

Effect Of Sorbitol Content On The Thermal Stability And Degradation Behaviour Of Cassava Cortex-Reinforced Biocomposites

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Abstract

Cassava processing residues are renewable lignocellulosic resources with potential for sustainable composite development. This study evaluated the influence of sorbitol content on the thermogravimetric behaviour of chemically treated cassava cortex (CCT)–based materials. Five formulations were investigated: CCT100, SBT100 and composites containing 5, 15 and 25 wt.% sorbitol (SBTCCT5, SBTCCT15 and SBTCCT25). Thermogravimetric analysis was conducted with a NETZSCH STA 449C using 8.0 ± 1.5 mg specimens heated under nitrogen from 20 to 900 °C at 10 °C min⁻¹. Analysis of the TGA and derivative data showed composition–dependent, multistage degradation. SBTCCT5 exhibited $T_5 = 314.4$ °C and $T_{10} = 333.8$ °C, while SBTCCT25 gave $T_5 = 314.1$ °C and the highest $T_{10} = 342.0$ °C. SBTCCT15 showed lower initial–degradation thresholds ($T_5 = 231.7$ °C; $T_{10} = 279.0$ °C) but retained 16.36% mass at 800 °C, close to SBTCCT5 (16.55%). The principal derivative peak temperatures were approximately 370.6–458.2 °C across the formulations. The results demonstrate a non–monotonic effect of sorbitol concentration: thermal stability depends on the specific CCT:SBT ratio rather than increasing uniformly with sorbitol content. The comparatively high T_5/T_{10} of SBTCCT5 and SBTCCT25 and the higher high–temperature residue of SBTCCT5/SBTCCT15 identify different formulation advantages. These findings support cassava cortex valorisation as a renewable

component for bio-based material systems, while further mechanical, moisture and long-term thermal studies are required for application qualification.

Keywords: Cassava Cortex, Cassava Peel, Sorbitol, Biocomposite, Thermogravimetric Analysis, Thermal Degradation, Agro-Waste Valorisation

1. INTRODUCTION

Interest in agricultural-residue composites has grown because plant-derived fillers can reduce dependence on wholly petroleum-derived feedstocks while providing low density, renewability and local availability. Their use is nevertheless constrained by variability in composition, hydrophilicity, interfacial compatibility and thermal sensitivity. Unlike conventional mineral fillers, lignocellulosic materials are chemically heterogeneous. Cellulose contributes ordered carbohydrate domains, hemicellulose is comparatively thermally labile, and lignin degrades over a broad temperature range. Residual starch and extractives further complicate the thermal response. Consequently, the behaviour of a cassava-cortex composite cannot be inferred reliably from a single constituent or from a single characteristic temperature.

Recent work has strengthened the case for cassava-processing residues as functional material feedstocks. Weligama Thuppahige et al. (2023a) characterised cassava peel and bagasse as starch-rich fibrous agro-industrial residues and showed that their physicochemical and thermal characteristics are relevant to biodegradable material development. A companion study demonstrated that starch extracted from cassava peel and bagasse can display characteristics approaching those of commercial cassava starch, although thermal stability varies with source and processing Weligama Thuppahige et al. (2023b). More recently, Afzia and Ghosh (2026) reviewed cassava-peel-derived starch, cellulose and nanocellulose for biocomposite applications and emphasised the importance of thermal, mechanical and barrier performance when such residues are converted into useful materials. These studies position cassava peel not merely as an agricultural waste but as a heterogeneous source of polymeric and fibrous fractions that can be engineered through treatment and formulation.

Thermal behaviour is especially important because natural-fibre composites may be exposed to elevated temperatures during drying, curing, moulding, post-processing or service. Thermogravimetric analysis (TGA) provides a direct measure of mass change with temperature and is widely used to identify the onset and progression of degradation. Its derivative, DTG, is useful for locating temperatures at which the rate of mass loss is greatest and for separating degradation events that overlap in the TG curve. For heterogeneous lignocellulosic systems, the combination of TG and DTG is more informative than a single onset value because moisture release, plasticiser loss, carbohydrate decomposition and gradual lignin/char transformations may occur over partially overlapping intervals. Hiremath et al. (2024) similarly noted that the thermal response of natural-fibre polymer composites depends on fibre chemistry, matrix type, treatment, filler loading and processing history.

