Role of Silica Aerogel Concentration in Improving the Thermal Behavior of Sugar Palm Fiber Reinforced Polyester Composites

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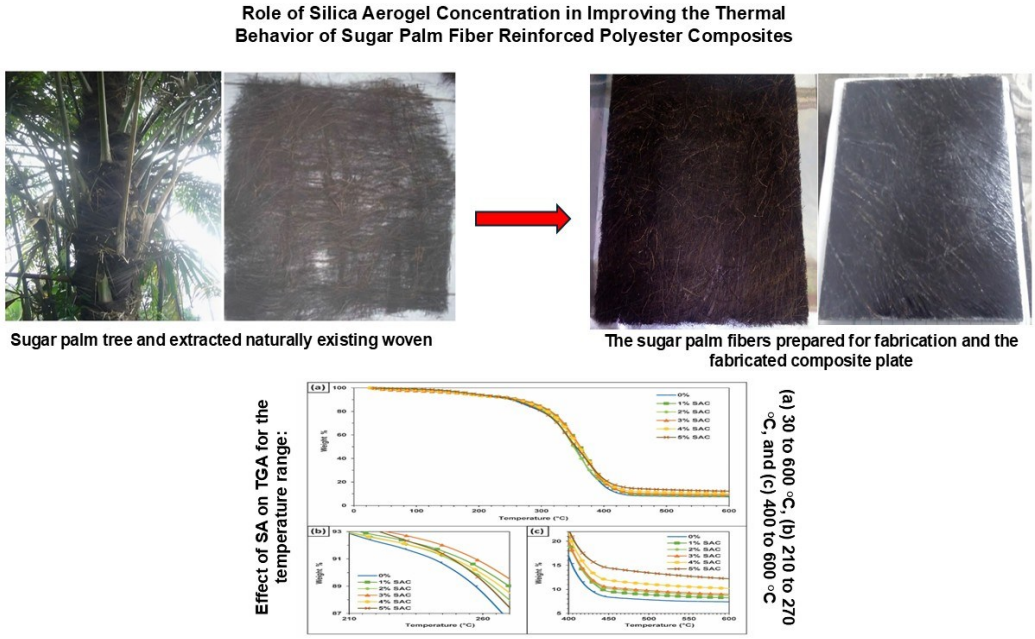
GRAPHICAL ABSTRACT



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Owing to their low cost, availability, and biodegradability, natural fibers can be suitable alternatives to synthetic fibers. Replacing traditional synthetic fiber-reinforced polymer composites with natural fiber-reinforced composites can substantially reduce environmental impact by lowering the reliance on non-renewable reinforcements, while still using industrially feasible polymer matrices such as unsaturated polyester. The present study utilised biodegradable reinforcements, sugar palm fiber (SPF) in unsaturated polyester (UPE), and silica aerogel (SA) as a filler material. A range of SA concentrations was tested, and an optimal concentration was identified to achieve the best thermal performance of SPF/UPE composites. The thermal properties were examined by dynamic mechanical analysis (DMA), whereas thermogravimetric analysis (TGA) was employed to assess thermal degradation of the samples. DMA results revealed that the best mechanical performance under thermal conditioning was achieved with 3 wt. % SA content. Similarly, the TGA study indicated that the onset temperature of thermal decay was highest in the 3 wt. % SA composites. Under extreme heat exposure, the composite with the highest SA content (5 wt. %) experienced the least decomposition. Across all samples, thermal performance was generally significantly improved compared to the control sample.

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Keywords: Sugar palm fibers; Polyester; Silica aerogel; Dynamic mechanical analysis; Thermogravimetric analysis

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INTRODUCTION

The increase in global population has led to a rise in demand for products made from wood; however, the shortage of wood in recent years has made it challenging to meet this demand (Qiu *et al*. 2021). Consequently, researchers have been pushed to search for alternative solutions to meet these needs in various fields such as transportation, construction, and furniture (Mukhtar *et al*. 2016). With the increasing demand for wood-based products, polymer composites with natural fiber fillers can be used as a suitable alternative to meet the needs. Natural fibers offer numerous benefits, such as abundance, economic, non-hazardous, lower energy utilization for production, and competitive

mechanical properties (Asyraf *et al*. 2021). These properties are favorable for producing a natural fiber-based polymer composite with superior characteristics compared to pure polymers. Despite its advantages, there are some drawbacks to using natural fibers as well, such as their hydrophilic nature and poor fiber-matrix bonding (Khan *et al*. 2021; Muhammed Shameem *et al*. 2021). Weaker thermal characteristics are also one of the significant reasons that may obstruct the mass utilization of natural fibers (Nasir *et al.* 2022). In general, natural fibers can sustain their structural integrity when exposed to temperatures below 200 °C, and any further rise in temperature results in degradation and shrinkage of the fibers. Elevated temperatures may compromise the chemical or physical characteristics of fiber (Ilyas *et al*. 2020).

