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Development of Environmentally Sustainable Epoxy Composites with Sunflower Seed Husk Fillers and Study on Thermal and Moisture Properties

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Abstract

This study examines the development and characterization of epoxy composites reinforced with sunflower seed husk (SSH) fillers fabricated using the compression molding technique. SSH fillers were categorized into two distinct particle size distributions, namely, 4–53 µm and 79–463 µm, and were incorporated into the epoxy matrix at varying volume fractions ranging from 5% to 20%. The water absorption behavior of the composites was assessed according to ASTM standards, and thermogravimetric analysis (TGA) was employed to evaluate their thermal stability. Furthermore, Grey Relational Analysis (GRA) was used to optimize the composite formulation based on both properties. Experimental findings indicate that an increase in the SSH filler content leads to higher water absorption, whereas thermal stability is generally enhanced with SSH reinforcement. The use of finer particles (4–53 µm) facilitates better filler-matrix interaction, effectively reducing moisture penetration while maintaining th ermal performance. In contrast, larger SSH fillers (79–463 µm) contributed to superior thermal resistance, but resulted in higher water absorption. Among the investigated formulations, the composite containing 15% fine SSH filler (S15) exhibited the most favorable balance between thermal stability and moisture resistance. These findings highlight the potential of SSH as an environmentally sustainable reinforcement material for epoxy-based composites. The enhanced thermal properties and controlled moisture absorption make these composites suitable for applications in automotive, marine, and construction industries, where durability and thermal efficiency are critical.

Keywords: Sunflower seed husk, epoxy, compression molding, water absorption, thermogravimetric analysis

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INTRODUCTION

Water absorption in polymer matrix composites (PMC) has a substantial impact on their durability and performance in various applications, as determined by the filler type, manufacturing technique, and environmental conditions. Studying water absorption, which normally follows Fickian diffusion, is critical to reduce its consequences. Moisture penetration, which is affected by temperature, pressure, and stress, can result in dimensional changes and mechanical deterioration [1,2]. Natural fibers, such as plantains, rice husks, and olive pits, absorb significant moisture because of their cellulose and hemicellulose content with hydroxyl groups that attract water. This high absorption accelerates polymer degradation, as biomass fillers such as reed and wheat straw promote enzymatic breakdown of poly (butylene adipate-co-terephthalate) composites, reducing their mechanical properties over time. Sodium polyacrylate and bentonite clay enhance water absorption in natural rubber, with sodium polyacrylate showing higher rates. The coconut shell exhibits the highest water absorption among natural fibers, driven by its cellulose content and structural properties [1,3-7]. Prolonged water exposure significantly reduced mechanical properties, such as flexural strength and interlaminar shear strength. Natural fibers, including banana, coconut shell, rambutan, and pineapple, exhibit varying water absorption levels, with fruit skin fibers, such as rambutan and pineapple, absorbing more moisture in thermoplastic polyurethane composites. This moisture absorption leads to changes in tensile and flexural properties [8-12]. The incorporation of fillers, such as halloysite nanotubes, cenosphere, and clamshell, improves water resistance and enhances mechanical properties by reinforcing the matrix and acting as barriers against moisture. The processing temperature and particle size of fillers also play a crucial role in water absorption rates, with higher temperatures and larger particle sizes typically reducing water uptake [9,13,14]. Different polymer compositions exhibit different water absorption characteristics. For example, polyamide composites reinforced with multiwalled carbon nanotubes (MWCNTs) show reduced water uptake compared to neat polymers [15]. Fillers such as nano-clay alter the absorption properties, reduce moisture ingress, and improve durability [16,17]. Carbon fillers reduce water absorption by up to 55.4%, thereby enhancing composite durability [18]. Surface modifications such as silane grafting on rice husks improve water resistance by maintaining higher contact angles after filler incorporation [19]. Smaller particle sizes led to the lowest moisture absorption in both the coir fiber-epoxy and groundnut shell-epoxy composites [20].

