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Dynamic Viscoelastic Properties of Kenaf Fiber Reinforced Shape Memory Polymer Composites

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Abstract

This paper comprehensively investigates the dynamic viscoelastic behavior of shape memory polymer composites (SMPCs) reinforced with different weight percentages of kenaf fiber (KF) ranging from 0%, 5%, 10%, 15%, 20%, 30% and 40%. The dynamic mechanical behavior of these composites was characterized using dynamic mechanical analysis (DMA) over a range of temperatures. The objective was to determine the optimal fiber content of KF as reinforcement in SMPCs, specifically on viscoelastic response, storage modulus, loss modulus, damping and glass transition behaviour. The results revealed a clear correlation between the KF contents and the dynamic mechanical properties of SMPCs. The storage modulus significantly improves at higher KF content, particularly at elevated temperatures. Additionally, a quantitative assessment of coefficient C demonstrates strong interfacial bonding between fibers and the matrix in samples 30KF and 40KF. These samples also exhibit higher loss modulus and lower tan delta values, providing evidence for the efficacy of KF in enhancing composite properties. Moreover, higher KF contents induce a shift in the glass transition temperature, signifying enhanced in fiber-matrix interaction and thermal stability. The Cole-cole further demonstrates that at higher KF content, the sample surpasses Neat SMPU, presenting compelling evidence of improved matrix-fiber bonding. Statistical analysis through one-way analysis of variance (ANOVA) substantiates the statistical significance of the dynamic mechanical properties across the different weight percentages of KF-SMPCs. Based on these findings, 30KF is the optimal fiber content, balancing mechanical enhancement and feasible fabrication. This decision is grounded in challenges encountered at 40KF, where ensuring composite homogeneity becomes complex. This study contributes to the growing body of knowledge on utilization of natural fibers in development of advanced polymer composites while maintaining eco-sustainability.

1. Introduction

Shape memory materials (SMMs) have garnered substantial acclaim within the manufacturing sector due to their exceptional ability to alter their shape and revert to their original form. This unique property of SMMs opens a wide application of SMMs in the aerospace, biomedical, textile, and automotive industries. SMMs have a wide range of categories, including shape memory alloys (SMAs), shape memory polymers (SMPs), and shape memory ceramics (SMCs) [1]. Within SMMs, SMPs have captured considerable interest from academic and industrial sectors due to their remarkable characteristics. These advantages encompass their ability to be engineered for biocompatibility, their ease of manufacturing, their inherently low density, and the capability to tailor their recovery temperature to specific applications [2].

Among the various types of SMPs, shape memory polyurethane (SMPU) stands out for its exceptional versatility, which has led to its use in a diverse range of applications, from self-repairing materials to expandable structures in the aerospace industry. The capacity to fine-tune its molecular structure endows SMPU with a variety of adjustable characteristics, including adjustable permeability, efficient energy damping, and excellent shock absorption. Nonetheless, the use of SMPU in composite applications is constrained due to its limited stiffness, thermal stability, and mechanical strength, prompting a demand for enhancements in mechanical properties [3].

To overcome these limitations, there has been an ongoing search for effective reinforcement materials. Glass fibers (GF) have been widely employed as reinforcement in shape memory polymer composites (SMPCs) to address these challenges. Research has shown that GF can significantly enhance the mechanical properties in SMPU, resulting in increased storage modulus and shift in the glass transition temperature (Tg), thereby expanding the materials' ability under a broader range of temperature [4]. For instance, Gupta & Rohit, [5] investigated the dynamic mechanical properties of composites that utilized GF as reinforcement in epoxy materials. The study explored the effect of varying number of layers of GF on the composite properties and discovered that the composite with the most layers exhibited the highest storage modulus value which indicates that the reinforcement had a substantial impact on the composite's mechanical performance.

While the utilization of GF and other synthetic fibers has contributed to the advancement of SMPCs, environmental sustainability has become an increasingly important consideration. The production and disposal of synthetic fibers comes with environmental challenges, including non-biodegradability and high energy consumption during the production processes. Consequently, there has been a shift towards exploring and employing natural fibers as eco-friendly alternatives. Among several candidates, kenaf fibers (KF) have emerged as a promising reinforcement material. Extracted from the bast of the kenaf plant (Hibiscus cannabinus L.), KF offers an attractive combination of high strength and stiffness, making it an environmentally sustainable choice with potential to match the performance of traditional synthetic fibers [6].

As the field of composite materials advances, the exploration and adaptation of new reinforcement strategies, such as the use of natural fibers, are gaining traction. The successful application of these innovative materials in real-world scenarios demands an in-depth understanding of their mechanical behaviors and performance under various conditions. To this end, dynamic mechanical analysis (DMA) becomes a pivotal technique. It allows for the precise characterization of materials under dynamic loading, providing valuable insights into how their properties evolve with temperature changes [7]. DMA sheds light on the viscoelastic attributes of a material, which are critical for predicting and tuning performance for specific applications.

Before implementing shape memory polymer composites (SMPCs) in structural applications, it is crucial to subject them to meticulous testing protocols. These protocols are designed to rigorously evaluate the performance of the composite structure under a wide range of stress conditions. Testing for damping behavior, for instance, is essential for applications that require energy dissipation such as vibration control systems or impact-resistant materials.

Hence, the current study is designed with the objective of exploring the viability of kenaf fibers (KF) as a reinforcing agent in SMPCs. It seeks to unravel the complex interplay between the KF volume fraction and the dynamic mechanical properties of the composites. Understanding the dynamic mechanical properties is essential, as they offer a direct view into the viscoelastic behavior of materials under varying thermal scenarios [7].

This research contends with several key issues. Firstly, it tackles environmental concerns by investigating eco-friendly reinforcement options. The use of natural fibers like KF is not only sustainable but also reduces the ecological footprint of composite materials. Secondly, the study examines how these enhanced mechanical properties can be harnessed to create bespoke solutions for specialized applications. Every field from aerospace to automotive to civil engineering demands materials with specific performance criteria, and tailored fiber-reinforced composites hold the promise of meeting these stringent requirements.

The relevance to industries in pursuit of lightweight yet durable materials is another facet of this study. In an era where fuel efficiency and high performance are paramount, the potential of KF-reinforced SMPCs to deliver strength without adding significant weight is of immense value. Furthermore, this research contributes to the broader scientific discourse on material design and innovation. By analyzing the dynamic responses of these composites, it adds a layer of fundamental understanding that is essential for future material development.