Most natural, plant-based fibers, including sugar palm fibers (SPF), are known to be lignocellulosic materials in which the lignin acts as the binder for cellulose and hemicellulose (Neto *et al*. 2021; Renjith and Nair 2023). Table 1 shows that the approximate cellulose content in SPF ranges from 40.5% to 66.5%. The constituents of SPF vary greatly based on several factors, including plantation time, plant maturity, plant component, growing atmosphere, soil characteristics, and plant height (Bachtiar *et al*. 2025). A previous investigation found that the cellulose concentration in SPF commonly depended on the part of the plant from which the fiber was extracted. It was found that cellulose content was highest in SPF from the frond, followed by the bunch, and finally, trunk fibers showed the least cellulose content (Chamola *et al*. 2024a,b). Bachtiar *et al.* (2025) also discovered that various properties, such as thermal and mechanical, followed the same pattern as the cellulose content of these fibers. A few studies on the hybridization of bamboo and banana fibers with glass fiber reinforcement in polypropylene (PP) revealed that lignin enhances thermal stability and increases char residue content, thereby improving the fire-retardant characteristics of materials (Hiremath *et al*. 2024; Ornaghi *et al*. 2024).

The chemical composition values shown in Table 1 are reported as controlled weight percentages on a dry basis to confirm that the sum of the main elements equals 100% for each fiber type.

Table 1. SPF Extracted from Different Plant Parts and their Corresponding Chemical Composition (Bachtiar *et al*. 2025)

Fiber Type	Cellulose (%)	Hemicellulose (%)	Lignin (%)
SPF Frond	66.5	12.0	18.90
SPF Bunch	61.80	12.20	23.50
SPF Fiber	52.30	11.70	31.50
SPF Trunk	40.60	11.5	46.4

SPF reinforced polymer composites have been the subject of numerous studies in the past. A detailed compilation of research studies on SPF and its composites was reviewed by Asmare *et al*. (2024), who considered advances in sugar palm fiber-reinforced composites. Other authors have focused on SPF reinforcement in thermoset polymers (Safri *et al*. 2018; Alsubari *et al*. 2021). SPF has been studies as a filler in various polymeric matrices, such as epoxy, unsaturated polyester, polyester resin, phenol formaldehyde, and high-impact polystyrene (Alshammari *et al*. 2019; Sherwani *et al*. 2022). Environmentally friendly composites were also fabricated using fibers and starch extracted from sugar palm trees (Rangappa *et al*. 2022). Although there have been several studies on improving SPF-reinforced composite properties, the potential of fillers (particularly silica aerogel) to achieve better or similar enhancements remains unexplored (Olanrewaju *et al*. 2025).

Silica aerogel (SA) can be considered an ultimate filler because of its unique porous structure and other exceptional benefits, such as high thermal resistance, high specific surface area, lower density, and high levels of porosity (Firoozi *et al*. 2025). The distinctive nanoporous molecular construct of SA makes it highly suitable for several applications that involve thermal insulation, *i.e.*, thermal blankets (Mazrouei-Sebdani *et al*. 2022), lightweight protective clothing (Mirzaei *et al*. 2023), and natural rubber latex-silica aerogel films (Azam *et al*. 2022). An aerogel/epoxy composite was exposed to a wide range of temperatures, and the researchers found low thermal conductivity (0.11 to 0.044 W/m K) of the composite due to the addition of aerogel in epoxy (Ge *et al.* 2009). In another study, the addition of nanosilica in polyurethane amplified the rubbery modulus and extended it to higher temperatures, making it more suitable for fabric coating (Chen *et al*. 2025).

In this study, SA with the trade name of Maerogel is employed as a filler material. This aerogel is produced from rice husk, which is usually wasted during the milling of rice. This makes the Maerogel both economical and environmentally friendly. It is reported in an earlier study that this aerogel consists of features that are comparable to the commercially available alternative tetraethoxysilane (Maghsoudi and Motahari 2018). Despite the visible potential of Maerogel, its usability in natural fiber composites as a filler is still mainly unexplored. More specifically, no literature was found on the infusion of silica aerogel on the thermal properties of SPF- reinforced unsaturated polyester (UPE) composites (Vahtrus *et al*. 2017).

The present work focuses on the thermal characterization of various weight percentages of maerogel in SPF/UPE composite. The thermal studies are conducted *via* thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA) of all the fabricated configurations. This new introduction of composites is assumed to improve thermal stability because of the presence of silica aerogels. With improved thermal stability, the usability of the SA-modified SPF composite may extend to a broader range of applications.

