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Green Reinforcement of Recycled Mild Steel with Date Palm Seed Particles for Enhanced Dynamic Properties in Automotive Applications

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ABSTRACT

As the need arises to replace hazardous chemicals such as asbestos used in brake pad production, recent developments have shifted towards a more sustainable replacement with available and naturally derived materials. This study investigates the dimensional and dynamic mechanical properties of brake pad composites reinforced with metal chips and date palm seed (DPS) particles. Three samples (A, B, and C) with varying DPS particle compositions (40%, 35%, and 30%) were produced and analysed. Dimensional stability analysis showed that Sample A exhibited shrinkage of -1.8%, demonstrating superior resistance to humidity-induced deformation compared to the dimensional growth of 9% and 12.8% in Samples B and C, respectively. Dynamic mechanical analysis (DMA) results revealed a decrease in storage modulus with increasing temperature, consistent with the glass-to-rubber transition. Sample B had the highest storage modulus across all frequencies, indicating superior stiffness. However, Sample A demonstrated better damping capacity (0.325 tan δ) and effective energy dissipation, attributed to strong filler-matrix interactions. Creep analysis further validated Samples A and B's enhanced thermal stability and deformation resistance. The study highlights Sample A as the most balanced composite, offering superior dimensional stability and damping properties.

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INTRODUCTION

The automobile sector is a typical example of how even modest actions can make a big difference and make the world greener. Although the automobile sector's sustainability is the twenty-first century's dominant subject, it has not been widely adopted in all the components [1], [2]. As a major player in the world economy, the automobile sector is under more pressure to implement sustainable practices. Political mandates, changing consumer tastes, and internal demands for ecologically friendly manufacturing practices are the sources of this pressure [3]. Specifically, there is a great chance to incorporate sustainable materials in manufacturing all vehicle parts, including braking systems. This is more desirable in modern high-performance car braking systems incorporating the newest technology developments [4].

Brake pads, essential parts of the braking system, have traditionally relied on asbestos fibres for reinforcement despite well-documented health concerns associated with their carcinogenic nature [5]. The automotive industry has sought alternatives to asbestos, with researchers exploring agricultural waste and natural fibres as potential replacements. Brake pad materials are categorised into metallic, semi-metallic, and non-asbestos organic (NAO) types, each offering unique compositions and performance attributes. Metallic brake pads predominantly feature fibres such as copper and steel, while semi-metallic variants combine organic compounds with metals like iron powder and ceramics. NAO brake pads, known for their eco-friendly nature, typically use modified resins and natural fibres like rubber [6].

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The composition of brake pads is a critical factor, as slight variations can significantly affect braking efficiency. Hence, various binders, reinforcements, fillers, and frictional additives optimise brake pad performance under thermal and mechanical stresses [5]. Phenolic resins, widely used as binders, offer good heat resistance and cost-efficiency but require modifications for high-performance applications. Epoxy resins, another common binder, enhance structural stability when reinforced with polymers or ceramics [7]. Reinforcements like carbon fibres and ceramics provide mechanical strength, thermal resilience, and improved braking performance [6]. Ceramics, such as potassium titanates, are particularly valued for their high-temperature stability but remain limited by brittleness [8]. Organic and inorganic fillers support the brake pad matrix and improve manufacturability while controlling costs. With the reinforcement of the brake pad matrix with fillers, it is essential to evaluate the performance, with most studies focusing on the sliding wear [6] properties and dynamic-thermal properties getting little attention.

Among the few studies that evaluated the dynamic-thermal properties of filler-reinforced brake pads, [9] compared two modified resin binders, resin A (binary structure) and resin B (ternary structure), in brake pads, highlighting resin B's superior toughness, impact strength, and braking stability due to its integrated epoxy, PF, and rubber components. Dynamic Mechanical Analysis (DMA) revealed that resin B had a higher tan δ peak, indicating better damping ability and more stable braking performance at high speeds. [10], investigated the impact of carbon fibre content (0–4 vol%) on the dynamic properties of phenolic-based brake friction composites. DMA analysis revealed that carbon fibre incorporation increased the storage modulus (E'), enhancing rigidity and reducing the damping factor (tan δ), which indicates restricted polymer chain mobility and improved stiffness. Also, [11] examine the impact of siloxane-treated alumina particles on the thermo-mechanical properties of NAO and Cu-free brake pads, focusing on filler-matrix adhesion. DMA results reveal that siloxane-treated brake pads (At) exhibit improved storage modulus (E') and reduced deformation, enhancing stiffness and reducing actual contact area during braking. Having established the improvement in the dynamic properties of the brake-pad matrix, more studies on the integration of sustainability through green reinforcement are essential.

