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Review Article

Dynamic mechanical properties of natural fiber reinforced hybrid polymer composites: a review

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ABSTRACT

The concerning waste management issue of natural fibers and the downsides of synthetic fibers have governed natural fibers' utilization as reinforcements in composites. Incorporating a single type of reinforcing fiber does not inevitably produce composites that meet exceptional quality standards, particularly in dynamic mechanical properties. Various studies have demonstrated excellent properties of natural fiber reinforced hybrid composites. Accordingly, this paper aims to review research related to natural fiber reinforced hybrid composites that emphasize the dynamic mechanical properties. A summary for each type of hybrid composites, including thermoset and thermoplastic polymers, biopolymers, nanocomposites, and bionanocomposites was provided. The variables of relevance in this overview are the loss modulus, storage modulus, damping factor, and glass transition temperature. Overall, the reviewed works revealed that lignocellulosic fibers are extensively used to reinforce composites. Nearly all hybridization of multiple reinforcing fibers had synergistic influences on the hybrid composites' dynamic mechanical properties. However, there are several cases whereby the addition of hybrid reinforcing particles leads

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to a detrimental effect on the composites' quality. There is a limitless possibility for further improvements of natural fiber reinforced hybrid composites.

1. Introduction

The utilization of natural fibers is emerging rapidly, owing to their biodegradability, abundance, reasonable cost, and minimal energy usage in processing [1,2]. The outstanding characteristics of natural fibers, such as high specific modulus, lightweight, and excellent resistance to wear and tear, have given rise to their utilization as reinforcing agents in polymers composites, including thermoset and thermoplastics [3,4]. Generally, natural fibers can be categorized into plant, animal, and mineral fibers. This review principally emphasizes natural fibers originating from plants as this source of fiber is recently becoming the center of attention of the research society and the industry [5]. Nonetheless, natural fibers of animal and mineral origins are not disregarded as this review aims to provide a holistic view of natural fiber reinforced polymer composites.

Bamboo, sugar palm, bagasse, wood, kenaf, jute, hemp, pineapple, and cotton fibers have been extensively used in various polymers, aiming to enhance the composites' properties, particularly from the environmental and biodegradability standpoints [5–8]. These fibers are similar in terms of their chemical constituents, namely cellulose, hemicellulose, and lignin, yet marginally different in terms of their physical and mechanical properties due to their variation of fiber diameter, length, and specific gravity [9]. Despite the extensive exploration of natural fibers as polymer reinforcing materials, various other natural fibers such as pines, banana, abaca, and ramie fibers with similar chemical composition are yet to be explored comprehensively [5]. The paucity of evidence on these fibers as composites reinforcing agents suggests that there is room for further exploration in the research field, and there are boundless possibilities to bring this technology to a greater extent.

Hybrid composites are fabricated using more than one reinforcing agents in the same polymer matrix, intending to boost the composite properties. Combining several reinforcing materials makes it possible to get either a synergistic or antagonistic effect of the materials [10]. In addition, the properties can also be tuned by balancing the positive aspects of one material with the downsides of another [11]. The incorporation of multiple reinforcements in a matrix offers a broader extent of properties that are unattainable by single fiber reinforced composites. Accordingly, the hybridization of natural and synthetic origins reinforcing agents has gained a considerable attention from the scientific community [12]. In the manufacturing and production industries, the use of synthetic fibre composites is inevitable because their qualities are acknowledged to be superior to those of natural fibre. High-performance polymer matrix composite products, such as fibre reinforced plastic tanks, aircraft components, vehicle parts, and building panels, have all been made with synthetic fibre. Glass, carbon, and aramid are some of the most regularly used synthetic fibres in the composites industry [13].

Upon fabricating reinforced composites, the performance should be tested to ensure it achieves the specified industrial standards [14]. It is crucial to check and validate the composites' performances and characteristics, particularly when subjected to periodic stress such as damping. Dynamic mechanical analysis (DMA) is a convenient analytical method to analyze a composite material's properties as a function of time, temperature, or frequency. This technique has been used for the last few decades, and until today, it is still considered an essential method in the material engineering field, owing to its outstanding detection sensitivity [15]. Over the past few years, several articles had comprehensively reviewed the dynamic mechanical properties of natural fiber composites [16–19]. However, none of the review focused solely on natural fiber reinforced hybrid polymer composites. In this review, the dynamic mechanical properties of natural fiber reinforced hybrid polymer composites are reviewed.