MATERIALS AND METHODS

Trunk Fiber Preparation

The SPF fibers used in this study were obtained from the sugar palm plantation in Kampung Kuala Jempol village, located in Malaysia, as depicted in Fig. 1. These fibers can be extracted from various parts of the plant; however, to ensure consistent properties, only trunk fibers are used to fabricate all the composites in this study.

Composite Fabrication

Commercially available unsaturated polyester (Resol P 9509) and methyl ethyl ketone peroxide (MEKP) were employed as a binder material and catalyst for curing polyester. Rice husk-based silica aerogel (Maerogel) was procured from Maerotech Sdn Bhd, Malaysia, and used without any further chemical modification. The properties of SA used are as follows: an approximate specific surface area of 900 m^2/g, melting and boiling temperatures ranging from 1700 to 2230 °C, and nano-dimensions of marogel particles ranging from 20 nm to 50 nm.

The fibers were prepared by carefully cleaning them with fresh running water to remove any additional contaminants, followed by drying. The fibers were stacked to achieve a weight fraction of approximately 30% of the desired composite plate. These

fibers were then arranged evenly in the mold. Furthermore, SA powder was mixed in unsaturated polyester using a mechanical stirrer for about an hour at a speed of 500 rpm. The weight percentage of the SA powder varied from 1% to 5% to obtain different compositions of the composite. Upon settling, 1% MEKP was added as a hardener to the prepared resin and mixed as well. Eventually, the resin was gradually poured into the mold with fibers and evenly spread using a steel roller, using the hand layup process. A hot press machine was used to press the mold containing all the materials at 80 °C for 30 min; upon removal, it was left at room temperature for at least 24 h for post-curing.

Fig. 1. Sugar palm tree and extracted naturally existing woven fibers

Performance Evaluation

The natural fiber composites were subjected to dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) to assess their thermal properties and viscoelastic behavior. All specimens were prepared under controlled environmental conditions prior to testing, and the analyses were conducted in accordance with appropriate ASTM standards.

At least five composite plates of each configuration were prepared to get better results. The fabricated composite sheets are displayed in Fig. 2. All configurations of a composite plate fabricated in this study are listed in Table 2. The table displays the wt. % of UPE and SA in SPF/UPE composites. Where SAC stands for SA infused in SPF/UPE composites.

Table 2. Weight Percentages of the Components of All the Composite Plate Configurations

S.N.	Configuration	SPF (wt. %)	UPE (wt.%)	SA (wt.%)
1	0% SAC	30.00	70.00	0.00
2	1% SAC	30.00	69.00	1.00
3	2% SAC	30.00	68.00	2.00
4	3% SAC	30.00	67.00	3.00
5	4% SAC	30.00	66.00	4.00
6	5% SAC	30.00	65.00	5.00

Fig. 2. The sugar palm fibers prepared for fabrication and the fabricated composite plate

Dynamic Mechanical Analysis (DMA)

A TA Instruments DMA Q800 (USA) was used to perform dynamic mechanical analysis (DMA) in dual cantilever bending mode. Rectangular specimens, roughly 50×10×3 mm, were cut from the composite plates. The tests were conducted at a fixed frequency of 1 Hz and a continuous strain within the linear viscoelastic region. The temperature increased from 30 °C to 175 °C at a heating rate of 5 °C/min under ambient air conditioning. At least three samples were tested for each composite configuration, and the average values were reported to confirm repeatability.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was performed using a TGA Q500 instrument from TA Instruments, USA. Approximately 8 to 12 mg of each composite sample was placed in a platinum pan and heated from 30 to 600 °C at a constant rate of 20 °C/min under a nitrogen atmosphere at a flow rate of 60 mL/min. At least three replicates were performed for each composite formulation, and the resulting curves were used for analysis.

RESULTS AND DISCUSSION

Dynamic Mechanical Analyzer

To investigate the viscoelastic characteristics of a material while being exposed to a defined range of temperatures, the DMA method is employed. The interfacial grip between the matrix and fibers can be investigated by the storage modulus (E'). The loss modulus (E") represents the viscous response of the composite with respect to temperature, and the damping property (tan δ) of the composites can be determined by dividing the E" by the E'.

Storage Modulus

The storage modulus (*E*') of composites with various percentages of silica aerogel reinforced SPF/UPE composites is shown in Fig. 3. The improvement in storage modulus (*E')* with the addition of silica aerogel can be attributed to enhanced stress-transfer efficiency and controlled molecular mobility within the polymer matrix, which is consistent with previous findings on natural fiber-reinforced polymer composites containing particulate and nanoscale fillers (Costa *et al.* 2016; Shejkar *et al.* 2023).