Thermogravimetric analysis (TGA) is a key method for assessing the thermal stability of polymer matrix composites by measuring the weight loss at specific temperatures, providing insights into their degradation behavior [21]. The presence of fillers enhances the thermal stability, as indicated by residual char, which improves the thermal performance compared to unfilled polymers [22,23,24]. Processing conditions such as extrusion speed have a minimal impact on the thermal stability of melt-processed composites [25]. Increasing the filler volume fraction and reducing the particle size improves the thermal conductivity of the composites [26]. Temperature significantly influences the degradation of recycled epoxy- and vinyl-ester-based carbon fiber composites under nitrogen [27]. Hybrid polymer composites exhibit higher thermal conductivity than single-filler systems, achieving synergistic effects and reducing costs [28]. A filler content of 15 % ensures optimal mechanical and thermal performance by promoting uniform dispersion and strong bonding with the matrix [29]. The thermal degradation temperature was increased to 430°C with 25% filler. The presence of lignin and cellulose in the filler enhanced thermal stability by delaying degradation [30]. TGA revealed higher degradation temperatures and increased residual percentages with the addition of cellulose micro filler (CMF) from peanut oil cake to the epoxy composites [31].

Grey Relational Analysis (GRA) is a robust methodology for evaluating multiple criteria decision-making problems. It has been widely employed in material selection [33] and in optimizing the proportion of reinforcement materials [34]. In addition, GRA has been utilized for optimizing auxetic fabric structures [35] and performing statistical analyses [34]. The integration of GRA with multiple linear regression (GRA-MLR) has proven to be an accurate and effective approach for investigating the mechanical properties based on input variables [36,37]. By converting multiple responses into a single performance characteristic, GRA enhances overall outcomes and facilitates decision making. Furthermore, it has been successfully applied to assess the feasibility of process parameters [38] and has contributed to improving the product quality by approximately 20% [39].

The novelty of this study lies in its investigation of the combined effects of sunflower seed husk (SSH) filler volume percentage and particle size on both the water absorption behavior and thermal stability of epoxy composites. Unlike previous studies that focused on conventional natural fillers, this study systematically evaluated SSH as a sustainable reinforcement material. Additionally, the use of Grey Relational Analysis (GRA) for statistical analysis in this context is novel, enabling a comprehensive performance evaluation of the composites based on multiple criteria.

Table 1. Sample Code, SSH Filler Particle Size Range and Composition of Composites.

Sample code	SSH filler particle size range (μm)	Composition (SSH filler : epoxy matrix) Vol. %
C0	Nil	0:100
S5	4 - 53	5:95
S10		10:90
S15		15:85
S20		20:80
L5	79 - 463	5:95
L10		10:90
L15		15:85
L20		20:80

MATERIALS AND METHODS

Materials

Epoxy resin (LY556 grade), a widely utilized thermosetting polymer, was combined with a low-viscosity hardener (HY951 grade) in a fixed weight ratio of 10:1 for the fabrication of natural composites [32,41]. The composite specimens were prepared through compression molding using a mild steel mold with dimensions of 210 × 170 × 3 mm, with sunflower seed husk (SSH) particles serving as the reinforcing filler [32]. The sample designations, filler particle size ranges, and corresponding compositions are detailed in Table 1. A systematic coding scheme was adopted to identify the samples, wherein "S" represents smaller particle size (4 to 53 µm), "L" represents larger particle size (79 to 463 µm), and the numeric (e.g., 5, 10, 15, 20) specifies the volume fraction of the filler. A control sample, consisting solely of epoxy resin without any filler, was labeled as C0.

Water Absorption Behavior

In accordance with ASTM D570-99, test specimens were prepared by cutting from the composite plates, followed by drying at 105°C for duration of 24 hours [29,30]. After drying, the specimens were submerged in bore water (TDS: 684 PPM, hardness: 440 PPM, pH: 6.85) at room temperature for 24 hours before testing [40]. The percentage of water absorption content was determined using the following Equation 1.

$$Water\ Absorption\ (\%) = \frac{W_{final} - W_{\text{initial}}}{W_{\text{initial}}} \times 100 \qquad (1)$$

Where, W_{final} = weight of the specimen after water immersion and $W_{initial}$ = weight of the dry specimen before immersion.

Thermogravimetric Analysis (TGA)

The thermal stability of the filler, matrix, and fabricated composite was assessed using the METTLER TOLEDO TGA 2 thermal analyzer. This instrument measures the rate of weight change and material behavior as a function of time or temperature in a controlled environment. The samples were placed in an alumina crucible and subjected to gradual heating from 30.0 °C to 600.0 °C at a heating rate of 10 °C/min [23,29,30,43]. To ensure a controlled environment, a continuous flow of pure nitrogen gas at a rate of 60 ml/min was maintained through the furnace.