2. Dynamic mechanical analysis (DMA)

DMA is an analytical method that evaluates the viscoelastic properties of materials, particularly polymers and composites, by measuring the stress or strain resulting from a dynamically varying stress or strain subjected to the sample. DMA is also termed as dynamic mechanical thermal analysis (DMTA) when the temperature response is analyzed. The typical DMA parameters of interest for composite materials are storage modulus, loss modulus, and damping factor as a function of time, temperature, or frequency [20]. The storage modulus (E′) or dynamic modulus describes a material's stiffness and elastic behavior. This parameter theoretically allies with Young's modulus, but it should be emphasized that they are not identical [20]. On the other hand, the loss modulus (E″) or dynamic loss modulus portrays a material's viscous response and is associated with the sample's heat energy dissipation. This variable is usually linked to the material's "internal friction". It is primarily affected by the molecular arrangements, heterogeneities, motions, and phase transition processes [21]. The storage modulus and loss modulus concept can be explained by a bouncing ball's behavior, which dissipates and stores energy, as illustrated in Fig. 1.

The damping factor (tan δ) or loss tangent is one of the vital variable obtained from DMA, expressed as the ratio of the loss modulus and the storage modulus (tan δ = E"/E′), as depicted in Fig. 2 [22]. The resulting parameter of loss modulus and storage modulus is termed as complex modulus (E*), which describes a material's resistance to deformation. A highly

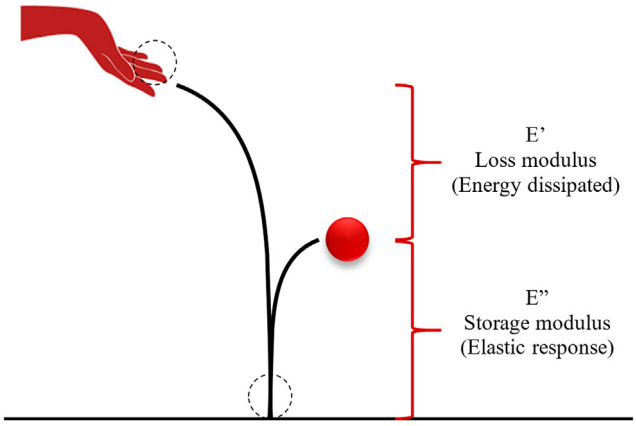


Fig. 1 – Representation of the loss modulus and storage modulus.

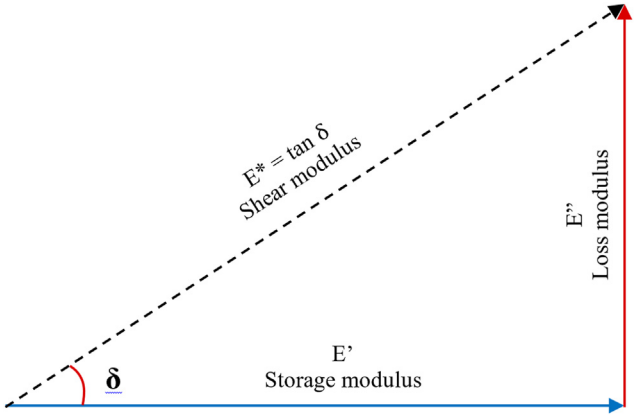


Fig. 2 – Correlation between storage modulus, loss modulus, shear modulus, and damping factor.

elastic material will have a low value of damping factor as it can easily deform when subjected to external force, whereas vice versa for a non-elastic or rigid material. From the viewpoint of fiber reinforced polymer composites, greater interfacial bonding between the fiber and matrix components will result in a higher value of damping factor [21]. This characteristic is caused by the lower energy loss due to internal molecular motion as the movement of interlocked molecular structures are restricted; hence, yielding higher storage modulus to loss modulus ratio [23].

2.1. *Glass transition temperature (T_g)*

The measurement of glass transition temperature (T_g) is one of the principal applications of DMA. The T_g is a temperature at which a thermosetting and amorphous polymer undergoes a transition from a rigid structure to a rubbery state. It should be noted that T_g is not the same as melting temperature (T_m), a temperature at which a material starts to melt, which often occurs for polymers with a crystalline structure. The T_g is highly dependent on the crosslink density of a polymer [24]. For thermosets, a high crosslink density restricts the molecular motions, resulting in higher energy requirement to enable segmental mobility during glass transition [25]. Consequently, this behavior yields a higher modulus, lower damping factor, and higher T_g. The crosslink density effect is distinctive in the rubbery and transition state of a material but not in the glassy region. Hence, the modulus values of lightly and highly cross-linked polymers are similar at the DMA curve's glassy region. In contrast, at the curve's rubbery and transition regions, highly cross-linked polymer will show much higher modulus values, portraying tighter molecular arrangement and higher stiffness. Fig. 3 depicts a comparison of composites with high and low crosslink densities. For amorphous thermoplastic materials with flexible backbone will have lower T_g values, whereas plastic materials whose molecular structure is stiff, rigid, and inflexible show higher T_g values [26]. Additionally, the degree of branching (DB) of polymers also affects the T_g in a way that a higher DB of a polymer will result in a higher T_g [27].