The highest *E*' was recorded at the initial temperature of the analysis for all configurations and exhibited a similar degradation trend as the temperature increased. The plots can be categorized into three sections, namely glassy (before the sharp decrease of *E*'), glass transition (during the sharp decrease), and rubbery (after the sharp decrease) regions. Similar increases in storage modulus, attributed to filler-induced trapping of polymer chains, have been reported in silica aerogel and silica-filled thermoset composites, where increased viscoelastic behavior was observed over a broad temperature range. (Ge *et al.* 2009; Maghsoudi and Motahari 2018). Temperature points representing these regions and their corresponding *E*' values are tabulated in Table 3.

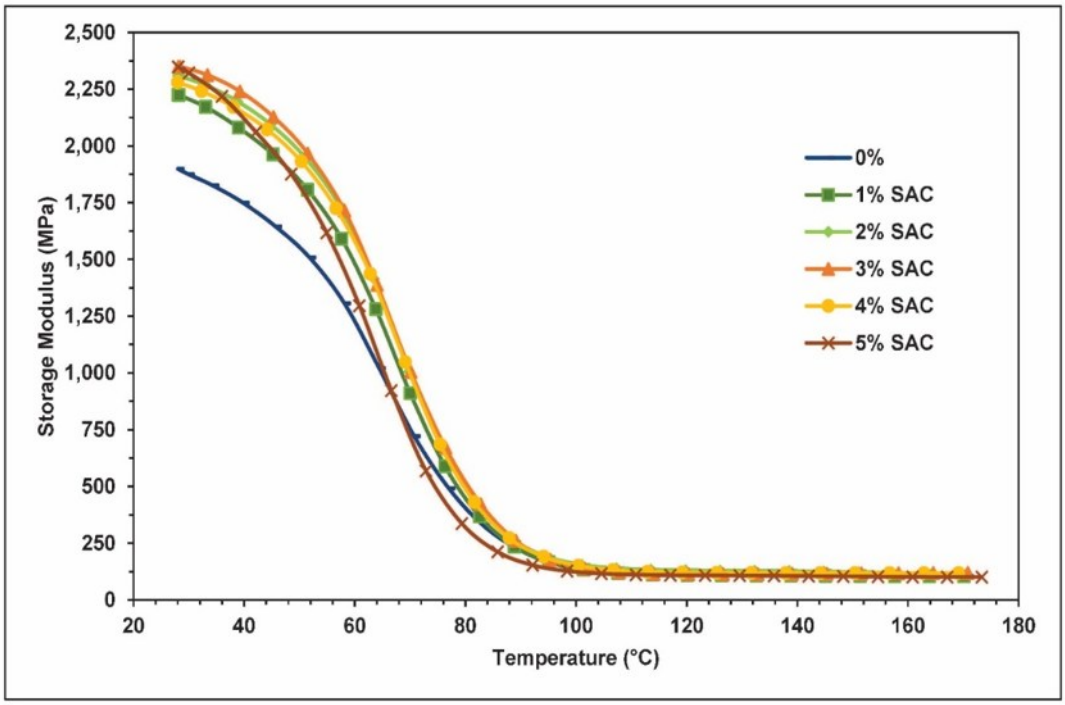

Fig. 3. Effect of SA concentration on storage modulus of SPF/UPE composite

It is evident from Fig. 3 that the composites with SA exhibited an improvement in the storage modulus. A higher *E*' indicates better stress-transferring characteristics, governed by better interfacial adhesion between the matrix and the reinforcements. The SAC level at 3% demonstrated the highest *E*' at 30 °C, nearly 24% higher than the 0% composite, and retained a superior *E*' throughout the exposed temperature range of the analysis. Molecular mobility and reinforcing effects are governing factors influencing the properties of composites at higher temperatures, as confirmed by previous studies (Shejkar *et al*. 2023). All the variants of SAC exhibited a similar deterioration in *E*' as the temperature increased, except for 5% SAC, which showed the highest drop in *E*' during the glass transition region.

Reduced storage modulus at higher filler loadings is common in particulate-filled polymer composites, often due to filler agglomeration, stress concentrations, and reduced matrix–filler interaction efficiency (Matykiewicz 2020; Shejkar *et al.* 2023). This unusual behavior can be attributed to the development of aggregates due to a higher loading of SA, which fails to maintain its packed structure with the polymer. Consequently, increased molecular mobility occurred in this temperature region, resulting in a sharper drop in *E*' exhibited by 5% SAC.

Furthermore, for all composites, a significant decrease in *E*' can be observed from 50 to 80 °C, which signifies the deterioration of the molecular structure of the composite. The *E*' for almost all the SAC showed higher values through the entire temperature range compared to the composite without SA. The test results confirmed that infusion of SA in SPF/UPE improved interfacial adhesion with UPE and limited the molecular mobility, regardless of the magnitude of heat exposure.

While the silica aerogel content affects various thermal indicators, the 3 wt. % SA composite shows the most stable improvement in viscoelastic performance, whereas higher loadings primarily improve specific thermal stability metrics such as T_g and residual mass.