Chemical treatment is commonly used to modify lignocellulosic surfaces before composite preparation. Alkali treatment can remove or redistribute surface impurities and portions of non-cellulosic constituents, while acid and oxidative treatments can further modify surface chemistry. Such treatments may improve wetting or alter the relative contribution of thermally labile fractions, but they can also cause structural changes when treatment severity is high. The thermal consequences of treatment must therefore be established experimentally rather than assumed. In the present work, cassava cortex was subjected to a

defined sequence involving KOH activation, acid washing/neutralisation and additional acid/oxidative processing before formulation.

Sorbitol is a low-molecular-mass polyol containing multiple hydroxyl groups. In starch-rich and other carbohydrate-based systems, it can participate in extensive hydrogen bonding and modify molecular mobility. It is therefore frequently used as a plasticising or modifying component. Increasing sorbitol content, however, does not guarantee a proportional increase or decrease in thermal stability. A lower concentration may promote a favourable distribution of the organic phase, whereas a higher concentration can change moisture affinity, free volume, phase interactions and the relative amount of thermally labile material. The resulting TG response is consequently expected to be formulation-specific. This is important because conclusions based only on visual inspection of thermograms can obscure differences between early degradation resistance, peak degradation temperature and final residue.

A further methodological issue in thermal-composite studies is the choice of stability indicator. The temperature at 5% mass loss (T5) is sensitive to the earliest significant degradation, while T10 provides a somewhat less moisture-sensitive indication of early decomposition. The DTG maximum (Tmax) identifies the temperature of the dominant mass-loss-rate event, and residual mass at high temperature reflects the fraction that remains as char, inorganic matter or other thermally persistent material under the test atmosphere. These parameters describe different aspects of thermal behaviour and should not be treated as interchangeable. A formulation can, for example, begin to lose mass relatively early but display a later principal DTG maximum, or it can show good early stability yet leave little final residue.

Against this background, the present study evaluates the thermal behaviour of chemically treated cassava cortex/sorbitol formulations. Five materials were compared: cassava cortex (CCT100), sorbitol (SBT100), and cassava-cortex formulations containing 5, 15 and 25 wt.% sorbitol (SBTCCT5, SBTCCT15 and SBTCCT25). Quantitative TG/DTG descriptors—temperatures at 5% and 10% mass loss (T5 and T10), principal DTG peak temperature (Tmax), and residual mass at 600 and 800 °C—were used to distinguish early degradation resistance, dominant degradation behaviour and high-temperature solid retention. Unlike studies concerned primarily with cassava-residue characterisation or constituent extraction, this work provides a formulation-specific comparison of these complementary thermal indices across controlled sorbitol levels within one fixed treated-CCT preparation route. The aim was therefore to determine whether sorbitol content produces a systematic or composition-specific thermal response, without assuming that any single TGA descriptor represents overall thermal stability.

2. METHODOLOGY

2.1. Preparation and Treatment of Cassava Cortex

Cassava cortex was washed, sun-dried for approximately two weeks, oven-dried for 24 h at 50 °C, milled and sieved to a particle size of 150 µm. Chemical activation was carried out using potassium hydroxide (KOH) at a CCT:KOH mass ratio of 2:5, with stirring at 50 °C for 3 h. The treated material was oven-dried at 110 °C for 24 h and subsequently washed/neutralised with 0.5 N hydrochloric acid (HCl), followed by hot distilled water until a pH of approximately 6.5–7.0 was obtained. Other materials used during preparation included 98 wt.% sulphuric acid (H₂SO₄), 65 wt.% nitric acid (HNO₃), 30 wt.% hydrogen peroxide (H₂O₂), polyvinylidene difluoride (PVdF) at 20 wt.% as binder, N-methyl-2-pyrrolidone (NMP) as solvent and polytetrafluoroethylene (PTFE). The treated cassava cortex was finally dried at 110 °C for 24 h before formulation. The treatment route was kept constant for all composite formulations so that sorbitol content remained the principal formulation variable examined in the thermal analysis.



Figure 1: Cassava cortex preparation used in this study: (a) dried cassava cortex and (b) 150 µm sieved milled cassava cortex.

The drying and particle-size-control steps were used to minimise variation associated with free moisture and coarse particles before treatment, and the 150 µm fraction was used consistently in subsequent preparation. Following KOH treatment, the material was washed to near-neutral pH. Because the same treatment route was applied to the CCT used for all composite formulations, the individual effects of the treatment reagents were not investigated separately in this study.

For interpretation of the thermal data, the treatment route is treated as a fixed preparation condition. All sorbitol-containing formulations were derived from the same treated-CCT route, while sorbitol level was the principal composition variable examined. The experiment was not designed as a factorial comparison of KOH, acid, oxidative, binder or solvent effects; consequently, the thermal shifts reported below cannot be attributed independently to any one of these materials.