In this study, attention is shifted from resin and reinforcement with fibre-based materials to the circular use of metal (mild-steel) chips and enhancing these properties through reinforcement with an indigenous fruit by-product (date palm). Metal chips, by-products of machining processes, offer significant potential for promoting sustainability through recycling and reuse [12]. Advancements in solid-state recycling methods bypass the energy-intensive remelting phase, reducing material losses and hazardous emissions and preserving the intrinsic properties of the metal, making it a greener alternative for manufacturing applications [13]. This foundation of sustainable metal recycling aligns with developing green reinforcement strategies for automobile applications, especially brake pads. Improving the circular use of mild-steel chips involves using date palm seeds (DPSs). DPSs, by-products of date palm fruits common in Africa and Asia, exhibit fibrous structures with a central lumen, enhancing their ability to absorb frictional forces and making them ideal for braking systems [14]. Studies have demonstrated the mechanical and thermal stability of DPSs [15], [16], further highlighting their potential in brake pad manufacturing while promoting eco-friendly and sustainable solutions. To this end, this study aims to consider the novel reinforcement of recycled mild steel with DFSs as green reinforcement for brake pad application and analysis of the resulting dynamic properties.

EXPERIMENTAL METHODS

Development of Brake Pad

The development of brake pads involved using locally sourced materials, including irregularly shaped metal chips, fresh DPSs, pure aluminium oxide powder, barium sulfate powder, and graphite powder. As illustrated in the schematic in Figure 1, the DPSs were prepared by

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washing, sun-drying, and grinding to reduce their density and enable particle production. To remove excess oil, the ground DPSs were soaked in a NaOH solution for one hour, thoroughly washed with water, and sun-dried for three weeks. It was then re-grounded and sieved using an aperture of 210 µm.

