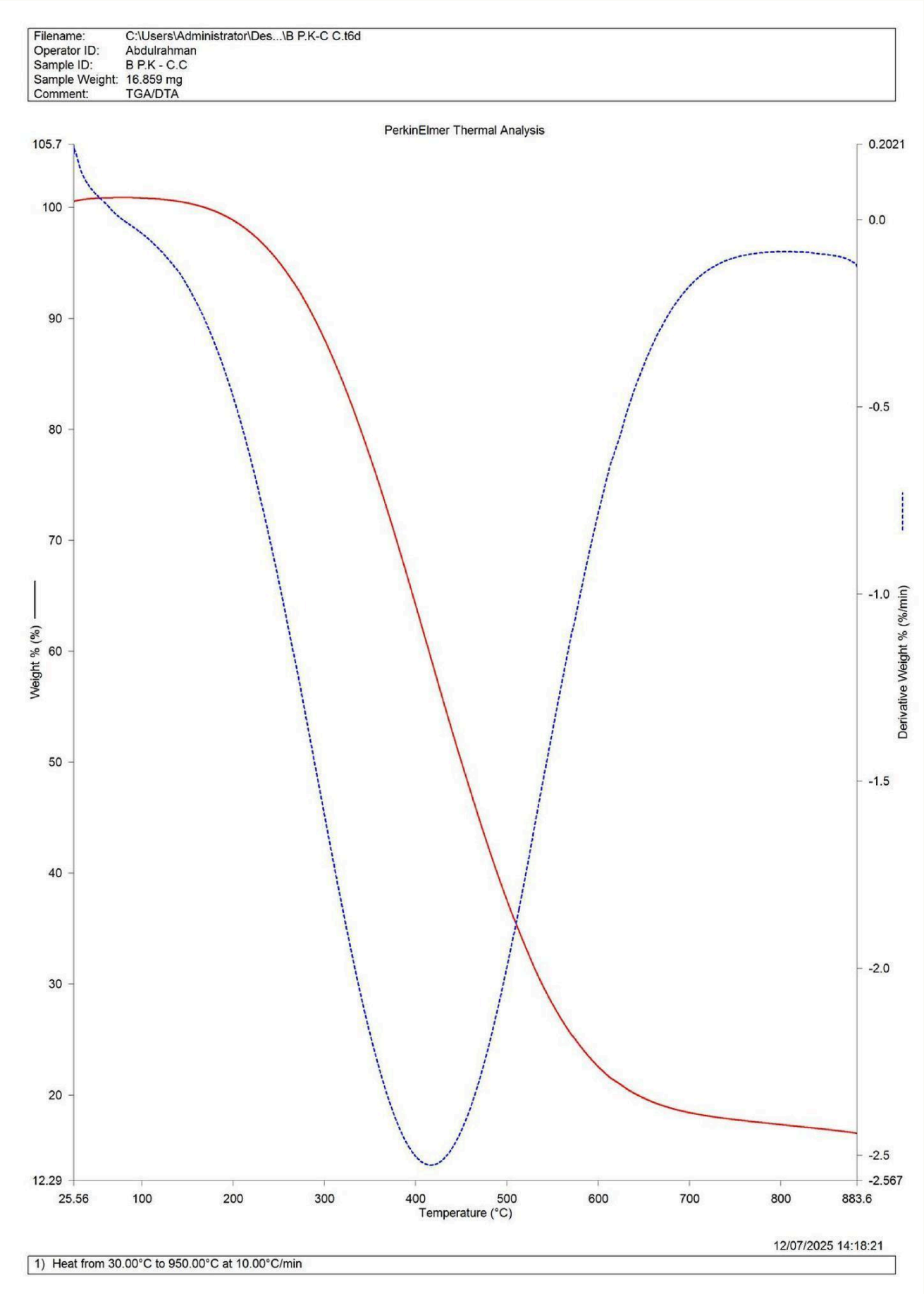


Figure 2 – TG/DTG thermogram of the cow-bone-reinforced composite RBT0PS100.