2.2. Composite Formulation

Treated CCT and sorbitol were combined in the nominal proportions shown in Table 1. The 5, 15 and 25 wt.% designations describe the sorbitol:CCT formulation basis used for the experimental samples. PVdF was used at 20 wt.% as binder, while NMP and PTFE were included during preparation. Methyl ethyl ketone peroxide (MEKP) and cobalt naphthenate were used as the catalyst system during fabrication. The constituents were thoroughly mixed, introduced into the mould by hand lay-up and allowed to cure before thermal characterisation.

Table 1: Nominal CCT:sorbitol formulations used for sample nomenclature. Note: the listed percentages describe the documented CCT:SBT basis and are not presented as a complete mass balance of all processing aids used during preparation.

Sample	Sorbitol (g)	CCT (g)	Nominal composition
SBT100	100	0	100 wt.% SBT
SBTCCT5	5	95	5 wt.% SBT / 95 wt.% CCT
SBTCCT15	15	85	15 wt.% SBT / 85 wt.% CCT
SBTCCT25	25	75	25 wt.% SBT / 75 wt.% CCT
CCT100	0	100	100 wt.% CCT

The formulation nomenclature was based on the relative CCT and sorbitol contents used in the study. CCT100 represents the cassava-cortex reference, SBT100 the sorbitol reference, and SBTCT5, SBTCT15 and SBTCT25 the formulations containing 5, 15 and 25 wt.% sorbitol, respectively, with the corresponding CCT proportions shown in Table 1. These composition levels provided low, intermediate and higher sorbitol contents for comparison of their thermal responses.

MEKP and cobalt naphthenate were used during fabrication as the catalyst system. The present study focused on the effect of sorbitol content on the thermal response of the prepared materials; consequently, the curing chemistry and the individual effects of the processing aids were not investigated as separate experimental factors.

2.3. Thermogravimetric Analysis

TGA was performed using a NETZSCH STA 449C. A sample mass of 8.0 ± 1.5 mg was heated from 20 to 900 °C at 10 °C min⁻¹ under a 50 mL min⁻¹ nitrogen purge. Three measurements were reported for each sample, and the available TG series were analysed on a common basis. Mass was normalised to the initial recorded mass. T5 and T10 were defined as the temperatures at 5% and 10% mass loss, respectively. The principal DTG peak temperature (T_{max}) was taken as the temperature of the most negative derivative-mass signal between 150 and 650 °C. Residual mass was evaluated at 600 and 800 °C.

Nitrogen was used to provide an inert purge during heating. This is important for interpretation because the high-temperature residue under nitrogen represents material that remains after pyrolytic degradation and carbonisation rather than residue produced under an oxidative combustion environment. Consequently, the residual-mass values reported here should not be interpreted as ash content unless a separate oxidative ash determination is performed.

The TG data for the five formulations were evaluated on a common temperature basis to permit direct comparison. Mass values were normalised relative to the initial value for each series. T5 and T10 were determined at 95% and 90% remaining mass, respectively. DTG curves were obtained from the temperature-dependent rate of mass loss, and the principal T_{max} was assigned to the dominant mass-loss-rate peak. Residual mass was determined at 600 and 800 °C to provide comparable high-temperature endpoints.

T5 and T10 characterise resistance to early measurable mass loss, T_{max} identifies the dominant mass-loss-rate event, and high-temperature residue describes the fraction remaining after extensive heating under nitrogen. These descriptors were interpreted separately rather than combined into a single ranking. The available data did not permit calculation of replicate standard deviations; consequently, dispersion values are not reported. Future studies should retain individual instrument runs to allow direct reporting of mean values and associated uncertainty.

2.4. Use of Generative AI in Manuscript Preparation

Generative AI tools were used only to assist with language editing, clarity and manuscript formatting. The authors reviewed and approved the final text and remain fully responsible for the experimental data, analysis, interpretation and conclusions. No generative AI tool was used to generate, alter or fabricate the experimental results reported in this study.

3. RESULTS AND DISCUSSION

3.1. Combined TGA Behaviour

The TGA curves (Figure 2) show multistage thermal degradation and clear differences among the five formulations. The early mass-loss region is attributable mainly to moisture and low-boiling volatiles,

whereas the broader high-temperature loss reflects overlapping degradation of the carbohydrate/lignocellulosic fractions and the sorbitol-containing phase. Cassava peel is compositionally heterogeneous, containing starch, cellulose, hemicellulose and lignin; accordingly, a broad degradation envelope is more plausible than a single decomposition event Afzia and Ghosh (2026).