Moreover, this investigation probes the potential for breakthroughs in material science. The unique properties of SMPCs reinforced with natural fibers could lead to the development of innovative products and applications, ranging from smart textiles that change shape with temperature, to components in electronics that must respond dynamically to environmental changes.

In addition to contributing to academic knowledge, the practical implications of this research are vast. Industries that could benefit include construction, where materials must be both strong and responsive to temperature and stress; automotive and aeronautics, which constantly seek out materials that offer high performance without compromising on weight or safety; and the biomedical field, where SMPCs could be used to create smart prosthetics that adapt to the user's movements.

By providing an extended analysis of the effects of varying KF content on the dynamic mechanical properties of SMPCs, this study aims to bridge a significant knowledge gap. It endeavors to inform and inspire the next generation of composite material engineering, leading to applications that are as diverse as they are transformative. The research also lays a foundation for future work, encouraging ongoing innovation and exploration in the realm of composite materials. Through this comprehensive approach, the study aspires to usher in a new era where material science not only addresses the needs of today but also anticipates the challenges and opportunities of tomorrow.

2. Methodology

2.1 Materials Preparation

The Shape Memory Polyurethane (MP5510) (SMPU) (see Fig. 1) was obtained from SMP Technologies Inc, Tokyo Japan while kenaf fiber (KF) chopped strand mat (see Fig. 2) utilized in this study was obtained from KEFI Malaysia Sdn. Bhd. The SMPU was supplied in two parts, resin, and hardener (A & B) and was meticulously processed in accordance with the comprehensive guidelines specified by the manufacturer. Table 1 below shows the properties of SMPU.

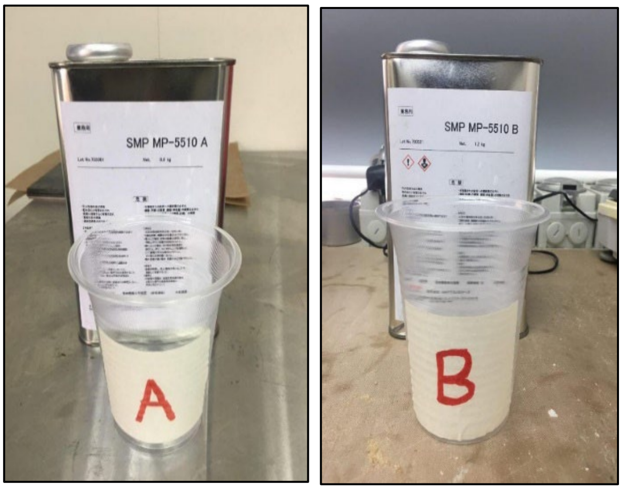

Fig. 1 *Resin (A) and hardener (B) of SMPU*

Fig. 2 *Physical appearance of Kenaf Fiber (KF) chopped strand mat*

Table 1 *Properties of shape memory polyurethane (SMPU) [8]*

Item	Properties	Results
SMPU Resin	Specific gravity	1.062
SMPU Hardener	Specific gravity	1.215
SMPU Resin/Hardener	Weight ratio	40:60
	Density	1.208
	Pot life (mins)	3
	Glass transition temperature (°C)	55
	Cure temperature (°C)	70
	Cure time (hr)	1-2
Physical properties (G/R)	Tensile strength (MPa)	52
	Elongation	10-30
Physical properties (R/R)	Tensile strength (MPa)	20
	Elongation (%)	>400

2.2 Composite Fabrication

All specimens in this study were formulated and designated following the manufacturer's recommended guidelines, as detailed in Table 2 below. This formulation adhered to a consistent weight ratio, comprising 40% reinforcement and 60% matrix components. The research entailed fabricating seven (7) composite specimens, each with different fiber weight percentages of 0%, 5%, 10%, 15%, 20%, 30%, and 40%. These series were designated as Neat SMPU, 5KF, 10KF, 15KF, 20KF, 30KF, and 40KF. The maximum percentages of fibers were set to 40% as higher contents since it can generate difficulties during the mixing process due to its high viscosity, which lead to improper dispersion of the fibers. As a result, the quality of the composites produced will be reduced. In this study, Neat SMPU was chosen as the control specimen while SMPU incorporated with KF was the main developed composites specimens in the present study.

Table 2 *Details of the composition and designation of the SMPCs*

No	Composition Designation	SMPU Content (wt.%)	Kenaf Fiber Content (wt.%)
1	Neat SMPU	100	0
2	5KF	95	5
3	10KF	90	10
4	15KF	85	15
5	20KF	80	20
6	30KF	70	30
7	40KF	60	40

The hand lay-up method was implemented for the manufacturing processes, and it was shown in Fig. 3 below. The first step involved pulverizing KF mat into chopped fibers using a crusher, ensuring a consistent fiber length that would facilitate even distribution within the composite matrix. These fibers were weighed according to the weight percentages specified in Table 2 above, ensuring precise quantities for the intended fiber contents. They were evenly distributed over an aluminum mold. Polytetrafluoroethylene (PTFE) release film was affixed to both upper and bottom of the mold to ensure easy removal of the cured composite from the mold while also contributing to the good surface finishing of the composite sample.

Subsequently, in preparing the matrix component, the SMPU resin and hardener were meticulously hand-mixed in a 40:60 mass ratio for 30 seconds. This mixing process was executed gradually to prevent excessive air bubble incorporation within the resin, which can compromise the composite's structural integrity and mechanical performance. Due to the reactive nature of the resultant mixture, which had a pot life of approximately 3 minutes, it was essential to pour the mixture of resin and hardener blend immediately into the mold containing chopped KF. Special attention was devoted to eradicating air bubbles using a hand-rolling tool. This is to ensure the elimination of any entrapped air that could lead to voids within the finalized composite. A roller was subsequently employed to uniformly spread the mixture evenly across the mold's surface to achieve the desired thickness. This step is critical to maintain uniform material properties. Next, the curing process was conducted in two stages. Initially, the composite was left to cure at room temperature for 10 minutes, allowing the resin to harden sufficiently. This was followed by a post-curing phase of 2 hours at 70°C, a temperature that facilitates complete polymerization and cross-linking while avoiding any thermal degradation of the KF. The post-curing stage is instrumental in enhancing the mechanical strength and thermal stability of the composites. After the curing process, all the specimens were carefully removed from the molds and stored at room temperature prior to experimental testing.