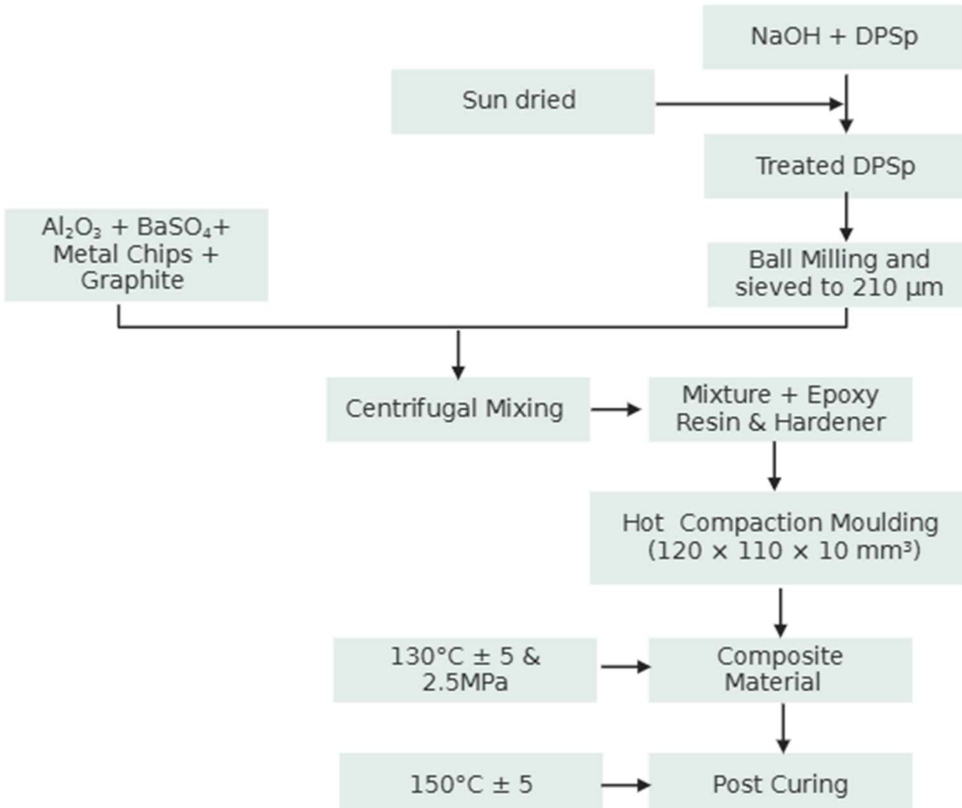

Figure 1: Brake pad development process flow chart

These materials were blended according to specific formulations (A, B, and C) in Table 1, with DPSs contributing 30–40% of the composition. At the same time, epoxy resin, hardener (Diethylenetriamine), graphite, metal chips, aluminium oxide, and barium sulfate were added in varying percentages. The components were mixed thoroughly with the epoxy resin and hardener for uniformity before being poured into a preheated hydraulic press mould (120 × 110 × 10 mm³) on a metal backing plate. The mixture was cured at 130 °C ± 5 under a compounding pressure of 2.5 MPa, followed by post-curing in an oven at 150 °C ± 5 for 120 minutes. The samples were left at ambient temperature for 14 days for optimal mechanical strength. This development process ensures the regulation of hydrophilic properties and moisture content, which are critical for enhancing the performance and durability of the brake pads.

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Table 1: Brake pad composite sample formulations

S/N	Ingredients	A	B	C
	DPSs particle	40	35	30
2	Epoxy resin	20	20	20
3	Hardener	10	10	10
4	Graphite	10	15	15
5	Metal chips	10	10	15
6	Aluminium oxide	5	5	5
7	Barium sulphate	5	5	5

Dimensional Stability Test

The percentage swell of friction composites is measured under varying temperature and humidity conditions to evaluate dimensional stability. Dimensional stability is vital in brake pad composites as it measures the ability of the material to retain its original dimensions when exposed to external factors such as temperature changes and humidity variations to help capture any dimensional changes, including expansion or contraction, that can negatively affect braking performance, increase wear, and lead to system failures [17]. Samples were subjected to a temperature range from sub-zero (-20 °C) to elevated heat (300 °C) and were also placed in humidity-controlled chambers to simulate environmental conditions such as heavy rain, high humidity, and arid climates. The swell of each sample was calculated using Equation (1).

$$Swell\ (\%) = \frac{L_i - L_f}{L_i} \times 100 \qquad (1)$$

L_i is the sample's initial dimension, and L_f is the sample's final dimension after swell testing.

Dynamic Mechanical Analysis Measurement

To evaluate a material's energy dissipation effectiveness, as a measurement of its viscoelasticity, the loss modulus, storage modulus, change in length (dL) and loss factor (tan δ) were analysed using Dynamic Mechanical Thermal Analysis (DMTA). The experiments were conducted at 2.5 Hz, 5 Hz, and 10 Hz under a dynamic load of 2000 × 10^{-3} N. The test temperature range was from 25.0°C to 145.0°C, with a heating rate of 6.0 K/min, to aid in understanding the material's energy dynamics under varying conditions. The DMTA machine used for these analyses is shown in Figure 2.

Figure 2. DMA machine

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RESULTS AND DISCUSSION

Dimension Analysis

The dimensional stability of the brake pad samples, as indicated by the swell growth results and shown in Figure 3, shows that Sample A demonstrates a shrinkage of 1.8%. At the same time, Samples B and C exhibit positive dimensional growth, with values of 9% and 12.8%, respectively. The shrinkage observed in Sample A suggests a high level of dimensional stability, as it can resist expansion when exposed to humidity, maintaining its structural integrity. This characteristic is crucial for brake pads, as excessive swelling can compromise their performance, particularly in fit and contact with braking surfaces [5]. In contrast, Samples B and C exhibit potential issues in high-humidity environments as the material expands with moisture absorption. The higher swelling in Sample C (12.8%) could be attributed to a higher proportion of graphite, which may absorb moisture more readily [18]. While this increase in size could enhance the material's ability to absorb shock and impact energy, it also raises concerns about long-term dimensional stability. In automotive applications, where consistent performance is paramount, excessive swelling could affect braking efficiency and lead to irregular wear patterns [19].