Loss Modulus

The change in loss modulus (*E*") with temperature for the SPF/UPE fiber composites is shown in Fig. 4. All silica aerogel-filled composites demonstrated superior *E*" values compared with the control specimens, with the highest increase reported at 3 wt. % SA. This behavior indicates increased energy dissipation within the composite, which may be attributed to restricted molecular mobility and increased interaction between the filler and the polymer matrix. The plot shows an increase in the value of *E*" with the addition of SA wt. % in SPF/UPE composites. All the composites with SA exhibited higher values of *E*" up to the peak *E*" compared to the 0% SA sample. The increase in peak *E*" was recorded up to 3 wt. % of SA, approximately 40% higher compared to the 0% SAC sample. The higher value of *E*" is attributed to enhanced interfacial adhesion between fillers and binders. A high value restricts the mobility of the polymer chain.

At higher SA content (5 wt. %), a decrease and a shift of the *E″* peak to lower temperatures were observed. This behavior may indicate altered segmental dynamics, potentially due to non-uniform filler dispersion at higher loading levels. These interpretations are inferred from thermomechanical trends, and direct microstructural analysis would be required for definitive confirmation.

The disordered SA particles within the polymer impede the molecular mobility, resulting in higher frictional losses in the SPF/UPE composite and consequently, a higher *E*". This reveals that a higher level of energy dissipation and structural mobility will occur in SA filled composites relative to 0% composites. Similar to *E*', 5% SAC showcased a sharper drop in *E*" in the transition region. The peak value of 5% SAC was also observed at a comparatively lower temperature, suggesting that high SA content may lead to higher rates of intermolecular oscillation at lower temperatures. Additionally, the addition of SA to SPF/UPE composites increased the T_g (Table 3). This increment may result from additives acting as inhibitors in the segmental motion of polymer chains formed between the additive and the matrix. The increased T_g indicates better thermal stability of the composites, attributed to the addition of SA. Table 3 displays the highest achieved *E*" values and T_g for all the composites.

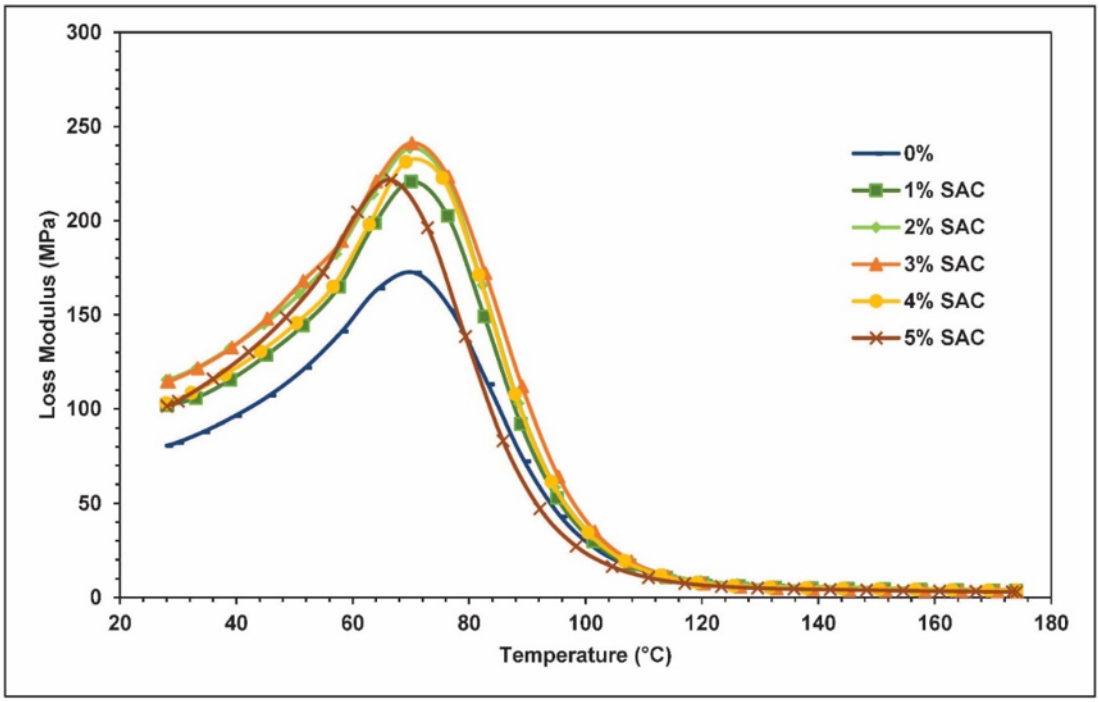

Fig. 4. Effect of SA concentration on loss modulus of SPF/UPE composite

Damping Factor

The outcome of the study revealed that the damping factor (tan δ) increased with the rise in temperature until the peak magnitude was achieved, as depicted in Fig. 5. A further rise in temperature exhibited almost a symmetrical reduction in tan δ magnitude, and the trends were common for all the composites. The peak values of tan δ for the composites with and without SA additives are tabulated in Table 3.