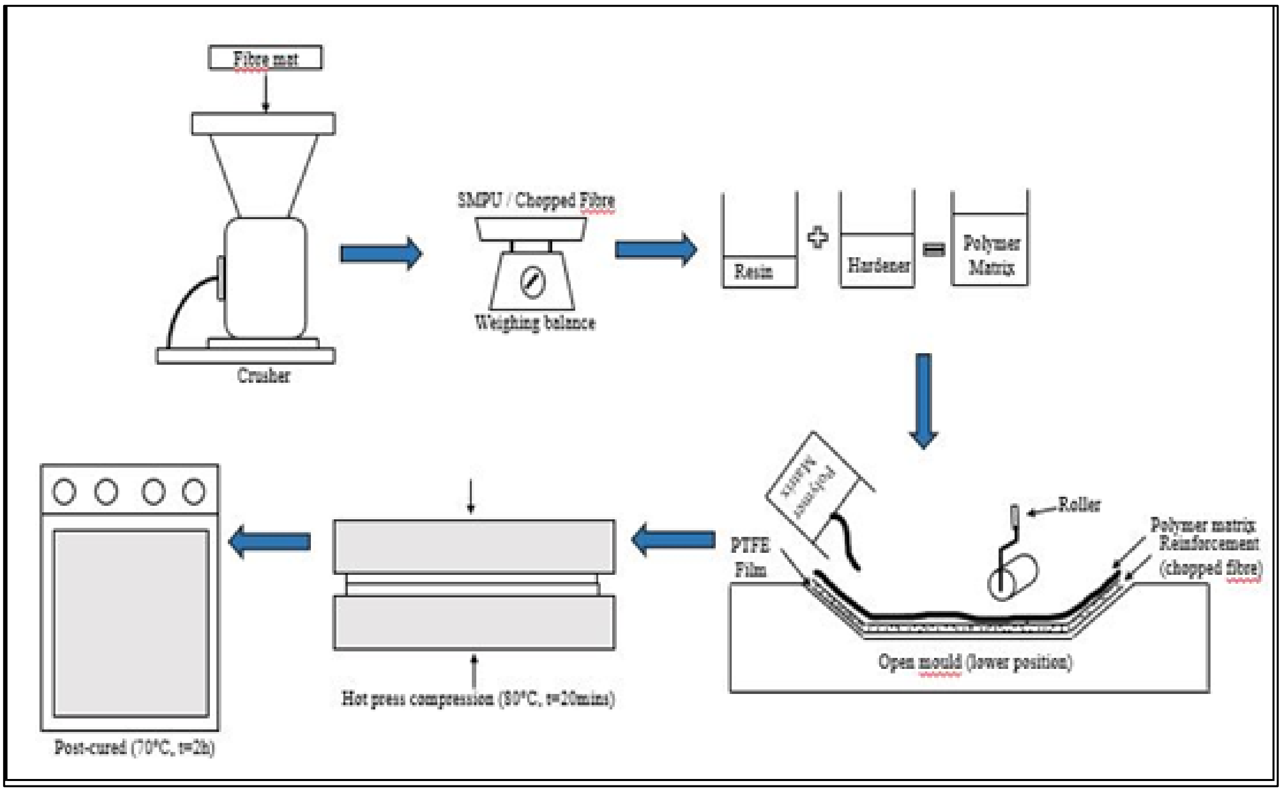

Fig. 3 *Fabrication process of SMPCs*

2.3 Characterization Techniques

Dynamic mechanical analysis (DMA) has emerged as a prevalent technique for ascertaining heterogeneous polymeric systems' viscoelastic properties and interfacial characteristics. The present investigation entailed the preparation of specimens in adherence to ASTM D406-01 standards, with a specimen dimension of 35mm x 6mm x 2mm. The DMA test was conducted in the room temperature range, 25°C to 150°C, using a three-point bending mode at a fixed frequency of 1Hz, 3 °C/min heating rate and a constant deformation of 0.1% (see Fig. 3). The parameters for the testing are tabulated in Table 3 below. The DMA parameters selected for this experiment are grounded in established research [9], ensuring that the methodology is both scientifically sound and tailored to the specific characteristics of the SMPCs under investigation.

DMA analysis is an effective method for assessing the effect of reinforcements in composites. Furthermore, due to the molecular movement of chain segments, DMA can provide insights into assessing the relaxation and transition processes [10]. This is accomplished by examining how the addition of fibers influences key mechanical attributes – specifically, the storage modulus (E'), loss modulus (E") and loss factor or tan delta (tan δ) within the composites. By examining how the fibers impact the viscoelastic properties, researchers can gain valuable insight into the composite's potential performance in practical applications, such as its ability to absorb shock, its resistance to deformation under loading, and its thermal stability.

Table 3 *Experimental conditions for DMA [9]*

Parameter	Unit	Range
Temperature	°C	25-150
Heating rate	°C/min	3
Frequency	Hz	1
Sample dimensions	mm	6 x 35
Deformation	%	0.1