Grey Relational Analysis (GRA)

The grey system theory, introduced by Deng in 1982, has been widely used in engineering for prediction and control as well as in social, economic, and agricultural systems [38], with Grey

Relational Analysis (GRA) serving as a statistical technique for optimizing multi-objective functions [34]. Table 2, which outline the steps and equations used in GRA.

Table 2. Steps and Equations Used in GRA [33,34,37,38].

Steps in GRA	**Equation**	**Nomenclature**
Data Pre-Processing and Normalizing	Expected data sequence is of the form "Higher-the-better" $x_i^*(k) = \frac{x_i^0(k) - \min x_i^0(k)}{\max x_i^0(k) - \min x_i^0(k)}$	$x_i^0(k)$ is the original sequence $x_i^*(k)$ the sequence after the data preprocessing $\max x_i^0(k)$ the largest value of $x_i^0(k)$ $\min x_i^0(k)$ simply the smallest value of $x_i^0(k)$
Grey Relational Coefficient (GRC)	$\xi_i(k) = \frac{\Delta_{min} + \zeta.\Delta_{max}}{\Delta_{0i}(k) + \zeta.\Delta_{max}}$	$\Delta_{0i}(k)$ is the deviation sequence of the reference sequence $\Delta_{0i}(k) = \lVert x_0^*(k) - x_i^*(k) \rVert$ $\Delta_{max} = \max_{\forall j \epsilon i} \max_{\forall k} \lVert x_0^*(k) - x_j^*(k) \rVert$ $\Delta_{min} = \min_{\forall j \epsilon i} \min_{\forall k} \lVert x_0^*(k) - x_j^*(k) \rVert$ ζ is distinguishing or identification coefficient [0,1] ζ = 0.5 is generally used
Grey Relational Grade (GRG)	$\gamma_i = \frac{1}{n}\sum_{k=1}^{n} w_i(k)\xi_i(k)$	$\xi_i(k)$ = Grey relational coefficient $w_i(k)$ = weight (1) n = Number of alternatives

RESULTS AND DISCUSSION

Water Absorption Behavior

The water absorption characteristics of the composites are illustrated in Figure 1. As the filler content was increased from 5% to 20%, water absorption demonstrated a progressive increase in both the L (L5 to L20) and S (S5 to S20) series. This trend is attributed to the higher volume fraction of hydrophilic filler materials, which naturally increase water uptake [30,42]. The pure epoxy resin (C0) exhibited the lowest water absorption (0.453%), consistent with its hydrophobic nature [30].

Composites containing larger particle sizes (79 to 463 µm) exhibited higher water absorption compared to those with smaller particle sizes [20]. For instance, at 20% filler content, the L20 sample absorbed 1.1096%, while the S20 sample absorbed 0.79361%. This trend can be attributed to the less efficient packing of larger particles, which may create microvoids, thereby increasing the pathways for water ingress [29]. Composites with smaller particles (4 to 53 µm) demonstrated lower water absorption values due to better encapsulation by the epoxy matrix [29], which reduces water penetration pathways despite their higher surface area.

The high total dissolved solids (TDS), hardness, and slightly acidic pH of bore water amplify water absorption, especially in composites containing larger filler particles. The S-series composites consistently exhibited lower water absorption than the L-series, with the percentage difference increasing from 12.81% at 5% filler content to 39.85% at 20% filler content (refer to Table 3). Sample S5 showed a 32.02% increase in water absorption compared to the pure epoxy control (C0), indicating the influence of filler hydrophilicity [30], even when effectively encapsulated by the matrix.

Thermogravimetric Analysis (TGA)

The thermogravimetric analysis (TGA) results (Figure 2.) revealed distinct differences in the thermal stability and residual char formation of the SSH filler, neat epoxy (C0), and SSH filler–epoxy composites (S and L series). The SSH filler displayed the highest residual char content, highlighting its intrinsic thermal stability. In contrast, the neat epoxy (C0) showed the lowest residual char value of 6.23%, highlighting its complete degradation at elevated temperatures. For the composites with smaller SSH filler particle sizes (4 to 53 µm), the residual char increased with filler content, peaking at 21.00% for S15 before decreasing to 13.39% for S20, possibly due to filler agglomeration or a reduction in effective interaction between the filler and the epoxy matrix. Composites with larger SSH filler particle sizes (79 to 463 µm) demonstrated generally higher residual char percentages, with the maximum value of 22.83% observed for L10. This indicates that larger particles contribute more effectively to the thermal stability of the composites, particularly at moderate filler content levels.