A polymer that comprises both amorphous and crystalline structure will have both T_g and T_m, with T_g that is always lower than T_m. The capability of polymers to exist in two different functional states enables them to have a wide range of applications. Some polymers such as polystyrene and polymethyl methacrylate are used below their T_g in their rigid or brittle state. In this state, the molecular bonds are interlocked and thus restricts molecular motion [15]. The hardness of the materials makes them suitable for packaging, furniture, and laboratory equipment. On the other hand, polyisoprene and polyisobutylene are used in their rubbery state above their T_g. In contrast to the rigid phase, the polymer's molecular chains are mobilized in the rubbery state, resulting in high flexibility and softness [15]. This property is useful for various applications such as adhesives, sealants, and lubricants. In terms of the T_g determination, the DMA technique is superior to other methods, with up to 100 times higher sensitivity than the differential scanning calorimetry (DSC) method [28]. The determination of T_g via DMA can be done using three methods; the temperature at (1) the onset of E′, (2) the maximum of E*, and (3) the highest point of tan δ. Fig. 4 shows a sample of T_g determination using the storage modulus curve of DMA.

3. General characteristic and classification of natural fiber reinforced hybrid polymer composites

Fiber reinforced hybrid polymer composites are extensively used as substitutes of metallic materials for automotive, building, and aerospace applications [29–31]. A combination of two or more reinforcing materials in a single polymer matrix forms hybrid composites that may have greater stiffness and strength compared to the individual components. The hybrid reinforcement may vary depending on its source (natural/natural and natural/synthetic) or scale (micro/nano), resulting in a wide diversity in properties [32]. Theoretically, the properties of hybrid composites should obey the rule of hybrid mixtures if no chemical or physical interaction occurs between the reinforcing fibers [33]. The rule is expressed as follows;

$$P_H = P_1V_1 + P_2V_2 \quad (1)$$

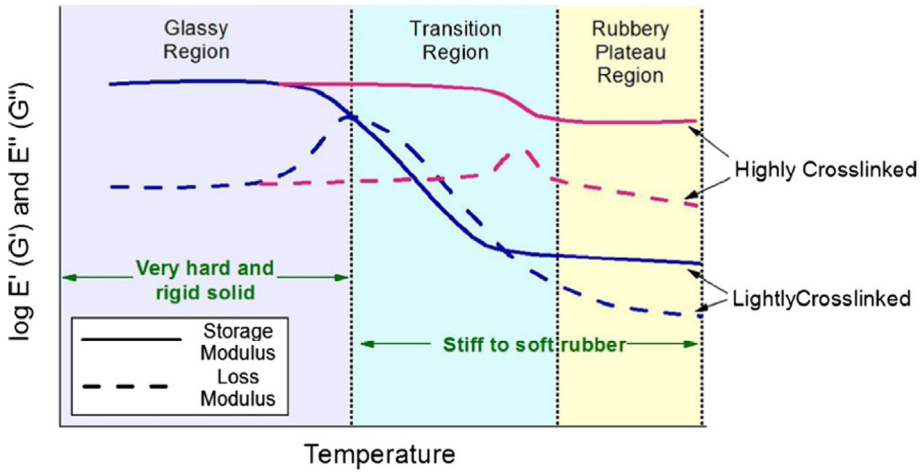


Fig. 3 – Storage modulus and loss modulus of highly and lightly cross-linked composites [21].