Loading mode	-	Three-point bending

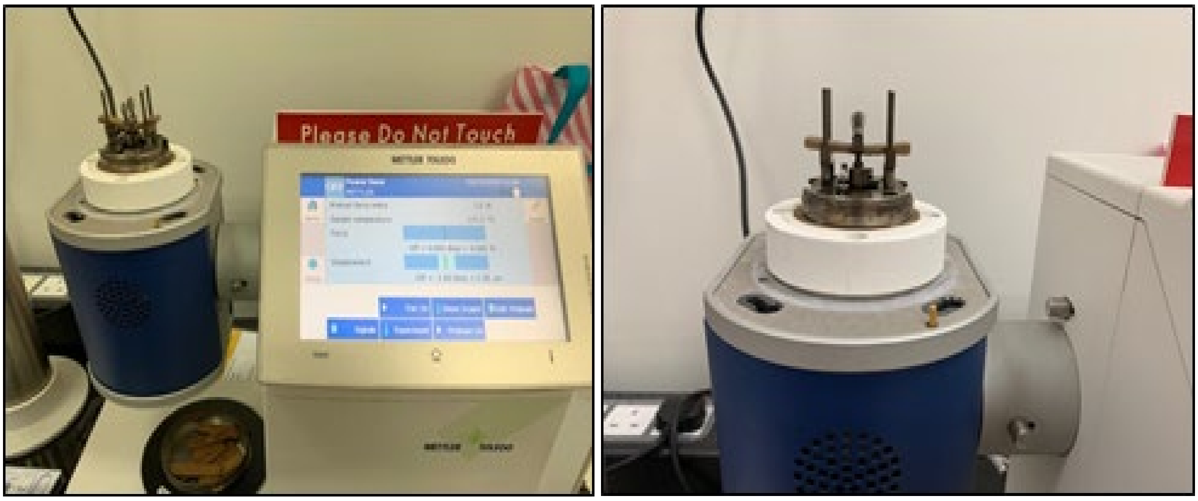

Fig. 3 *Dynamic mechanical analyzer and sample testing for DMA test*

3. Results and Discussions

The experimental results are presented in graphical representations, with Fig. 4 depicts the correlation between storage modulus (E') versus temperature, whereas Fig. 5 depicts the correlation between loss modulus (E") versus temperature. Both figures provide insights into how these modulus values vary with different weight percentages of kenaf fibers (KF). Furthermore, additional insights are gained from Fig. 6, which presents the tan delta (tan δ) values versus temperature for varying weight percentages of KF. This will showcase the material's energy dissipation characteristics. Fig. 7 presents the Cole-cole plots, which provide a comprehensive visualization of the complex viscoelastic behavior of the composites, revealing aspects such as relaxation and molecular mobility. In the final part of the study, a statistical analysis was conducted using one-way analysis of variance (ANOVA) to assess the significance of different weight percentages of KF in SMPCs. This analysis helps determine whether the variations observed in the dynamic mechanical properties across the various KF content levels are statistically significant, thus providing a robust foundation for drawing meaningful conclusions from the experimental data.

3.1 Storage Modulus

The storage modulus (E') is a fundamental parameter characterizing a materials' elastic behavior, reflecting its ability to resist deformation and maintain dimensional stability. This property is especially critical in applications that demand high dimensional stability and load-bearing capacity, such as structural components in aerospace, automotive and civil engineering. In the investigation of shape memory polymer composites (SMPCs) reinforced with varying kenaf fiber (KF) contents, the storage modulus exhibited a crucial role in determining the composite's stiffness across different temperatures.

As depicted in Fig. 4 below, the storage modulus for SMPCs reinforced with varying weight percentages of KF exhibits an inverse relationship with temperature, a phenomenon commonly observed in polymer composites [11]. The general trend shows a decrease in storage modulus with increasing temperature. This decline in stiffness can be attributed to the increased mobility of the polymer chains as the temperature rises, leading to a softening of the matrix material. This observation is in line with the findings reported by previous researchers. For instance, Khiyon *et al.*, [4] studied the dynamic mechanical behavior of glass fiber-reinforced SMPCs and reported a reduction in storage modulus with increasing temperature due to polymer chain mobility.

Comparing the Neat SMPU with SMPCs incorporating KF, it was evident that the Neat SMPU exhibits higher storage modulus at lower temperature, which is 2172.64MPa. However, the storage modulus noticeably decreases as temperature increases. This signifies that the Neat SMPU experiences loss stiffness due to the higher molecular movement at elevated temperatures. The introduction of reinforcement fibers into the composite was aimed at enhancing material stiffness, leading to increased storage modulus for KF-reinforced SMPCs at elevated temperatures, as evident in Table 4.

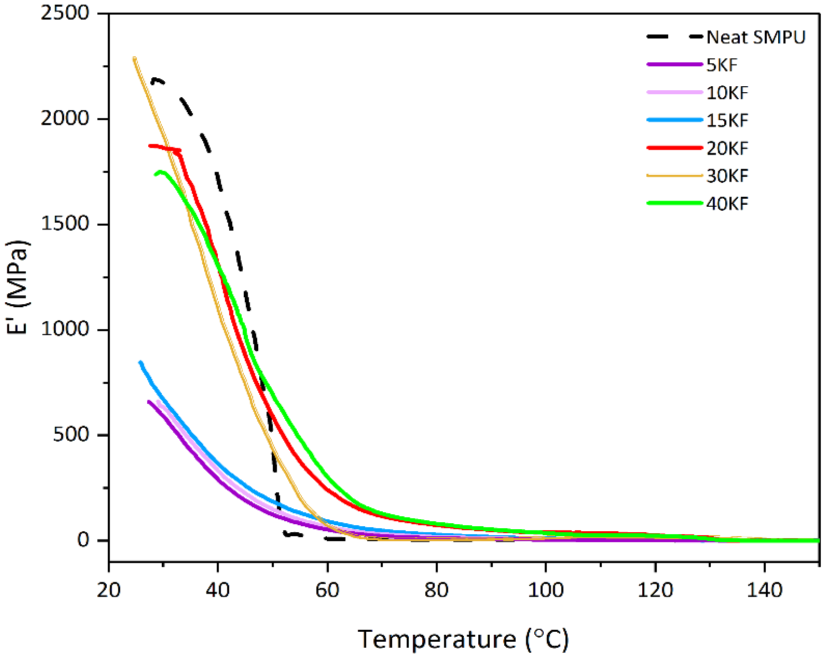

Fig. 4 *The storage modulus (E') of different weight percentages of KF-Reinforced SMPCs*

Table 4 *DMA parameters for KF-reinforced SMPCs at different weight percentages of fibers*

Sample	E'_g, 30°C (MPa)	E', 55°C (Mpa)	E'_r, 120°C (MPa)	E" (MPa)	Tg (°C)	Tan delta	T_g (°C)	C
Neat SMPU	2172.64	26.11	3.45	249.65	46.77	0.47	53.06	-
5KF	577.70	80.88	3.65	138.91	29.03	0.29	51.87	0.25
10KF	626.31	98.73	4.20	148.39	27.33	0.32	52.36	0.24
15KF	684.13	135.15	4.86	163.73	25.76	0.36	52.90	0.22
20KF	1862.00	380.31	18.74	291.34	35.96	0.40	53.18	0.16
30KF	1927.69	214.43	26.85	324.64	35.98	0.33	55.40	0.11
40KF	1742.35	495.52	24.67	264.38	38.92	0.33	61.24	0.11

Table 4 above provides a detailed overview of the parameters for the dynamic mechanical properties at different temperatures for various weight percentages of KF in SMPCs. Significantly, SMPCs with KF contents surpassed Neat SMPU in storage modulus within the rubbery region. This enhancement is attributed to the constrained molecular mobility of polymer chains and the establishment of strong interfacial bonding between the matrix and KF, as elucidated by previous studies [12]. The reinforced material demonstrated improved thermal stability at higher temperatures [13].