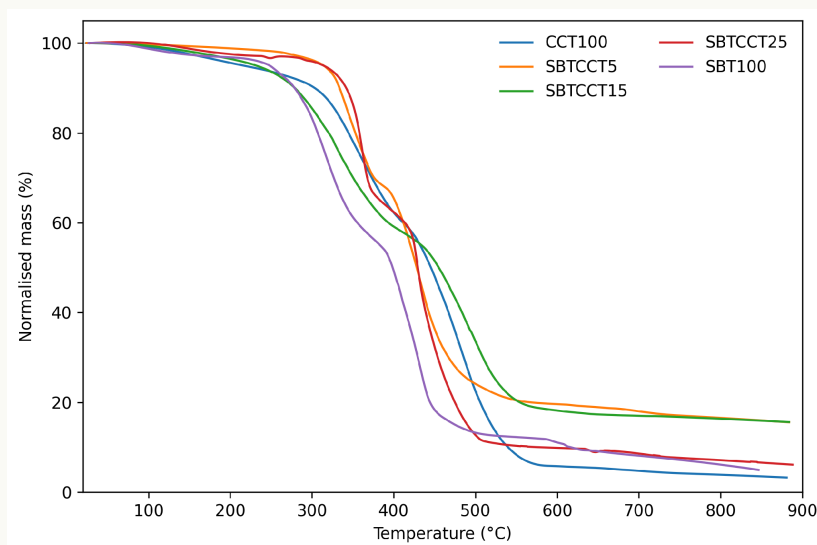


Figure 2: Thermogravimetric curves of CCT100, SBT100 and SBTCCT formulations.

The low-temperature region is interpreted cautiously because physically retained water and low-boiling species can contribute to initial mass loss, with the magnitude depending on drying history, hydrophilicity and storage. At higher temperatures, overlapping degradation of starch, cellulose, hemicellulose and lignin produces the broader mass-loss envelope expected for heterogeneous cassava-derived material Weligama Thuppahige et al. (2023a).

Normalising all formulations on a common temperature basis makes the composition effect clearer: the curves separate in the early significant-degradation region and change relative order again at high temperature. This behaviour indicates that sorbitol level affects more than one part of the degradation response and supports evaluation using several complementary indices rather than visual inspection of a single thermogram.

3.2. Quantitative Thermal-Stability Indices

Table 2 provides the central quantitative comparison. The results revise the earlier qualitative interpretation that increasing sorbitol necessarily produces a progressive increase in thermal stability. Instead, the response is non-monotonic. SBTCCT5 and SBTCCT25 have nearly identical T5 values (314.4 and 314.1 °C), both markedly higher than CCT100 (215.9 °C), SBT100 (248.2 °C) and SBTCCT15 (231.7 °C). At 10% mass loss, SBTCCT25 records the highest threshold (342.0 °C), followed by SBTCCT5 (333.8 °C). Thus, for early-stage mass-loss resistance, the 5 and 25 wt.% sorbitol formulations perform more favourably than the 15 wt.% formulation in the experimental dataset.

Table 2: Thermal degradation indices of CCT100, SBT100 and SBTCCT formulations.

Sample	T5 (°C)	T10 (°C)	DTG Tmax (°C)	Residue at 600 °C (%)	Residue at 800 °C (%)
CCT100	215.9	303.4	439.6	5.81	3.90
SBT100	248.2	278.8	370.6	11.08	6.14
SBTCCT5	314.4	333.8	398.8	19.60	16.55

Sample	T5 (°C)	T10 (°C)	DTG Tmax (°C)	Residue at 600 °C (%)	Residue at 800 °C (%)
SBTCCT15	231.7	279.0	458.2	18.18	16.36
SBTCCT25	314.1	342.0	408.4	9.87	7.14

High-temperature residue gives a different pattern from T5/T10. At 800 °C, SBTCCT5 and SBTCCT15 retained 16.55% and 16.36%, respectively, compared with 7.14% for SBTCCT25, 6.14% for SBT100 and 3.90% for CCT100. Thus, the formulation with the highest T10 did not produce the highest final residue. Under nitrogen, the residue can include carbonaceous char and inorganic fractions; its magnitude should therefore not be interpreted as ash content or as proof of overall thermal superiority without complementary compositional analysis.