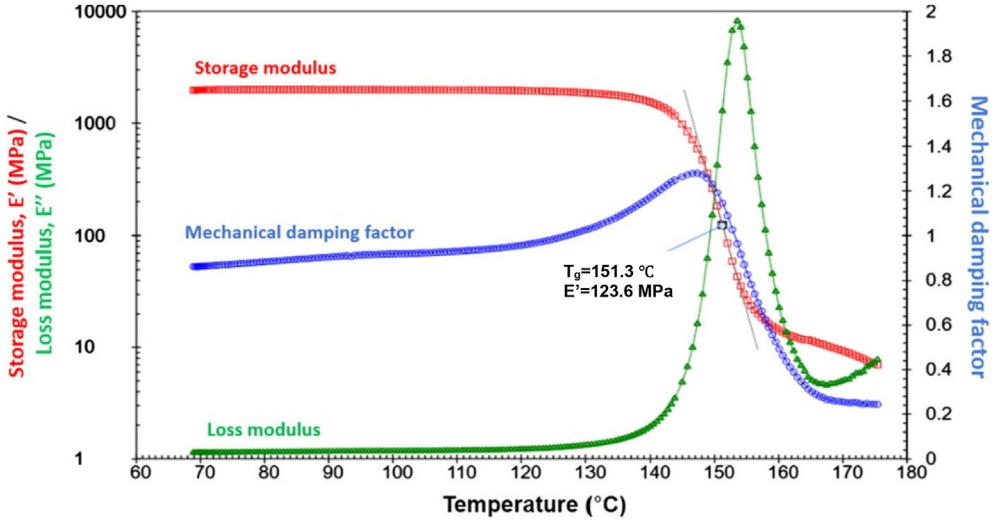


Fig. 4 – Determination of T_g from storage modulus curve [15].

where P_H is the estimated property of hybrid composite, P_1 and P_2 are the properties of composites with individual fiber reinforcements 1 and 2, respectively, and V_1 and V_2 are the volume fraction of each fiber in the hybrid composite such that:

$$V_1 + V_2 = 1 \quad (2)$$

Hybrid composites can be classified according to various aspects such as its polymer matrix, scale of reinforcing material, reinforcement types, and bond strength [34]. Typically, the classification made based on matrix materials is divided in two categories; (1). Thermoset such as epoxy and polyester; and (2). Thermoplastic such as acrylonitrile butadiene styrene (ABS), polypropylene (PP), and polycarbonate (PC). In this work, the authors proposed another category to be included, which is bio-polymer. Recently, the use of environmentally friendly matrices such as cellulose, starch, poly (lactic acid), and biodegradable epoxy are increasing due to its desired biodegradable characteristic [35–38]. In addition, the development of fiber reinforced biopolymer has a potential to reduce carbon footprint [39]. Taking all into considerations, hybrid bio-polymer composites are worth to be discussed more in the future.

Another categorical group of hybrid composites is made based on the reinforcing fiber scale. If one of the reinforcing materials used in the matrix is in the nanometric scale (between 1 nm and 100 nm), the composite is termed nanocomposite. Nanoparticles, nanotubes, and lamellar nanostructures are often incorporated in a polymer matrix, resulting in nanocomposites with unprecedented flexibility and enhanced properties [40]. This review emphasizes on the dynamic mechanical properties of thermoset, thermoplastic, and bio-polymer composites, nanocomposites, and bionanocomposites.

4. Dynamic mechanical properties of natural fibers reinforced hybrid composites

4.1. *Hybrid thermoset and thermoplastic composites*

To date, numerous studies have fabricated and investigated the performance of natural fibers reinforced hybrid thermoset

and thermoplastic composites. Some significant works of the composites that considered DMA properties are summarized in Table 1.

Recently, Nurazzi and co-authors [52] have carried out an extensive study on the dynamic mechanical properties of sugar palm yarn/glass fiber reinforced unsaturated polyester hybrid composites. The sugar palm yarn fiber was chemically treated as a remarkable attempt to improve the fiber/matrix interfacial adhesion alongside other properties enhancements. The results have shown that the inclusion of alkali treated sugar palm yarn had improved the stiffness considerably, evident by the increase of storage modulus as shown in Fig. 5. At higher temperature, there is no notable change in storage modulus due to hybridization or an increase of the glass fiber ratio and alkaline treatment. It was claimed that the treated fiber had better compatibility with the glass fiber and matrix as the hydrophilicity of the sugar palm fiber was reduced after being treated. In addition, the T_g of treated fiber hybrid composite had shifted from 61.20 to 73.84 °C, implying enhanced interfacial adhesion between the reinforcement and the matrix.

A DMA study of jute/sisal fiber reinforced hybrid polyester composite was presented by Gupta et al. [53]. The fibers in each composite were maintained at 30% of the total weight. The findings revealed that hybrid composites with higher sisal fiber content (jute to sisal ratio of 25:75) exhibited higher storage and loss moduli than composites with hybrid ratios of 50:50 and 75:25. Furthermore, the hybrid composite also portrayed the most desired damping factor (0.278) and higher T_g (94 °C) as compared to other fabricated composites with different fibers ratios. On the other hand, composites with a single type of reinforcing fiber showed minor enhancements of DMA properties.