Intriguingly, the reinforcing effect became more pronounced with higher KF content. For instance, E'g values at 30°C for 20KF, 30KF, and 40KF were 1862MPa, 1927.69MPa and 1742.35MPa, respectively, indicating a substantial enhancement in the rigidity of the composites [14]. This phenomenon is likely attributed to the intensified interaction between the matrix and fiber composition, facilitating improved stress transmission from the resin to the fiber. The addition of KF not only improved the strength but also increased the mechanical rigidity of the composites [15].

Moreover, the effectiveness of KF as a reinforcing agent can be quantified using the coefficient C, as defined in Equation (1) below.

$$C = \frac{\left(\frac{E_g'}{E_r'}\right)_{composite}}{\left(\frac{E_g'}{E_r'}\right)_{resin}} \tag{1}$$

A lower value of constant C indicates higher effectiveness of fibers or fillers in the composite material [16]. The values presented in Table 3 showcase the efficiency of KF in reinforcing the SMPCs, with the lowest constant C observed for 30KF and 40KF at 0.11. This reaffirms the effectiveness of fiber-matrix interactions in these composites.

In conclusion, the analysis of storage modulus, supported by coefficient C values, underscores the efficacy of KF as reinforcement in SMPCs. The balance achieved between enhanced mechanical properties and manageable fabrication processes, particularly at 30KF for storage modulus, positions it as the optimal fiber content for practical applications. In the subsequent section, we will delve into further aspects, including the loss modulus, tan delta and shifting in glass transition temperature, providing a comprehensive understanding of the multifaceted behavior of these KF-reinforced SMPCs.

3.2 Loss Modulus

The loss modulus (E'') is a pivotal parameter in the characterization of viscoelastic materials, measures the energy dissipation within a material when it is subjected to an oscillatory deformation. This energy is dissipated as heat and is indicative of the viscous component of the material's response to stress. materials. Fig. 5 below displays the variation of loss modulus with temperature for SMPCs incorporated with varying KF contents and Neat SMPU. It can be observed that loss modulus decreases upon the increment of temperature. It was the same trend as the storage modulus discussed previously.

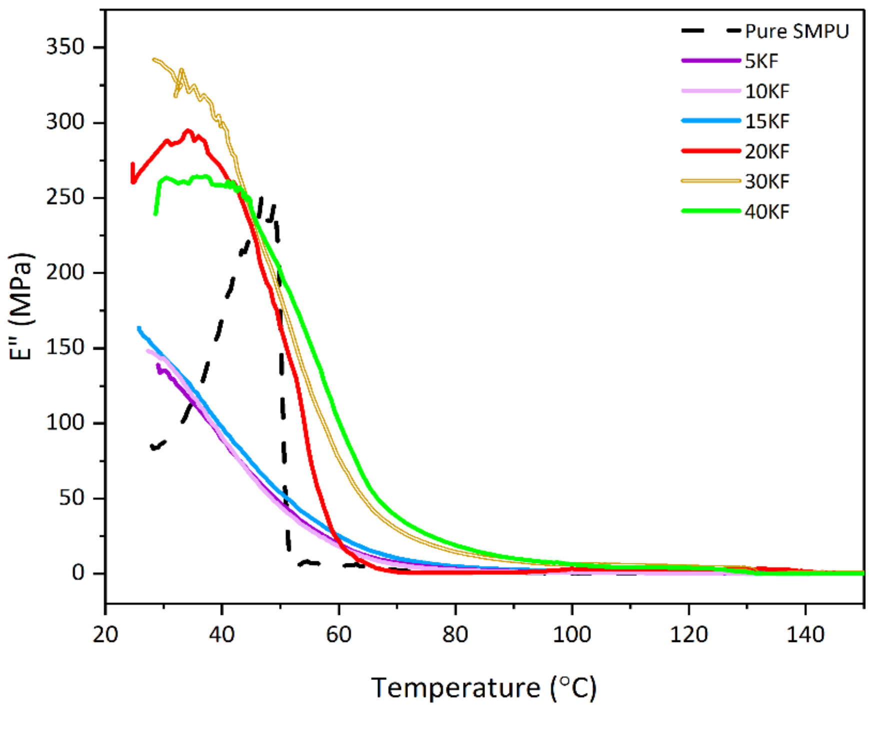

Fig. 5 *The loss modulus (E”) of different weight percentages of KF-Reinforced SMPCs*

At lower temperature, the Neat SMPU exhibits a loss modulus of 249.65 MPa, which signifies a significant amount of energy dissipation likely due to the movement of the polyurethane chains within the SMPU matrix. However, as the temperature increases, there is a discernible decrease in loss modulus across all samples. This reduction can be connected to the transition from a glassy to a rubbery state, where the material's internal friction and thus energy dissipation decrease as the polymer chains gain mobility and the material softens.

When KF is introduced into the SMPU matrix at lower contents, the loss modulus experiences a reduction. Previous authors suggested that this behavior can be related to the fact that the fibers act as flaws in the composites at such low loadings and low temperatures [17]. Besides, this decrease may be due to the fibers acting as stress concentrators or initiating points for the relaxation of the polymer chains. In such cases, the reinforcing fibers can disrupt the continuity of the matrix and create regions where energy dissipation is less efficient than in the Neat SMPU.

However, as the fiber content increases to 20KF, 30KF, and 40KF, the loss modulus values exhibit a notable increase, suggesting an enhancement in the composite's ability to dissipate energy. This trend aligns with findings from previous studies such as those by Rana et al. [18], where the addition of fibers to a polymer matrix led to an increase in loss modulus. The higher loss modulus values at increased fiber loadings suggest that the KF are effectively interacting with the SMPU matrix to restrict the viscoelastic behavior by impeding the molecular displacement under external forces [15].

The order in which the peaks of the loss modulus curves appear—30KF > 20KF > 40KF > Neat SMPU > 15KF > 10KF > 5KF—indicates that the amount and distribution of KF within the composite significantly influence the energy dissipation characteristics. The peak of the loss modulus curve represents the point of maximum energy dissipation within the material, which typically occurs around the glass transition temperature (Tg). The temperature at which this peak occurs can provide insights into the composite's Tg, an important thermal property that dictates a material's performance in varying temperature environments.

Moreover, the shift in the peak of the loss modulus curve with varying KF content can also be related to changes in the composite's microstructure. The addition of fibers can affect the mobility of the polymer chains and the degree of crosslinking within the matrix, leading to altered relaxation processes and energy dissipation mechanisms. A higher peak in the loss modulus curve suggests that the fibers are effectively transferring stress and increasing the internal friction within the composite, which is desirable in applications requiring efficient vibration damping or energy absorption.