The early-degradation thresholds provide the clearest evidence of a non-monotonic sorbitol effect. SBTCCT5 and SBTCCT25 reached T5 at approximately 314 °C, nearly 100 °C above CCT100 (215.9 °C), while SBTCCT15 reached T5 at 231.7 °C. At T10, SBTCCT25 remained highest at 342.0 °C, followed by SBTCCT5 at 333.8 °C. The intermediate composition therefore cannot be treated as evidence of a linear concentration-response relationship.

Taken together, the threshold and residue data distinguish two performance attributes: SBTCCT5 and SBTCCT25 show stronger resistance to early measurable mass loss, whereas SBTCCT5 and SBTCCT15 retain more solid material at 800 °C. Selection of a formulation must therefore be tied to the intended thermal-performance criterion.

3.3. Derivative Thermogravimetric Behaviour

The derivative curves (Figure 3) clarify the temperatures of maximum mass-loss rate. The principal DTG peaks occur at approximately 439.6 °C for CCT100, 370.6 °C for SBT100, 398.8 °C for SBTCCT5, 458.2 °C for SBTCCT15 and 408.4 °C for SBTCCT25. The high Tmax of SBTCCT15 contrasts with its relatively low T5/T10 values, demonstrating why thermal stability should not be represented by a single metric. SBTCCT15 begins losing mass earlier but its dominant high-temperature degradation event occurs later. Conversely, SBTCCT5 and SBTCCT25 resist the initial 5–10% mass loss more effectively but show principal derivative peaks at lower temperatures than SBTCCT15.

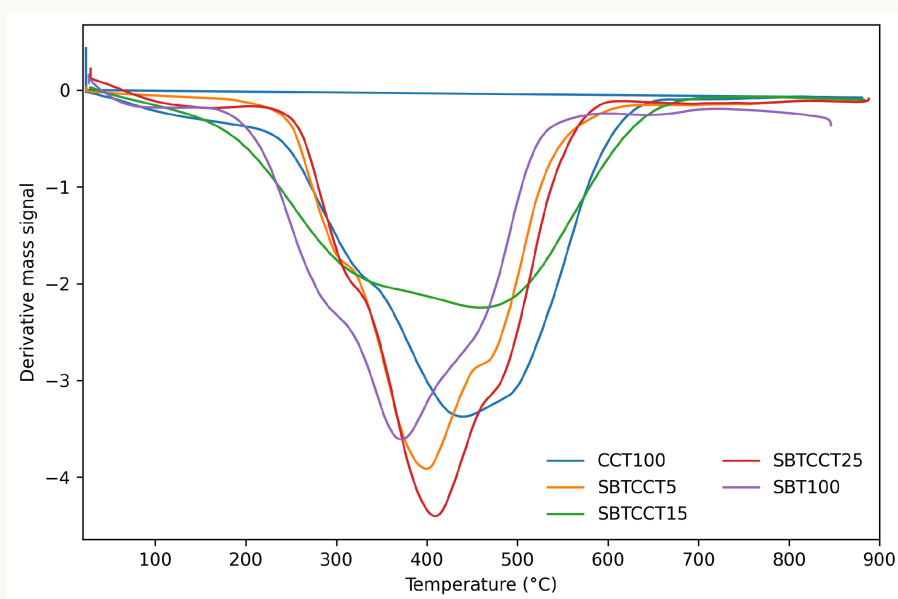


Figure 3: Derivative thermogravimetric curves showing the principal mass-loss-rate events of the five formulations.

DTG adds information that T5 and T10 cannot provide. The principal T_{max} values range from 370.6 °C for SBT100 to 458.2 °C for SBTCT15. In particular, SBTCT15 reaches the early mass-loss thresholds relatively soon but displays the latest dominant mass-loss-rate event, behaviour consistent with a multistage system containing fractions of different thermal resistance.

CCT100 shows a principal T_{max} of 439.6 °C, while the sorbitol-containing formulations shift the dominant event to different temperatures. Such shifts may reflect changes in constituent proportions, interactions, dispersion and overlapping degradation events, but the present TGA data cannot assign individual peaks uniquely to cellulose, lignin, sorbitol or another constituent. Complementary evolved-gas or component-resolved analysis would be required for that attribution.

3.4. Effect of Sorbitol Content and Comparison with Current Literature

The quantitative results establish a formulation-specific rather than monotonic sorbitol effect. This is consistent with the broader natural-fibre-composite literature, in which thermal behaviour depends on constituent chemistry, treatment, filler content, matrix interactions and processing history Hiremath et al. (2024). In the present system, sorbitol may influence hydrogen bonding, moisture association and molecular mobility, but these mechanisms were not measured directly and are therefore treated as plausible explanations rather than demonstrated pathways.