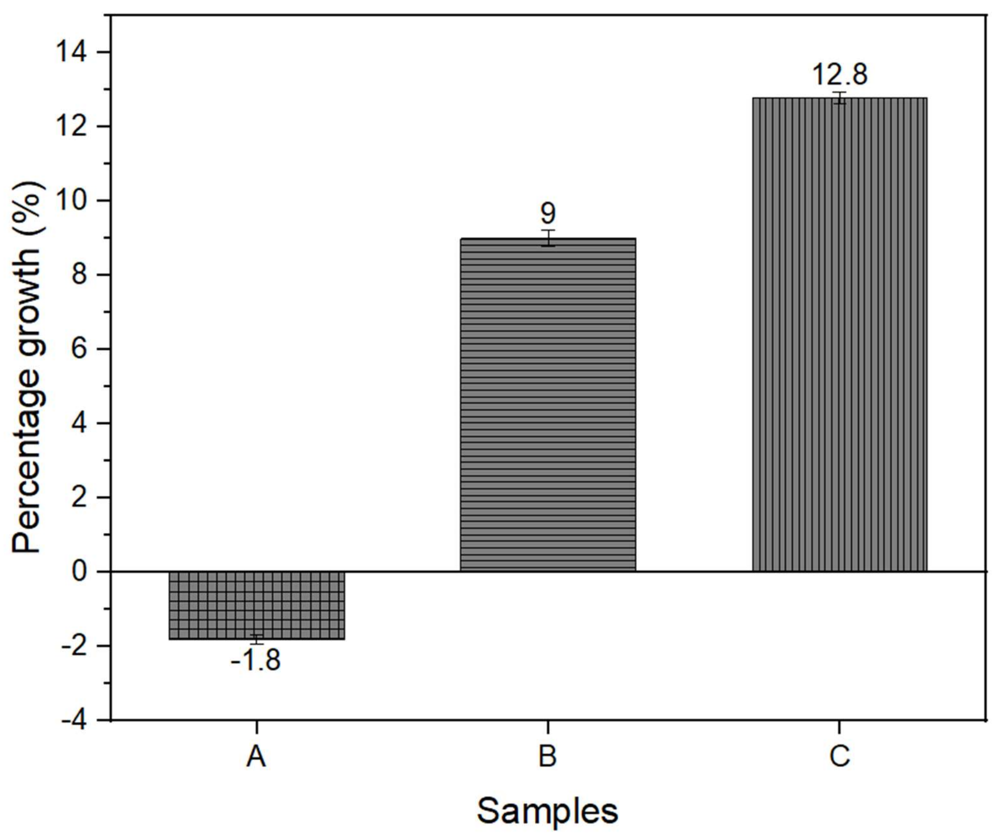

Figure 3. Swell growth of the developed brake pad composites

Comparing the results in Figure 3 to those of brake pads developed using palm kernel shell (PKS) by [20], where the reported swell growth was within the range of 0.17% and 0.39%,

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it was reported that an increase in the reinforcement, the water absorption of the brake pad reduced. Sample A of this study exhibits superior dimensional stability under varying humidity conditions, suggesting that the DPS contribute to better moisture resistance as observed in other fibre reinforcement [21], [22].

Dynamic Mechanical Properties

The DMA results of Samples A, B, and C, as given in Figure 4(a), show that all the developed metal-chip/DPS exhibited a general decrease in storage modulus (E') with increasing temperature, consistent with the typical behaviour of polymer composites transitioning from a glassy to a rubbery state [23]. Sample B consistently demonstrated the highest storage modulus across all frequencies, followed by Samples A and C. This trend suggests that Sample B possesses superior stiffness, associated with the effect of the higher composition graphite in the composite. The loss modulus (E") behaviour, given in Figure 4(b), further elucidates the internal friction and energy dissipation mechanisms within the samples. Samples A and B exhibited pronounced peaks in the loss modulus curves, signalling the glass transition temperature (T_g) where polymer chains gain mobility and internal friction peaks. Conversely, Sample C displayed a continuous decline in loss modulus without distinct peaks, implying a more gradual transition or possibly weaker molecular interactions within its matrix [24]. This observation could be attributed to suboptimal filler distribution in Sample C (30%).