In summary, the analysis of the loss modulus provides valuable information about the damping properties of KF-reinforced SMPCs. The results demonstrate that by adjusting the fiber content, one can tailor the energy dissipation capabilities of the composites to meet the requirements of various applications. The findings also underscore the potential of using natural fibers like KF to enhance the viscoelastic properties of SMPUs, contributing to the development of more sustainable and high-performance materials for industries that prioritize energy efficiency and environmental responsibility.

3.3 Damping Factor (Tan δ)

The damping factor, often represented by tan δ, is an essential parameter in material science, especially when evaluating the energy dissipation characteristics of viscoelastic materials such as SMPCs. Tan δ is calculated as the ratio of the loss modulus (E") to the storage modulus (E'), and it provides insights into the material's ability to dissipate energy as heat when subjected to cyclic loading. The damping factor is associated with the internal friction of the material and is indicative of the transition between elastic and viscous behavior. In the context of SMPCs, this property is critical because it can reveal the efficiency of the material in applications requiring energy absorption, such as vibration damping or impact resistance. This introduction of fibers into the system imparts notable effects on the damping behavior of the composite. This is principally due to the formed shear stress between matrix-fibers, which reduces the additional power dissipation in the composite material [9].

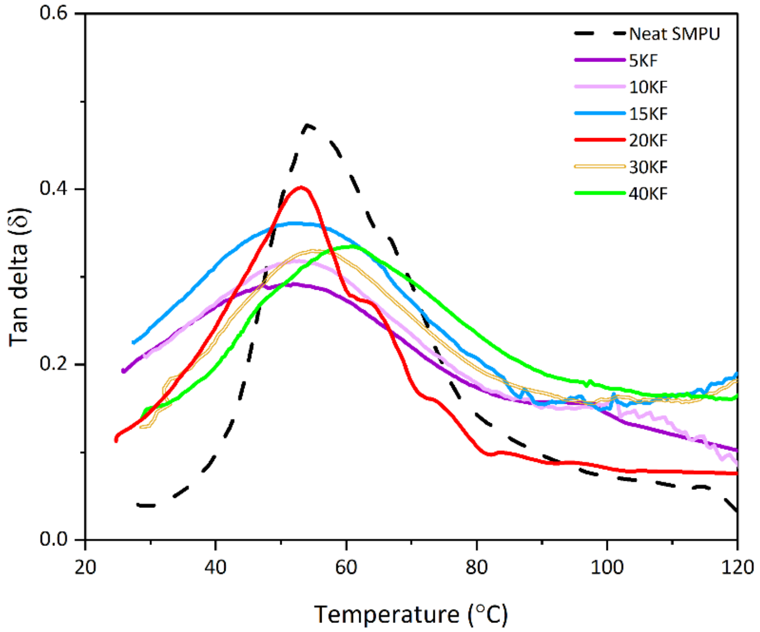

Fig. 6 *The damping factor (tan δ) of different weight percentages of KF-Reinforced SMPCs*

Figure 6 above presents the damping factor, represented by tan δ for a series of KF-reinforced SMPCs with varying fiber contents at variation of temperature. A lower tan δ value implies that the composite has a reduced capacity for energy dissipation, which can be a positive or negative attribute depending on the intended application of the material. For applications where energy dissipation is desired, such as in seismic dampers or noise reduction materials, a higher tan δ is beneficial. Conversely, for applications where energy conservation and reduced energy loss are important, such as in structural components, a lower tan δ is advantageous.

The observed pattern indicates that the tan δ values for the SMPCs reinforced with KF are typically lower when compared to Neat SMPU. Such a trend suggests that integrating fibers into the composite bolsters its capacity for energy storage, corroborating the previously noted upsurge in storage modulus. This is also consistent with outcomes from prior research, which identified that the incorporation of glass fibers into composites led to a decrease in tan δ peak values, thereby highlighting the fibers' role in reinforcing the material [4]. Furthermore, the decrease in tan δ peak suggesting a more cohesive bond between the fibers and the matrix. This effective stress transfer contributes to a reduction in the energy dissipated through internal friction, leading to a lower damping factor.

Moreover, it is important to note that the packing efficiency of the fibers within the composite plays a significant role in its damping behavior. At lower fiber contents, the packing may be inefficient, resulting in matrix-rich areas where the fibers do not effectively constrain the matrix. This can lead to localized deformations and higher energy dissipation, as the polymer matrix is free to exhibit its inherent viscoelastic behavior. However, as the fiber content increases, and the fibers become more closely packed, their presence restricts the movement of the matrix polymer chains, limiting the propagation of cracks and contributing to a decrease in tan δ.

The observed variation in glass transition temperature (Tg) with increased KF contents (namely 20KF, 30KF, and 40KF) is also significant. Previous research has found that the loading and dispersion of MWCNT in the SMPU matrix affected the Tg [19]. Tg, discerned as the temperature at which tan δ reaches a peak, indicates the material's transition from a glassy to a rubbery state. This shift indicates and enhanced interaction between the fibers and matrix, in turn demonstrating kenaf fibres' efficacy as reinforcements in SMPCs and their improved thermal stability, as indicated by the altered curve [20]. Such fiber-matrix interactions potentially affect the mobility of the polymer chains and the extent of crosslinking within the matrix, resulting in altered molecular dynamics and changes in the viscoelastic behavior of the composite.

The enhancement of the Tg and the improved thermal stability observed with higher KF contents demonstrates the efficacy of kenaf fibers as a reinforcing agent in SMPCs. This outcome highlights the potential of using natural fibers to tailor the dynamic mechanical properties of polymers, offering a sustainable approach to developing materials with specific energy dissipation characteristics.

In summary, the damping factor analysis offers valuable information about the composite's potential applications. The results indicate that by adjusting the KF content, the energy dissipation capabilities of the SMPCs can be tuned to meet the requirements of various applications. This tunability, combined with the sustainable nature of KF, allows for the design of high-performance materials with reduced environmental impact, promoting innovation in fields ranging from automotive to aerospace, where energy efficiency and material sustainability are increasingly prioritized.

3.4 Cole-Cole Plots

The Cole-Cole plot is a graphical representation of the complex viscoelastic behavior of materials and is particularly insightful for analyzing the frequency-dependent properties of polymers and their composites. By plotting the loss modulus (E'') versus the storage modulus (E'), the Cole-Cole plot provides a visual assessment of the material's damping characteristics and the homogeneity of its structure.