The measured degradation range is also compatible with recent cassava-residue studies. Samimi et al. (2026) reported a broad principal degradation region of approximately 200–400 °C for untreated cassava peel, while Weligama Thuppahige et al. (2023a), Weligama Thuppahige et al. (2023b) showed that cassava peel, bagasse and extracted starch display source- and processing-dependent thermal behaviour. These materials and treatment routes differ from the present formulations, so their numerical values are used for context rather than direct equivalence.

Recent formulation studies further support the importance of composition. Marengo-Orozco and Tadini (2026) showed that cassava-processing by-product ratios altered extruded biocomposite properties, while Ogbeh et al. (2026) linked the behaviour of cassava-peel/sugarcane-bagasse films to composition and interfacial interactions. The contribution of the present study is narrower but complementary: it separates early mass-loss resistance, dominant degradation temperature and high-temperature residue across defined sorbitol levels within one treated-CCT route.

3.5. Engineering Implications and Limitations

The engineering implication is that thermal-performance criteria should not be collapsed into a single 'best formulation'. SBTCT5 and SBTCT25 provide the strongest early mass-loss resistance in this dataset, whereas SBTCT5 and SBTCT15 retain the greatest solid fraction at 800 °C. Which descriptor matters most depends on the intended processing or service requirement.

TGA nevertheless measures mass change under a specified heating programme and atmosphere; it does not establish softening, glass transition, heat-deflection temperature, dimensional stability, creep, mechanical-property retention or continuous-use temperature. Application qualification therefore requires complementary DSC/DMA, mechanical testing, moisture conditioning and controlled thermal-ageing studies.

A limitation of the study is that replicate variability could not be evaluated from the available thermal dataset. Although three measurements were reported for each sample, individual replicate values were not available for separate statistical analysis. Future studies should retain the individual run files and report mean ± standard deviation for T5, T10, T_{max} and residual mass.

The study varied sorbitol content within one established treated-CCT preparation route and therefore did not isolate the individual effects of KOH, H₂SO₄, HNO₃, H₂O₂, PVdF, NMP, PTFE, MEKP or cobalt naphthenate. These materials formed part of the preparation procedure, while sorbitol content was the principal formulation variable considered in the thermal comparison.

Despite these limitations, the dataset provides a useful quantitative baseline for cassava-cortex formulation development. The combination of defined sorbitol levels and multiple TG/DTG descriptors demonstrates that the thermal response is composition-specific and identifies the thermal characteristics that should be considered in subsequent formulation studies.

4. CONCLUSION

This study evaluated the thermal degradation of chemically treated cassava cortex/sorbitol formulations using TG and DTG analysis. Sorbitol content altered thermal behaviour substantially but non-monotonically. SBTCT5 and SBTCT25 showed the greatest resistance to early mass loss, with T₅ values of 314.4 and 314.1 °C, respectively, while SBTCT25 produced the highest T₁₀ at 342.0 °C.

The principal DTG peaks and high-temperature residues produced different patterns. SBTCT15 displayed the highest T_{max} (458.2 °C) despite lower early mass-loss thresholds, whereas SBTCT5 and SBTCT15 retained 16.55% and 16.36% mass at 800 °C. Early degradation resistance, dominant degradation temperature and residual solid yield therefore represent distinct aspects of thermal behaviour.

Accordingly, no formulation should be described as universally superior without defining the performance criterion. SBTCT5 combines delayed early mass loss with relatively high final residue, while SBTCT25 gives the highest T₁₀. The study supports composition-specific optimisation of cassava-cortex materials and demonstrates the value of reporting multiple TG/DTG indices rather than relying on a single qualitative thermogram assessment.

TGA alone does not establish continuous service temperature or mechanical suitability. Further work should include full replicate analysis and combine the thermal indices with mechanical performance, moisture uptake, DSC/DMA, microstructural analysis and controlled thermal ageing. Within these limits, the present study provides a useful thermal baseline for continued development of cassava-cortex-based composite materials.

Data Availability

The data supporting the reported T₅, T₁₀, T_{max} and residual-mass values are available from the corresponding author on reasonable request.

Declarations

- Funding: No external funding was reported for the underlying experimental study.
- Conflict of interest: The authors declare no conflict of interest.
- Ethics approval: Not applicable. The study did not involve human participants or experimental animals.

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