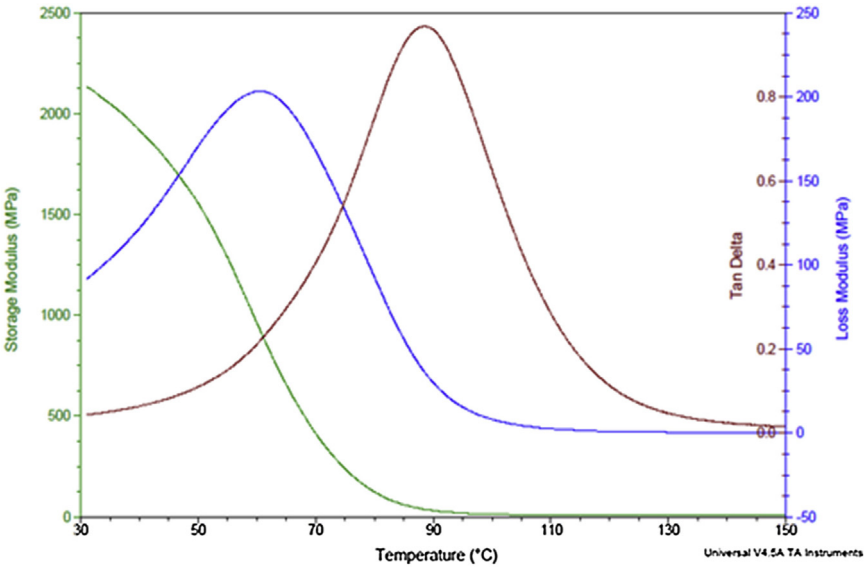
Recently, areca and kenaf fibers hybridization as reinforcing material for an epoxy matrix was examined by Sathyaseelan et al. [41]. The study emphasized the different stacking sequence of the reinforcing fibers. The DMA results showed that the areca/kenaf hybrid composite showed intermediate storage and loss moduli enhancement, whereas the kenaf fiber reinforced composites exhibited superior properties. Regarding the damping factor, it was reported that kenaf fiber reinforced composite had approximately 13% lower value compared to areca reinforced composite. Upon the hybridization of both fibers, an intermediate damping factor value was obtained.

An interesting study by Arulmurugan et al. [42] has attempted a slightly different approach by adding $BaSO_4$ filler in aloe vera/hemp fiber and flax/hemp fiber reinforced hybrid epoxy composites. The result has demonstrated that without the filler, flax/hemp fiber hybrid composite exhibited higher storage modulus compared to aloe vera/hemp fiber hybrid composite. The inclusion of filler has only a slight enhancement effect for $BaSO_4$/flax/hemp composite, while a tremendous improvement was portrayed by $BaSO_4$/aloe vera/hemp hybrid composite. Similar trends were observed for the loss modulus and damping factor of the composites. It was declared that the filler particles promote molecular mobility and minimize the fiber-matrix bonds.

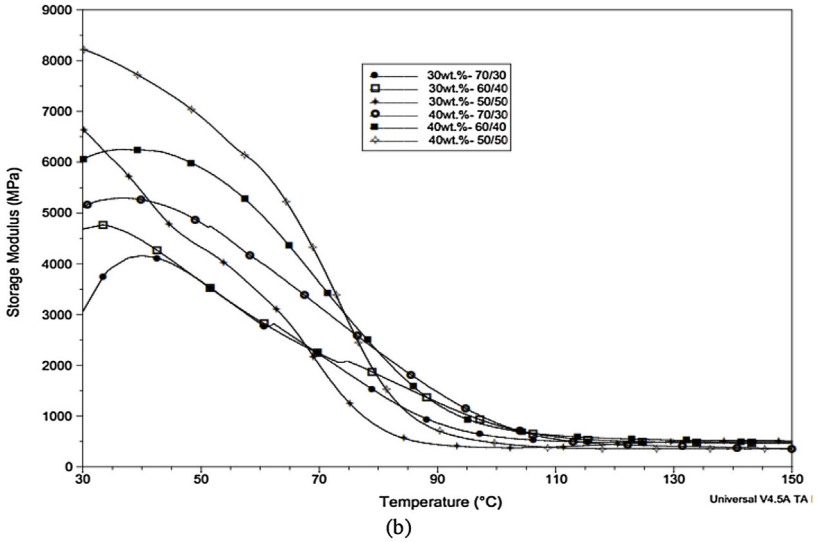
The number of works for thermoplastic polymers are not as extensive as the research done on thermoset polymers

Table 1 – Reported work on dynamic mechanical analysis of natural fibers reinforced hybrid thermoset and thermoplastic polymer composites.