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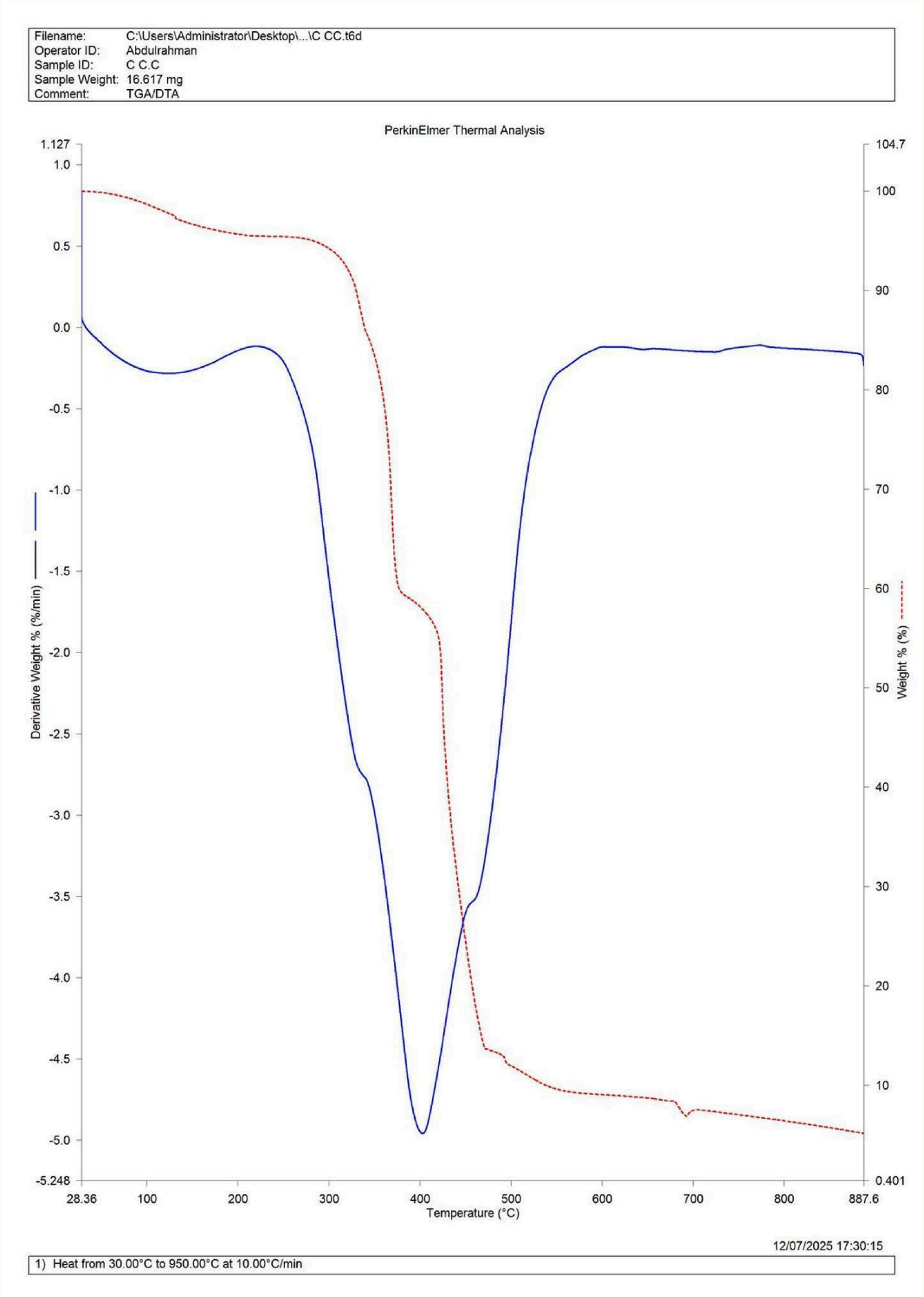


Figure 3 – TG/DTG thermogram of the cassava-cortex-reinforced composite RFT4PS500.

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3.2 Quantitative thermal parameters and residual mass

Table 2 Thermal degradation parameters determined from TG/DTG thermograms (approximate values).

Formulation	DTG peak temperature (°C)	Peak mass-loss rate (%/min)	Endpoint residue (%)
RDT4PS100	≈400	≈4.2	≈16
RBT0PS100	≈415	≈2.5	≈16–17
RFT4PS500	≈400	≈5.0	≈5