Figure 5 shows that the addition of SA in SPF/UPE composites resulted in higher tan δ peaks compared to 0% composite. A loading of 3% SA resulted in the maximum tan δ value compared to other variants of SA-filled composites. It has been suggested that higher tan δ can be attributed to higher energy at the interface due to greater interfacial area (Costa *et al*. 2016). An increase in tan δ was noticed, while E' and E" were also seen to increase. However, the comparative trends of increments in E' and E" demonstrated that the rate of energy dissipation within the composite increased to a greater degree, while the improvement in stiffness or interfacial adhesion was less pronounced. Therefore, it can be concluded that a composite can have good load-bearing capacity and still exhibit a higher tan δ value, provided that heat dissipation occurs at a higher rate. This can be confirmed with the data from Table 3, where SA-based composites showed a greater increase in E" and a smaller increase in E'.

As discussed in the earlier section, the higher rate of heat dissipation was due to the presence of additional phases in the composites with SA compared to the 0% composite. In contrast, higher load-bearing capacity was the result of improved bonding characteristics. The peak shift of 5% SAC towards lower temperatures is observable in Fig. 5. In comparison, all other SAC composites exhibited peak points at higher temperatures compared to the 0% composite, indicating improved thermal stability in composites with filler concentrations below 5%.

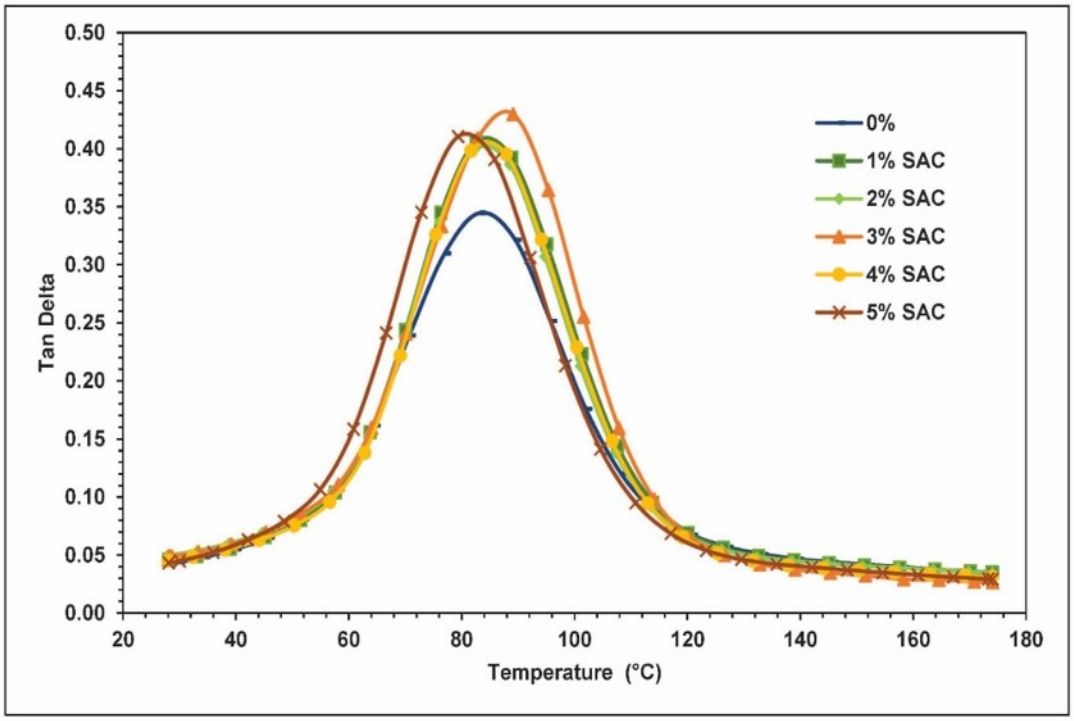

Fig. 5. Effect of SA concentration on tan δ of SPF/UPE composite

Table 3. Variation in DMA Dynamic Parameters of Composites at Different Temperatures

Configurations	*E*' (MPa)			*E*'' (MPa)	T_g (°C)	tan δ
	30 °C	60 °C	90 °C			
0%	1,874	1,232	219	173	69.22	0.345
1% SAC	2,209	1,480	217	221	70.79	0.410
2% SAC	2,295	1,612	243	239	70.54	0.404
3% SAC	2,339	1,623	247	241	70.60	0.433
4% SAC	2,264	1,587	240	234	71.16	0.408
5% SAC	2,322	1,350	169	222	66.60	0.414

Thermogravimetric Analysis

TGA was conducted from room temperature to 600 °C for all samples as shown in Fig. 6. It is clear from the TGA curve that gradual decomposition was observed up to nearly 200 °C, and rapid decomposition began between 220 and 300 °C. Most of the constituents of all the composites decomposed by 440 °C, and the curve became almost parallel to the X-axis, marking the end of primary decomposition. The significant decomposition of mass for a temperature range of 220 to 440 °C for all the composites is denoted by D_0, whereas T_0 represents the extrapolated onset temperatures from the plots in Fig. 6.