Fiber	Fiber Type	Fiber Loading	Fiber Ratio	Matrix	Composite type	Modulus (MPa)		Tan d	Ref.
						Storage	Loss		
Areca/Kenaf	Long, Long	22 vol.%	–	Epoxy	Laminate	1650	190	0.25	[41]
Aloe vera/Hemp/$BaSO_4$	Long woven, Long woven, powder	–	9.74/10.34/5 wt%	Epoxy	Blend/Laminate	35,500	3550	0.55	[42]
Hemp/Polyethylene terepthalate	Long, Long	–	–	Epoxy	Intra-ply	3500	500,000	0.5	[43]
Coir/*Luffa cylindrica*	Long, Long interwoven	–	–	Epoxy	Laminate	70,000	6000	0.65	[44]
Bamboo/Kenaf	Long, long woven	40 wt.%	50/50 wt.%	Epoxy	Laminate	133	68.67	0.21	[45]
Glass/Sugar palm	Long, Long	10 v/v%	7/3 v/v%	Epoxy	Laminate	2203	296	0.68	[46]
Flax/Sugar palm	–	30 wt.%	15/15 wt.%	Epoxy	Laminate	6232	970	0.41	[47]
Bamboo/Glass	Short, Long woven	30 wt% bamboo	–	Epoxy	Laminate	4400	650	0.6	[48]
Pineapple leaf/Basalt	Long, Long	–	–	Epoxy	Laminate	6650	–	0.420	[49]
Kenaf/Date palm/Pineapple leaf	Short, short, short	–	40/50/10 wt.%	Epoxy	Laminate	10	10	2.0	[50]
Pineapple leaf/Kenaf	Powder, powder	50 wt.%	35/15 wt.%	Phenolic	Blend	3600	190	0.9	[51]
Sugar palm/Glass	Long Woven, Long	40 wt.%	50/50 wt.%	Polyester	Laminate	8450	1184	0.35	[52]
Jute/Sisal	Long, Long	30 wt.%	25/75 wt.%	Polyester	Laminate	3800	500	0.25	[53]
Jute/Glass	Long, Long interwoven	–	–	Polyester	Intra-ply	2000	200	0.4	[54]
Sugar palm/Glass fiber	Powder, short	40 wt.	30/10 wt.%	Polyurethane	Blend	3200	275	0.2	[55]
Date palm petiole/Glass	Long, long	30 wt.%	10/20 wt.%	Vinyl ester	Laminate	3850	400	0.66	[56]
Flax/Almond shell	Long, powder	25 wt.%	15/10 wt.%	Vinyl ester	Blend/Laminate	4500	450	0.6	[57]
Bagasse/Kevlar	Short, short	10 wt.%	5/5 wt.%	Vinyl ester	Blend	1800	500	1.5	[58]



(a)



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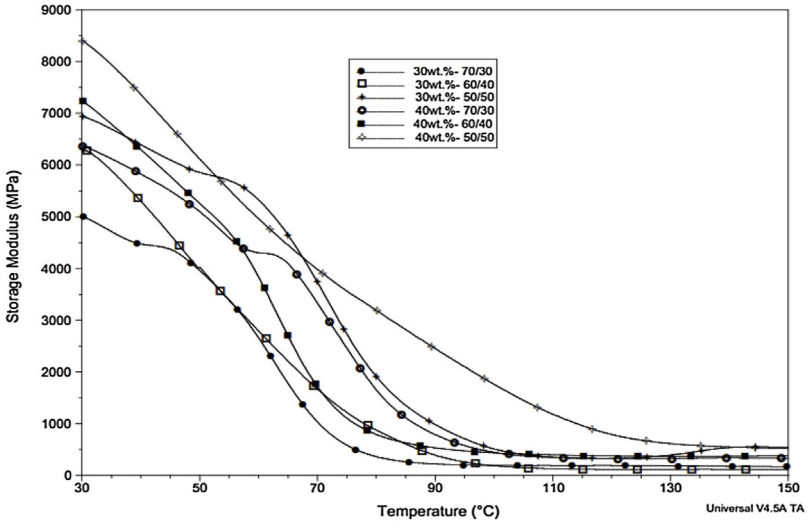

(c)

Fig. 5 – Storage modulus of (a) untreated and (b) treated sugar palm yarn fiber hybrid composites [52].