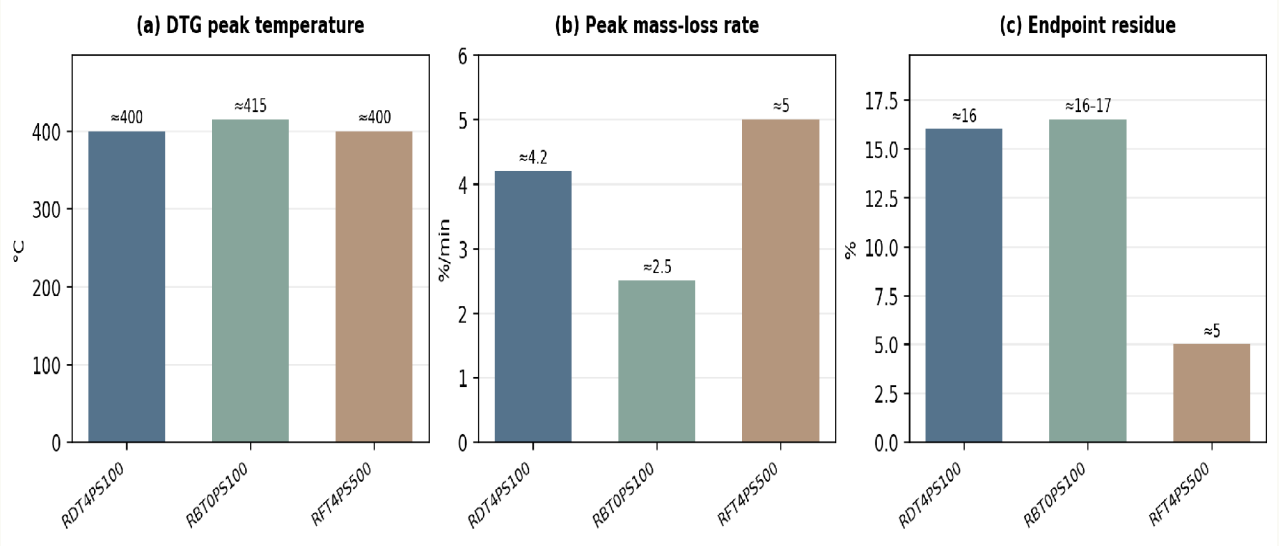


Figure 4 – Comparison of (a) principal DTG peak temperature, (b) peak mass-loss-rate magnitude and (c) residual mass at the plotted endpoint for the three composite formulations.

The separately reported powder measurements gave residues at 600 °C of 16.54% for cow bone and 9.30% for cassava cortex (Table 3), a difference of 7.24 percentage points (16.54 − 9.30). This comparison is consistent with different non-volatile contributions from the two raw reinforcements. It does not establish a mass balance for the epoxy composites: the powder and composite measurements concern different systems and, in this manuscript, different temperature endpoints. In addition, the powder data alone do not identify the chemical composition of the retained material.

Table 3 Residual mass of cow-bone and cassava-cortex powders at 600 °C.

Reinforcement powder	Residual mass at 600 °C (%)
Cow bone	16.54
Cassava cortex	9.30

3.3 Interpretation in relation to published research

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The three formulations show distinct combinations of thermal-response indicators. RBT0PS100 has the highest estimated principal DTG peak temperature (≈415 °C) and the lowest peak mass-loss-rate magnitude (≈2.5%/min). RDT4PS100 has a larger peak-rate magnitude (≈4.2%/min) but approximately comparable endpoint residue. RFT4PS500 combines a relatively intense principal degradation event (≈5.0%/min) with the lowest remaining mass (≈5%). The separation of these indicators matters: a formulation can retain a relatively large non-volatile fraction without resisting initial degradation or maintaining useful strength during heating. Figure 4 summarises these three complementary descriptors and highlights the contrast between the formulations. The peak-rate magnitude of RFT4PS500 is approximately twice that of RBT0PS100 (5.0/2.5).

A plausible contribution to the higher residual fractions of the cow-bone-containing formulations is the persistence of non-volatile constituents associated with the mineral-bearing reinforcement. However, the present TG/DTG curves do not quantify ash composition, establish chemical reaction pathways or demonstrate that the remaining material retains load-bearing integrity. The broader literature on natural-fibre/epoxy systems identifies treatment and reinforcement chemistry as influential factors (Mahmud et al., 2025; Rahman et al., 2025; Soni & Sinha, 2022; Alsuwait et al., 2023), while bone-containing hybrid systems provide a useful material comparison (Molla & Belay, 2024; Kabave Kilincarslan et al., 2023). The present observations are compatible with that context but cannot be treated as a direct replication of studies performed under different atmospheres or formulations. The source thesis also discusses potential effects of NaOH treatment and particle size, but these effects cannot be disentangled from reinforcement composition in the three selected formulations.