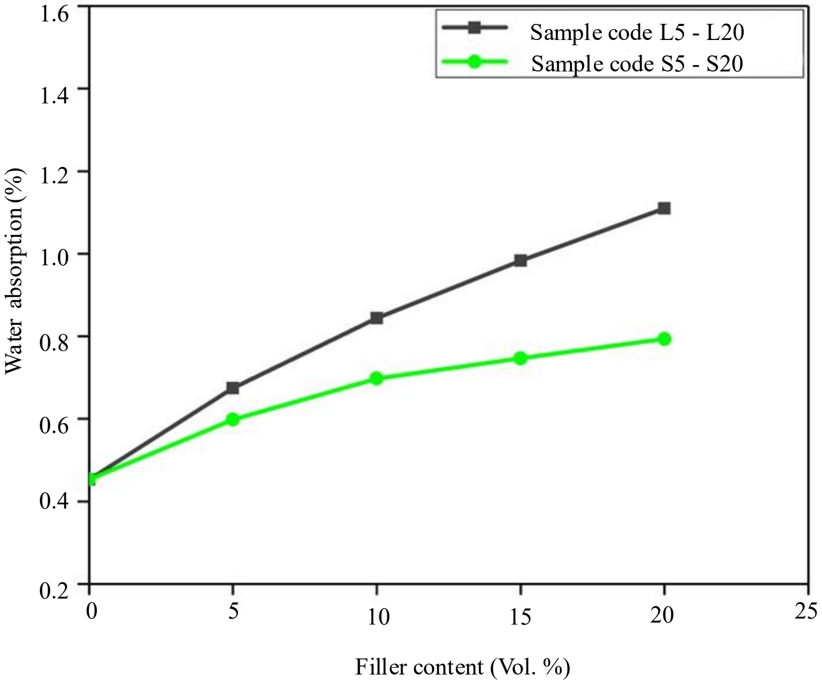

Figure 1. Water absorption of SSH Filler – epoxy composites.

Table 3. Water Absorption Percentage Difference between S and L Series Sample Code.

Filler content (Vol. %)	Water absorption (%) of S series sample code	Water absorption (%) of L series sample code	Percentage difference (%)
5	0.59804	0.6745	12.81
10	0.69739	0.8441	21.04
15	0.74655	0.9829	31.65
20	0.79361	1.1096	39.85

Comparing the SSH filler to both S and L series composites, it is evident that the residual char of the composites is significantly influenced by the filler particle size and volume percentage. While the smaller particle size composites (S series) exhibited better thermal performance at moderate filler content (e.g., S15 with 21.00% residual char), the larger particle size composites (L series) outperformed at moderate filler content levels (e.g., L10 with 22.83%). Across all samples, the decomposition followed a two-stage pattern: initial weight loss (50 to 250 °C) due to moisture loss [24,31] and decomposition of low-molecular-weight compounds, followed by major degradation of the epoxy matrix and SSH filler (300 to 500 °C). The incorporation of SSH fillers enhanced the thermal stability of the composites compared to neat epoxy (C0), with the SSH filler itself setting a benchmark for the highest thermal resistance [24].

The impact of the filler particle size on the heat resistance of the composite was significant. Smaller particles (P2) enhance thermal stability at 5%, 15%, and 20% filler loadings owing to their higher surface-area-to-volume ratio, improved dispersion, filler-matrix bonding, and char formation for better heat dissipation. However, at 10% loading, larger particles (P1) exhibited the highest residual char, forming a stable char layer that acts as a thermal barrier, slowing degradation. The filler-to-matrix ratio also influences thermal conductivity, where moderate filler content (10–15%) enhances dispersion and bonding, with smaller particles aiding heat transfer and larger particles stabilizing char at 10% loading. Excessive filler (>15%) can lead to agglomeration, which weakens adhesion and creates thermal barriers. Optimizing the filler-to-matrix ratio is key to balancing thermal resistance and conductivity in SSH-epoxy composites.