[59,60]. A recent study carried out by Atiqah et al. [55] attempted to hybridize natural and synthetic fibers, namely sugar palm and glass fibers, as reinforcement for polyurethane matrix. In contrast to other studies that typically manipulate the ratio of reinforcing fibers, this particular work used a constant sugar palm to glass fiber ratio of 30:10. The sugar palm fiber was subjected to several treatment methods, which are alkaline and silane treatments, aiming to enhance the composite's properties. The impressive effort results in remarkable findings as better properties were obtained upon the inclusion of treated fiber. In particular, hybrid composites with treated sugar palm fiber exhibited lower storage modulus, indicating that the resulting composite was more flexible and had a lower stiffness degree. A similar trend was reported for the loss modulus variable, with broadening of the loss modulus peaks. Concerning the damping factor, hybrid composites with untreated, alkaline treated, and alkaline/silane treated sugar palm fibers portrayed a lower damping factor relative to hybrid composite with saline-treated sugar palm fiber. The T_g of the hybrid composites derived from the loss modulus curve revealed that the addition of treated sugar palm fiber in the matrix had lowered the T_g by up to 5 °C.

4.2. *Hybrid bio-polymer composites*

Starch, poly (lactic acid), chitosan, polycaprolactone, and biodegradable epoxy are categorized as bio-polymer matrixes [36,37,61–63]. Up to now, a number of studies have begun to explore the use of bio-polymers as a matrix for hybrid composites as the material is environmentally benign and biodegradable. Some of the reported works on the DMA study of natural fiber reinforced hybrid biopolymer composites are tabulated in Table 2. An impactful work published by Pappu et al. [64] has comprehensively elucidated the characteristics of sisal/hemp fibers hybrid poly (lactic acid) composites. Unlike other studies that emphasized on varying fiber compositions and ratios, this study prioritize on only one fixed composition, that was 15% sisal, 15% hemp, and 70% poly (lactic acid). Five replicates of the same composition were tested and lucidly reported in the article, and hence, all results are well grounded. The DMA results showed that the damping ability of hybrid fiber composite was enhanced as evidenced by the decrease of tan δ peak. Also, the T_g increased from 50 °C for the neat matrix to 80 °C for the hybrid composites.

Table 2 – Reported work on dynamic mechanical analysis of natural fibers reinforced hybrid bio-polymer composites.

Reinforcement	Bio-polymer matrix	References
Hemp/Sisal fiber	Poly (lactic acid)	[64]
Cassava bagasse/Sugar palm fiber	Cassava starch	[65]
Sisal/Corn fiber	Poly (lactic acid)	[66]
Flax/Basalt fiber	Poly (lactic acid)	[67]
Basalt/*Cissus quadrangularis* fiber	Poly (lactic acid)	[68]
Hemp/Sisal fiber	Biodegradable epoxy	[69]
Oil palm/Kenaf fiber	Poly (lactic acid)	[70]
Cotton/Starch	Poly (lactic acid)	[71]
Flax/Jute fiber	Poly (lactic acid)	[72]

Edhirej et al. [65] examined the DMA properties of cassava bagasse/sugar palm fiber reinforced hybrid starch composite. The cassava bagasse loading was remained at 6% w/w, whereas the sugar palm fiber loading was varied from 0% to 8% w/w. In accordance with the reported data, it has been demonstrated that the sugar palm fiber inclusion in the composite had substantially increased the storage modulus values at low-temperature range by up to three folds. This finding indicates that the reinforcing particles are compatible with the matrix and, in turn, had increased the hybrid composite's stiffness. With respect to the loss modulus, the hybrid composites' loss modulus peak had shifted to lower temperatures and higher values, suggesting higher flexibility upon the addition of hybrid reinforcement.

A compelling study by Luo et al. [66] had attempted to hybridize corn fiber, an underutilized natural fiber due to its poor mechanical properties, with sisal fiber as a reinforcement for poly (lactic acid) matrix. The total fiber content was maintained at 30% of the total composite's volume, whereas both fibers' ratio was varied. Based on the DMA result, it was deduced that the sisal fiber provided more stiffness to the composite as compared to the corn fiber. This statement was made on the basis of higher storage modulus values exhibited by hybrid composites with higher loading of sisal fiber. In terms of the damping characteristic, the hybrid composites showed lower damping factors than the neat poly (lactic acid), suggesting that fiber particles restrict the polymeric molecular chains' movement. The derived T_g also revealed that the presence of corn and sisal fibers reduced the mobility of the molecular chains as the T_g shifted to higher temperatures. Among all hybrid composites, composite with sisal to corn fiber ratio of 7:3 demonstrated the best DMA properties.

An investigation on the DMA properties of flax/basalt fiber reinforced hybrid poly (lactic acid) composite was undertaken by Eselini et al. [67]. Unlike other research works, this particular study attempted to hybridize two different origins of natural fibers, plant (flax) and mineral (basalt) fibers, in a single matrix. Basalt fiber has a nearly similar performance in comparison to synthetic glass fiber yet relatively cheaper to be obtained. Several hybrid composites were fabricated with various ratios of flax to basalt fiber. The DMA data revealed that the storage modulus was increased with higher basalt fiber loading, indicating that the basalt fiber restricts the motion of polymeric chains. Hybrid composites with higher loading of flax fiber exhibited relatively low storage modulus, which may relate to the plant fiber's deterioration. Concerning the damping factor, a similar trend to the storage modulus was observed. The incorporation of hybridized flax and basalt fiber in the matrix appeared to have no substantial effect on the T_g, with only 1–2 degree of variation compared to the neat poly (lactic acid).