The dominant DTG peak marks the temperature of greatest measured mass-loss rate under the applied programme; it is not the onset of decomposition, a glass-transition temperature or an allowable operating temperature. Similarly, terminal residue is not a direct fire-resistance rating. The thermograms may contain earlier and later mass-loss features, but reliable stage boundaries and onset temperatures cannot be assigned from the available plots without consistent criteria and the underlying numerical data. The nitrogen atmosphere should be considered when comparing these data with measurements performed in air or other purge environments (Hiremath et al., 2024; Kumar et al., 2023; Xie et al., 2025).

In the wider literature, chemically modified hemp and Borassus husk fibres have shown that treatment changes both interface behaviour and thermal-response indicators (Rahman et al., 2025; Soni & Sinha, 2022). The date-palm/kenaf and Calamus tenuis studies likewise demonstrate that fibre fraction and treatment conditions should be interpreted together (Ghori et al., 2024; Kar et al., 2026). In the present specimens, differences in the principal DTG peaks and residue cannot be attributed to any one factor without matched-control formulations.

The residue contrast is consistent with the importance of reinforcement chemistry discussed in studies of bone powder and mineral/biomass filler combinations (Molla & Belay, 2024; Kabave Kilincarslan et al., 2023; Karagöz et al., 2025; Kanaginahal et al., 2024). Nevertheless, mineral retention after decomposition does not demonstrate that the composite maintains stiffness or strength at elevated temperatures. The distinction between decomposition and thermal transport is also important: studies measuring conductivity or diffusivity address different material functions from the mass-loss curves analysed here (Tasgin et al., 2024).

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Taken together, the TG and DTG profiles indicate that peak degradation temperature, peak mass-loss rate and residual fraction capture different aspects of the thermal response. A relatively high residual fraction may reflect non-volatile mineral constituents, while a lower DTG peak rate indicates a less intense dominant mass-loss event under the applied heating programme. These measures should therefore be interpreted together when comparing the formulations (Kumar et al., 2023; Xie et al., 2025). The related cassava-cortex/sorbitol biocomposite study of Daodu and Agbonko (2026) provides context for biomass-derived thermal behaviour, but its matrix chemistry differs from the epoxy systems examined here; numerical results should not be directly equated.

3.4 Contribution to composite material development

The original contribution of this study is a direct, formulation-level TG/DTG comparison of a cassava-cortex/cow-bone hybrid epoxy composite with cow-bone-only and cassava-cortex-only epoxy composites at a common total reinforcement fraction of 40 vol.%. Rather than relying on a single thermal-stability indicator, the analysis jointly considers the temperature of the principal mass-loss event, its rate magnitude and the mass retained at the plotted endpoint. The comparison shows that the cow-bone-only formulation exhibits the smallest estimated dominant mass-loss rate (≈2.5%/min), whereas the two cow-bone-containing formulations retain approximately 16–17% of their initial mass compared with approximately 5% for the cassava-cortex-only formulation. These observations provide a materials-specific thermal benchmark that complements the authors' earlier mechanical and tribological investigations. They also demonstrate why mineral-associated residue must not be interpreted as proof of high-temperature mechanical performance. Since treatment and particle size differ among the selected formulations, the work establishes comparative thermal behaviour without claiming a separately identified hybridisation effect. The results provide a basis for matched-control experiments and future thermal–mechanical qualification of candidate automotive components.

4. CONCLUSIONS

The three cassava cortex–cow bone particulate epoxy formulations exhibited distinct TG/DTG responses under nitrogen. The principal DTG peak temperatures were approximately 400 °C for RDT4PS100, 415 °C for RBT0PS100 and 400 °C for RFT4PS500, with corresponding peak mass-loss rates of about 4.2, 2.5 and 5.0%/min. Their residual masses at the plotted endpoints were approximately 16%, 16–17% and 5%, respectively. RBT0PS100 showed the lowest dominant mass-loss rate, while both cow-bone-containing formulations retained considerably more mass than the cassava-cortex-only formulation. These results establish differences in thermal degradation behaviour and remaining mass among the tested formulations. They do not, by themselves, establish allowable operating temperatures or isolate the influence of individual processing variables.

Data availability

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The thermograms used in this study are reproduced in Figures 1–3, and the formulation-level comparisons and plotted-endpoint values are presented in Figure 4 and Tables 1–3. Enquiries about the supporting materials may be directed to the corresponding author.

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