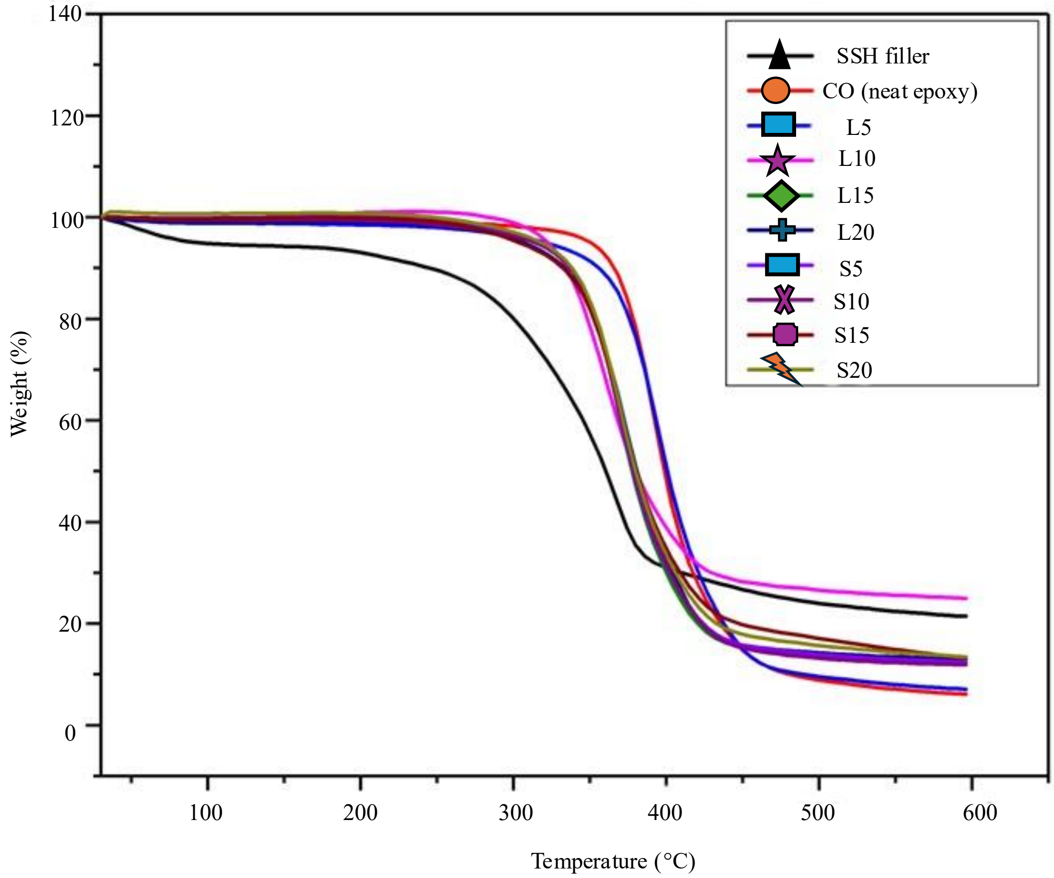

Figure 2. TGA curves of SSH filler and fabricated composites (C0, S5–S20, and L5–L20).

Grey Relational Analysis (GRA)

Table 4 outlines the data pre-processing, normalization, and deviation sequence of water absorption and TGA residual char for sample codes C0–L20. The results indicate that C0 had the best water absorption resistance, with the lowest deviation sequence (0.000), followed by S5 (0.221), while L20 had the highest deviation sequence (1.000), indicating the poorest water absorption resistance. Similarly, L10 had the highest TGA Residual Char (%) with the lowest deviation sequence (0.000), making it the most thermally stable sample. S15 also exhibited high TGA Residual Char (%) with a relatively low deviation sequence (0.110), ranking second in thermal stability. In contrast, C0 had the lowest residual char (%) and the highest deviation sequence (1.000), indicating the poorest thermal stability. The trends observed in Figures 1 and 2 strongly support the deviation sequence results presented in Table 4, further validating the findings on the water absorption behavior and thermal stability.