In a Cole-Cole plot, a perfectly homogeneous polymeric system typically exhibits a semicircular arc, with the degree of deviation from this ideal shape providing insights into the system's heterogeneity [21]. The presence of a perfect semicircle indicates that the material has a single relaxation time, suggesting uniformity in the polymer matrix and its interactions with any incorporated fillers. In contrast, deviations from a perfect semicircle can reveal the presence of multiple relaxation times, which are indicative of a heterogeneous system with varying domains of molecular mobility.

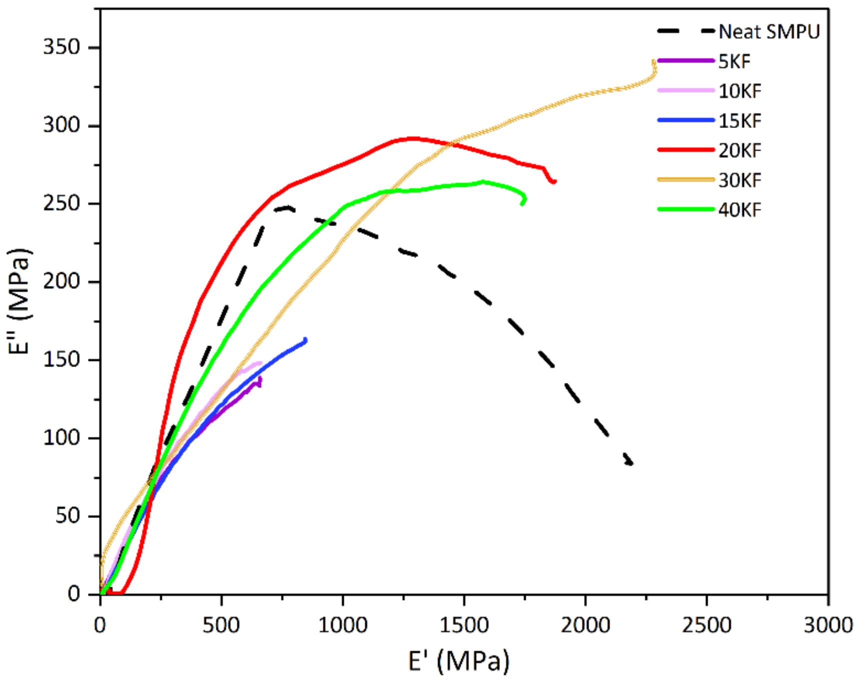

Fig. 7 *Cole-cole plot of the different weight percentages of KF-Reinforced SMPCs*

Figure 7 illustrates the Cole-Cole plots for different weight percentages of KF-SMPCs. The curves shown do not form perfect semicircles, implying that the KF-reinforced SMPCs are heterogeneous systems. This heterogeneity is likely due to variations in the distribution of the reinforcing fibers, the fiber-matrix interface, and the presence of different regions within the polymer matrix itself. The imperfect semicircular shape suggests good adhesion between the fiber and the matrix, as the fibers introduce additional relaxation mechanisms and contribute to the overall viscoelastic response of the composite [22].

The Cole-Cole plots for composites with higher KF content, specifically 20KF, 30KF, and 40KF, demonstrate more pronounced arcs compared to that of the Neat SMPU. The increased arc size for these composites is indicative of enhanced interfacial bonding, which can lead to improved mechanical properties such as stiffness and strength. Enhanced interfacial adhesion is critical for the effective transfer of stress between the matrix and the fibers during loading, allowing the composite to maintain its structural integrity under mechanical stress.

Moreover, the shape and size of the arcs in the Cole-Cole plots can also be related to the composite's microstructure. For example, the introduction of KF into the polymer matrix creates new interfaces and potentially alters the composite's morphology. The interaction between the KF and the SMPU matrix may affect the polymer chains' mobility, leading to changes in the relaxation behavior of the composite. These interactions can manifest as changes in the tan delta peak, shifts in the glass transition temperature (Tg), and variations in the storage and loss moduli, as previously discussed.

The Cole-Cole plots provide a means to evaluate the uniformity of fiber dispersion within the composite. A more uniform dispersion will generally result in a more homogenous composite, potentially yielding a Cole-Cole plot closer to the ideal semicircle. Conversely, areas of non-uniform fiber dispersion can lead to localized stress concentrations and may impact the composite's mechanical and viscoelastic performance.

Importantly, the Cole-Cole plot analysis complements other characterization techniques by offering a different perspective on the material's behavior. When combined with scanning electron microscopy (SEM) images of the composite's fracture surface or transmission electron microscopy (TEM) images of the composite's microstructure, the Cole-Cole plot can provide a more comprehensive understanding of the relationship between the composite's structure and its properties.

In summary, the Cole-Cole plot analysis of KF-reinforced SMPCs reveals the complex interplay between the polymer matrix and the reinforcing fibers, providing valuable information on the composite's viscoelastic behavior, structural homogeneity, and the efficacy of fiber reinforcement. This analysis is crucial for the design and optimization of composite materials, where achieving the desired balance of mechanical properties and damping characteristics is key to their performance in practical applications.

3.5 Statistical Analysis (ANOVA)

One-way analysis of variance (ANOVA) was used to assess the significant variations among the average storage modulus, average loss modulus, and average tan delta values across different weight percentages of KF in SMPCs. This analysis helps us discern the distinctions between the groups (BG) and the variations within the groups (WG), as summarized in Table 5. The between groups comparison elucidates the influence of varying KF weight percentages, while the within groups assessment accounts for inherent measurement errors. The F-value, derived from the ratio of BG to WG differences, is a crucial indicator of the impact of weight percentages of KF on SMPC properties.

Table 5 *One-way ANOVA data analysis on storage modulus, loss modulus, and tan delta of KF-reinforced SMPCs at different weight percentages of fibers*

Properties	*Source of Difference*	*SS*	*df*	*MS*	*F-Value*	*p-Value*	*F critical*
Storage Modulus	Between Groups (BG)	5098034	2	2549017	16.075	0.000186	3.682
	Within Groups (WG)	2378625	15	158575			
Loss Modulus	Between Groups (BG)	107998.10	1	107998.10	32.721	0.000193	4.964
	Within Groups (WG)	33005.60	10	3300.56			
Tan delta	Between Groups (BG)	8797.75	1	8797.75	1418.005	0	4.964
	Within Groups (WG)	62.04	10	6.20			

One-way analysis of variance (ANOVA) is a powerful statistical technique employed for the purpose of comparing means across various groups to ascertain the presence of statistically significant disparities between them. In materials science, ANOVA can be applied to dynamic mechanical analysis data to discern the effect of varying material compositions, such as the weight percentages of fiber reinforcement in SMPCs.