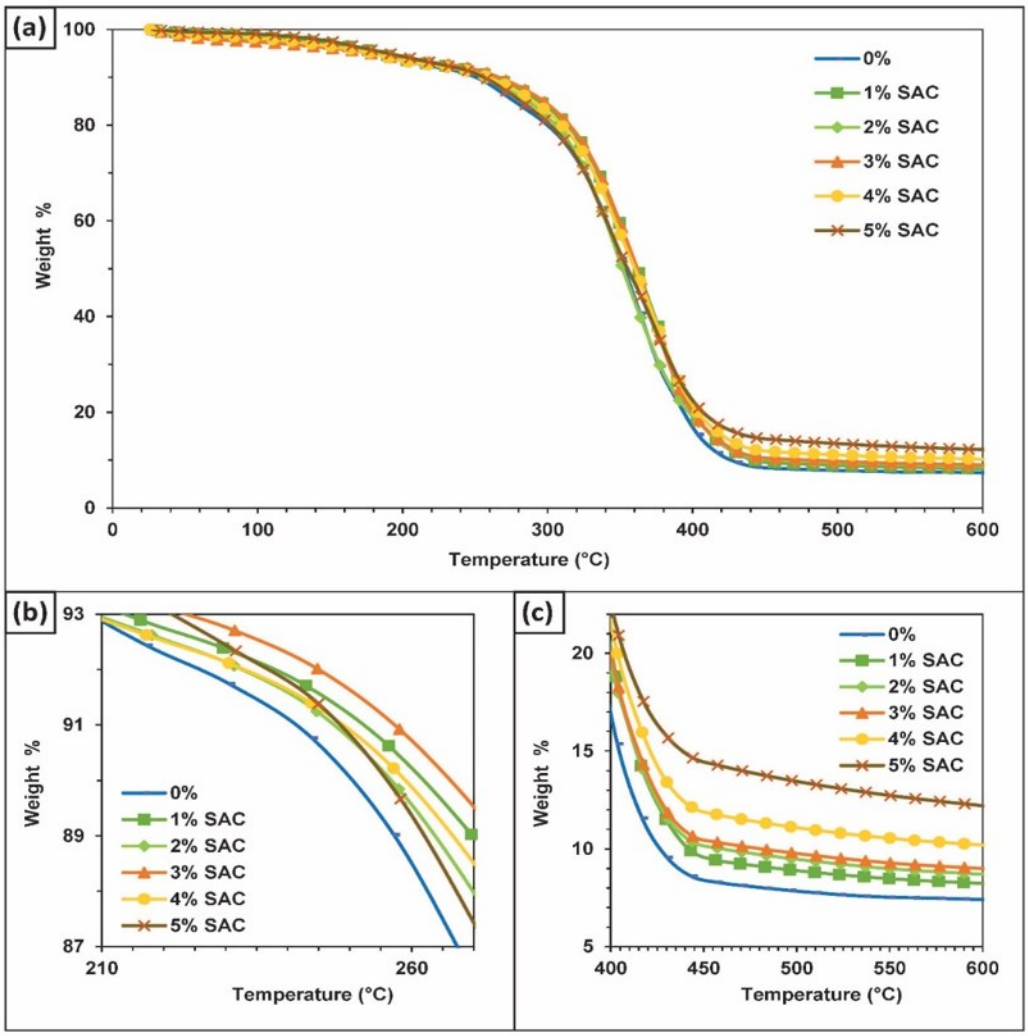

Fig. 6. Effect of SA on TGA for temperature range: (a) 30 to 600 °C, (b) 210 to 270 °C, and (c) 400 to 600 °C

The initial loss of weight, between 50 and 150 °C, can be attributed to the removal of moisture and volatile content from the composites. A rapid decomposition was seen between the temperatures of 220 and 440 °C. The constituents of SPF, such as hemicellulose, cellulose, and lignin, are known to decompose in the temperature range of 200 to 300 °C, 300 to 400 °C, and 165 to 900 °C, respectively, as suggested by (Domínguez *et al.* 2008). The primary thermal decomposition of UPE resins is likely to occur between 350 and 450 °C, as per the work of (Tongco and McDonald 2026). The scission of polymeric chains within UPE results in rapid decomposition of the resin and mainly produces low molecular weight volatiles such as carbon monoxide, carbon dioxide, methane, propylene, *etc*.