The Grey Relational Grade (GRG) is obtained by averaging the GRC values for both criteria (water absorption and TGA residual char). This represents the overall performance of each sample, with a higher GRG value indicating better overall properties. Table 5 outlines the GRG values and their respective rankings. L10 had the highest GRG (0.162), making it the best overall sample (Rank 1), mainly because of its superior thermal stability. S15 ranked second (GRG = 0.150), as it performed well in terms of both water absorption and thermal stability. C0 ranked third (GRG = 0.148), with excellent water absorption resistance but weak thermal stability. L20 has the lowest GRG (0.088), making it the weakest overall sample (rank 9) because of its poor performance in terms of both water absorption resistance and thermal stability. The trends observed in Figures 1 and 2 strongly support the GRG results presented in Table 5, further validating the findings on the water absorption behavior and thermal stability.

Table 4. Normalization and Deviation Sequence of Water Absorption and TGA Residual Char of SSH Filler-Epoxy Composites.

Sample code	Water absorption (%)	TGA residual char (%)	Normalization		Deviation sequence	
			Water absorption (%)	*TGA residual char (%)*	*Water absorption (%)*	*TGA residual char (%)*
C0	0.453	6.23	1.000	0.000	0.000	1.000
S5	0.598	12.54	0.779	0.380	0.221	0.620
S10	0.697	11.90	0.628	0.342	0.372	0.658
S15	0.747	21.00	0.553	0.890	0.447	0.110
S20	0.794	13.39	0.481	0.431	0.519	0.569
L5	0.674	7.32	0.663	0.065	0.337	0.935
L10	0.844	22.83	0.404	1.000	0.596	0.000
L15	0.983	12.41	0.193	0.372	0.807	0.628
L20	1.110	13.19	0.000	0.419	1.000	0.581

Table 5. GRC and GRG of water absorption and TGA residual char of SSH Filler-epoxy composites.

Sample code	GRC		GRG	Rank
	Water absorption (%)	*TGA residual char (%)*		
C0	1.000	0.333	0.148	3
S5	0.694	0.446	0.127	4
S10	0.573	0.432	0.112	5
S15	0.528	0.819	0.150	2
S20	0.491	0.468	0.107	6
L5	0.597	0.349	0.105	7
L10	0.456	1.000	0.162	1
L15	0.383	0.443	0.092	8
L20	0.333	0.463	0.088	9

Scalability and Cost Considerations

The scalability of SSH-epoxy composites for commercial production depends on the availability of the raw materials, processing adaptability, and cost-effectiveness. SSH, an abundant agricultural byproduct, provides a low-cost and sustainable alternative to synthetic fillers. The compression molding process used in this study is highly scalable and can be adapted for automated production lines with minimal modifications. The primary cost implications involve preprocessing steps such as drying, size reduction, and uniform dispersion, but these costs remain lower than conventional fillers like SiC, $CaCO_3$, and talcum powder, which require high-energy processing. Additionally, SSH-filled composites offer comparable thermal stability while reducing the environmental impact, making them an economically viable option for large-scale applications in the automotive, marine, and construction industries.

CONCLUSIONS

This study demonstrated that incorporating sunflower seed husk (SSH) fillers into epoxy composites significantly influences their thermal stability and water absorption, with effects varying based on particle size and filler content. Neat epoxy (C0) exhibited the lowest water absorption (0.453%) and residual char (6.23%), indicating limited thermal stability. Composites with smaller SSH fillers (4–53 µm) improved thermal performance, with S15 achieving the highest residual char (21.00%), though water absorption increased to 0.794% at higher filler contents. Composites with larger SSH fillers (79–463 µm) exhibited superior thermal stability, with L10 achieving the highest residual char (22.83%);

however, these composites showed increased moisture uptake, reaching 1.110% in L20, likely due to weaker interfacial bonding.

Grey Relational Analysis (GRA) further validated these findings, ranking L10 as the top-performing composite (Rank 1), followed by S15 (Rank 2). Despite its superior thermal stability, L10's higher water absorption (0.844%) makes it less suitable for moisture-sensitive applications. S15 emerged as the optimal composite, balancing thermal stability (21.00% residual char) and lower water absorption (0.747%), making it particularly viable for automotive, marine, and construction applications, where both thermal performance and durability against moisture exposure are critical.

The use of SSH fillers from renewable waste enhances composite sustainability while offering a cost-effective alternative to synthetic reinforcements. Future work should focus on surface treatments of fillers to further enhance moisture resistance and expand the usability of SSH-epoxy composites in broader engineering applications.

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