The one-way ANOVA evaluates the hypothesis that all groups share a common mean in contrast to the alternative proposition that at least one group possesses a distinct mean. It divides the overall observed variance in the data into two components: between group variance (BG) which is linked to the experimental manipulations or treatments, and within group variance (WG), which is ascribed to random error or inherent variability in the measurements.

Table 5 above displays the results from the one-way ANOVA conducted on the storage modulus, loss modulus, and tan delta of KF-reinforced SMPCs with different fiber weight percentages. The table encompasses sums of squares (SS), degrees of freedom (df), mean squares (MS), F-values, p-values, and the critical F-values (F critical). The sums of squares (SS) signify the overall variation in the data. The degrees of freedom (df) are associated with the quantity of levels for the factor being analyzed and quantity of observations. The mean squares (MS) are calculated by dividing the sum of squares by their respective degrees of freedom and represent the average variation.

The F-value is a key metric in ANOVA, calculated as the ratio of the mean square between groups (MS_BG) to the mean square within groups (MS_WG). If the groups are indeed different, we expect a larger variation between groups than within groups, which would lead to a higher F-value. The p-value provides the probability of observing such an F-value if the null hypothesis were true (i.e., all group means are equal). A low p-value (typically less than 0.05) indicates that it is unlikely to observe such a difference by chance alone, leading to the rejection of the null hypothesis.

In the presented data, the F-values for storage modulus, loss modulus, and tan delta are all significantly higher than the corresponding F critical values. This suggests that the variations in the means of these properties are not likely due to random chance and that the differences are statistically significant. The p-values for all properties are lower than the threshold of 0.05, reinforcing the conclusion that the differences in means across the different weight percentages of KF in SMPCs are statistically significant. This signifies that the incorporation of KF into SMPCs has a meaningful impact on their dynamic mechanical properties.

The statistical analysis demonstrates that the variation in KF content is a significant factor influencing the storage modulus, loss modulus, and tan delta of the composites. As the weight percentage of KF increases, the changes in these properties reflect the effectiveness of KF reinforcement in modifying the viscoelastic behavior of the SMPCs. The significant p-values indicate that these are not random variations but are directly correlated with the fiber content, emphasizing the role of KF in enhancing the material properties of SMPCs.

In practical terms, this statistical validation is valuable for materials scientists and engineers who are designing and optimizing SMPCs for specific applications. Comprehending the statistical significance of fiber reinforcement enables a higher degree of accuracy in managing the characteristics of the material, thereby guaranteeing that the end product satisfies the requisite performance standards for its intended application. Additionally, statistical examinations such as ANOVA play a vital role in the realm of quality control within manufacturing processes. By confirming that the variations in material properties are attributable to alterations in composition rather than random deviations, manufacturers can enhance their confidence in both their production techniques and the formulation of materials.

In conclusion, the one-way ANOVA analysis provides a rigorous statistical framework to confirm that the differences observed in the dynamic mechanical properties of KF-reinforced SMPCs are indeed due to the variations in KF.

4. Conclusions

In this study, the dynamic mechanical properties of KF-SMPCs have been systematically investigated to elucidate the effects of kfcontent on the material's viscoelastic behavior. The findings provide compelling evidence that the incorporation of KF significantly enhances the stiffness of SMPCs, particularly at elevated temperatures where the intrinsic stiffness of the fibers and their effective bonding with the SMPU matrix become more pronounced.

The use of coefficient C as a quantitative measure of fiber-matrix interaction strength has proven insightful. The results clearly indicate that samples with 30% and 40% KF content (30KF and 40KF) exhibit strong fiber-matrix interactions, as reflected by higher values of the loss modulus and lower tan delta values. These observations are consistent with the notion that an increase in KF content leads to more effective load transfer and energy dissipation mechanisms within the composite system. Additionally, the observed shift in the glass transition temperature (Tg) with higher KF content is a testament to the improved interaction between the fibers and the matrix, which in turn manifests as enhanced thermal stability in the composite material.

The Cole-Cole plot analysis further substantiates that the samples with higher KF content display improved performance compared to the Neat SMPU. This is indicative of the synergistic effects that arise from the integration of KF into the SMPU matrix, leading to a composite material that is not only stiffer but also exhibits superior energy dissipation characteristics.

However, the study also underscores the practical limitations associated with excessive KF content. While a 40KF sample may possess superior mechanical properties in theory, the practical challenges of homogenously incorporating such a high volume of fibers into the matrix cannot be overlooked. These challenges include difficulties in material processing, potential for the introduction of voids, and the risk of uneven fiber distribution, all of which can negatively impact the overall quality and consistency of the final composite material. As such, a KF content of 30% (30KF) emerges as the optimal choice, striking an ideal balance between mechanical property enhancement and manufacturability.

The statistical robustness of these conclusions is confirmed by ANOVA analysis, which reveals that the variation in KF weight percentage significantly influences the dynamic mechanical properties of the SMPCs. The calculated F-values for storage modulus, loss modulus, and tan delta are all well above the critical values, and the associated p-values fall below the threshold of 0.05. This signifies that the differences in dynamic mechanical properties with varying KF content are statistically significant and not due to random chance, lending strong support to the conclusions drawn from this study.

In conclusion, the research conducted presents a comprehensive analysis of the effects of KF reinforcement on the dynamic mechanical behavior of SMPCs. The results demonstrate that KF reinforcement not only bolsters the stiffness and thermal stability of SMPCs but also finely tunes their damping properties, making them suitable for a myriad of applications where such characteristics are desirable. From automotive components to aerospace structures, and even in the fields of civil engineering and consumer electronics, the implications of this study offer a pathway to the development of advanced composites that are both performance-driven and eco-friendly. This innovative use of natural fibers like KF in SMPCs paves the way for the creation of new materials that are more aligned with sustainability goals without compromising on the functional demands of modern technology.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nor Hanim Khiyon, Mohd Fadzil Arshad;* ***data collection:*** *Nor Hanim Khiyon, Nurshamimie Muhammad Fauzi;* ***analysis and interpretation of results:*** *Mohd Fadzil Arshad, Nor Hanim Khiyon, Mohd Khairul Kamarudin;* ***draft manuscript preparation:*** *Nor Hanim Khiyon, Mohd Fadzil Arshad. All authors reviewed the results and approved the final version of the manuscript.*

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