The overlap of these primary thermal decompositions of SPF and UPE is evident in the weight-loss trend in Fig. 6(a) between 200 and 450 °C. Figure 6(a) indicates that, with the addition of SA, higher thermal performance is achieved across almost all stages of TGA, except for 5% SAC, which shows a rapid weight decrease in the central decomposition region. Despite the shift of decomposition temperature to a higher degree for SA-filled composites, Fig. 6(b) illustrates that the initiation of the decomposition process had no definite relation with the wt.% of SA. This may be due to the varying

reinforcing effect of SA agglomeration in the composite, as evidenced by DMA results. However, Fig. 6(c) showed a range of temperatures over which the decomposition trend was highly correlated with the SA percentage. In this temperature range, the governing factor for lesser decomposition is the increased amount of thermally resistant SA residuals. The rise in residue content indicated that, in addition to char content from SPF and UPE, partially decomposed SA content also contributed to the residue content at the analysis's end temperatures. Therefore, 5% SAC (with the highest SA wt.%) showed the highest residual weight, and 0% composite showed the highest decomposition. The increase in residue also suggests that the undecomposed SA content may have formed a thermal barrier that resisted the decomposition of other constituents at lower temperatures.

The data extracted from the TGA for all the composites are tabulated in Table 4. It is observed from Table 4 that D_0 decreased with the addition of SA, which signifies the thermal inhibiting effect of SA particles. The onset temperature of the composites was also seen to increase with the addition of up to 4% SA. A SA content of 5% had the highest degrading effect in the first half of the analysis. This can be attributed to the catalytic impact due to the high concentration of silica additives in the composite, as suggested in earlier research (Matykiewicz 2020). However, the thermal performance of 5% SAC was seen to improve at higher temperatures. The addition of SA enhanced the thermal stability of SPF/UPE composites, as evident from the improvements analyzed in Table 4.

Table 4. Weight Percentage Values (%) at Various Temperatures, Decomposed Weight during the Central Decomposition Region (D_0), Onset Temperature (T_0), and Final Residue Values of the Thermally Degraded Samples

Sample	Weight Loss (%) at Selected Temperatures			D_0 (%)	T_0 (°C)	Final Residue (%)
	100 °C	**220 °C**	**440 °C**			
0%	98.10	92.30	8.75	83.55	305.62	7.40
1% SAC	98.49	92.75	10.17	82.58	309.54	8.22
2% SAC	97.90	92.55	10.56	81.99	316.32	8.70
3% SAC	97.38	93.13	10.87	82.26	317.73	8.99
4% SAC	98.18	92.52	12.33	80.19	313.75	10.20
5% SAC	99.04	93.07	14.88	78.20	300.16	12.20

The TGA results indicated that the 5 wt.% SA composites experienced greater mass loss during the initial decomposition phase while maintaining the highest residual mass at higher temperatures. This behavior is common in lignocellulosic composites undergoing thermal degradation and isn't contradictory. In the initial phases of degradation, some thermally sensitive components break down, producing reactive intermediates. These intermediates can then participate in condensation and crosslinking reactions with other degradation products, such as char-forming components, resulting in the formation of more thermally stable carbon-based structures. Although more decomposition may occur at intermediate temperatures, the resulting condensed char may exhibit higher thermal resistance, leading to a greater residual mass by the end of the analysis. In addition, the inherent thermal stability of silica aerogel further contributes to the higher residue content observed in the 5 wt.% SA composites. Thus, numerous thermal degradation processes govern mass loss across different temperature ranges. Transition mass loss is mainly due to decomposition reactions, while the final residue results from char formation and thermally stable inorganic components.

CONCLUSIONS

The present study investigates the effect of varying weight percentages of silica aerogel (SA) on enhancing the thermal properties of sugar palm fiber/unsaturated polyester (SPF/UPE) composites. The results revealed that SA contributed to increased thermal performance of the composites.

1. Dynamic mechanical analysis (DMA) showed that 3 wt.% SA exhibited the highest *E*' and *E*", indicating improved elastic response, interfacial adhesion, fiber–matrix interaction, and greater energy dissipation. Additionally, a significant increase in T_g was observed with the addition of SA.
2. Thermogravimetric analysis (TGA) results demonstrated an increase of nearly 12 °C in the onset temperature at 3 wt.% SA, while 5 wt.% SA showed the highest residue weight at the final temperature.
3. The increased residue content with SA addition signifies enhanced thermal stability of the composites.
4. Hence, it can be concluded that SA reinforcement enhanced the thermal efficiency of SPF/UPE composites, and the present research opens new avenues for broader applications.

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The authors declare that no funding was received for this study.

Conflicts of Interest

The authors declare no conflict of interest.

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