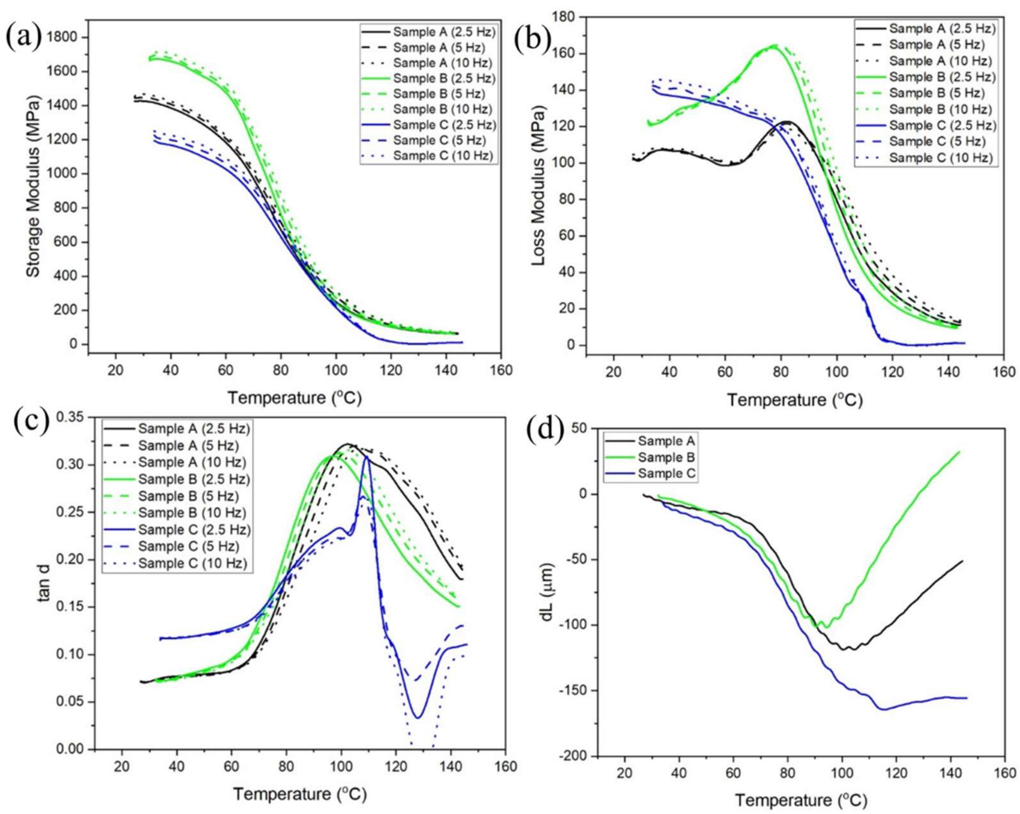

Figure 4: Variation of (a) storage modulus, (b) loss modulus, (c) tan δ, and (d) dL of the developed brake pad composites at 2.5 Hz, 5 Hz and 10 Hz frequency

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The tan δ curves, given in Figure 4(c), show a consistent increase in damping capacity was observed across all samples as the temperature increased. The highest tan δ peaks were recorded at 2.5 Hz for Samples A and C, while Sample B peaked at 10 Hz, indicating frequency-dependent damping behaviour, as indicated in Table 2. The damping behaviour of Sample A, characterised by a sharp tan δ peak, points to well-dispersed fillers and strong interfacial bonding, which enhances energy dissipation [25]. In contrast, the subdued damping peaks in Sample C could indicate poor filler-matrix interactions, as observed in the dimension analysis result in Figure 3, reducing its ability to dissipate mechanical energy effectively. Comparative analysis with existing studies and the creep result in Figure 4(d) supports these observations. Samples A and B demonstrated resistance to creep, whereas Sample C showed a continuous and sharp decrease in deflection rates as the transition temperature was approached. Notably, Samples A and B experienced a clear transition between 80°C and 120°C, reaching their respective peak deflections of -50 µm and 35 µm. This highlights the samples' varying creep resistance and deformation behaviours under thermal stress. Previous studies have shown that incorporating reinforcing fibres in polymer composites increases stiffness while potentially decreasing damping capacity due to restricted polymer chain mobility [23]. Similar trends were reported by [24], where composites with optimal filler dispersion exhibited higher storage moduli and distinct loss modulus peaks, reflecting effective energy dissipation mechanisms.

Table 2: Maximum loss factor and maximum glass transition temperature of the composites at 2.5Hz, 5.0 Hz and 10 Hz

	Minimum peak Tg (°C)			Maximum peak (tan δ) 10^{-3}		
Specimen	2.5 Hz	5 Hz	10 Hz	2.5 Hz	5 Hz	10 Hz
Sample A	120	199	197	0.325	0.324	0.323
Sample B	162	163	164	0.30	0.323	0.324
Sample C	140	141	142	0.30	0.27	0.27

CONCLUSION

The study evaluated the dynamic properties of metal-chip/DPS-based brake pad composites and also evaluated the static mechanical and dimension stability, with the following key findings:

1. Sample A demonstrated superior dimensional stability with a shrinkage of -1.8%, compared to 9% and 12.8% dimensional growth for Samples B and C, respectively.
2. Sample B displayed the highest storage modulus across all frequencies, reaching 162°C as the peak glass transition temperature at 2.5 Hz, indicating better stiffness. However, Sample A showed higher damping capacity (0.325 tan δ) and better filler-matrix interaction, resulting in effective energy dissipation and creep resistance.

These findings suggest Sample A's suitability as the optimal choice for brake pad applications due to its balanced mechanical and dimensional properties. Future studies should optimise the filler composition mathematically by fine-tuning the percentage of graphite and DPS particles. Additionally, investigating the long-term thermal and wear behaviours under actual operational conditions will provide more insights into their practical applications.

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