A year later, a similar work that hybridizes mineral and plant fibers was reported by Kumar and Prakash [68]. They hybridized basalt and *Cissus quadrangularis* (CQ) fibers, an indigenous medicinal plant widely available in India, as reinforcement for poly (lactic acid) polymer. Three composites with varying fiber loadings and total fiber compositions were fabricated and examined using DMA. The DMA study found

Table 3 – Reported work on dynamic mechanical analysis of hybrid bionanocomposites.

Nano-reinforcement	Macro-size reinforcement	Matrix	Mixing ratio	Fabrication	Remark	References
Graphene nanoplateletes	Kevlar/*Cocos nucifera*	Epoxy	GNP(45wt.%) and Epoxy (55wt.%)	Ultra-sonicator	The addition of GNP up to 0.5 wt.% increased the loss modulus by enhancing fiber/matrix adhesion.	[76]
Cellulose nanofibers and montmorillonite nanoclay	Kenaf fiber	Epoxy	CNF (0.75wt.%), kenaf (40.00 wt%) and Epoxy (59.25wt.%)	Mechanical stirrer	Addition of OMMT improved thermal stability owing to the shielding effect.	[77]
rGO	Cotton fiber	Epoxy	rGO (0-1wt%), epoxy resin (64.14 wt %) and cotton (34.86–25.86 wt%)	Hand mixing, compression molding technique	Filler particles improve the mechanical properties of the textile waste composites.	[78]
MWCNTs	Glass/Kenaf and carbon/kenaf fiber	Epoxy	MWCNT (1.0 vol%), Chinese ink (5.6 vol%) and Epoxy (70 vol%)	Sonication, airbrush spray, and vacuum bagging	The addition of the China ink/MWCNT combination increased the rigidity of the glass-kenaf laminate.	[79]
Nanoclay	Bamboo/Kenaf fiber	Epoxy	Nanoclay (1wt.%) and Epoxy (95 –99 wt%)	Mechanical stirrer, homogenizer, and hand lay-up	Insoluble combination of epoxy with unmodified nanoclay resulted a lower E′ value	[80]
Nanoclay	Jute fiber	Epoxy	Nano-clay (1, 3, 5 and 7 wt.%), jute (20wt.%), and Epoxy (73-80wt.%)	magnetic stirrer, ultrasonicator and compression molding	The jute fibre treatment and the addition of nano-clay proved the enhanced interfacial bonding.	[81]
Nano $CaCO_3$	Bamboo pulp fiber	High-density polyethylene	HDPE (65wt.%), BPF (0 & 30wt.%), MAPE (4wt.%) and PE-wax (1wt.%)	Hot press molding (HPMP), extrusion molding (EMP) and injection molding process (IMP)	The EMP composites had the highest interfacial bonding, whereas the HPMP composites had the poorest.	[82]
SiO_2 Nanoparticles	Wheat straw fiber	Low-density polyethylene	LDPE (50wt.%), MA-g-PE (2 phr), SiO2(0–5phr), and wheat (50wt.%)	Injection molding	The amalgamation of SiO2 particles resulted in significant decreases in the rate of heat release, burning, and mass loss.	[83]
Nanoclay	Jute fiber	Natural rubber	Nanoclay (2.5wt.%), jute (0-20wt.%) and NR (80-100wt.%)	Sonicator	The storage modulus of the nanocomposite NR-nanoclay and NR-jute was noticeably higher than the neat compound.	[84]
Nano alumina	Coir pith fiber	Polyester	Nil	Mix blend	In comparison to plain polyester, the composites exhibit superior filler-matrix adhesion and interfacial bond strength.	[85]
Graphene oxide/ nanoclay platelets	–	Polyglycerol-sebacate/Gelatin	PGS (80wt.%), Gel (20%), GO (0-1wt.%) and Clay (0-1wt.%)	In situ polymerisation	Clay nanoplates played a vital part in the synergetic of the hybrid nanocomposite.	[86]
Halloysite nanotubes	Sisal fiber	Polypropylene	PP(82-100wt.%), sisal (10wt.%) and HNT (0-850wt.%)	Internal mixer and compression molding	The mixture of 6% HNTs filler and 10% sisal fibres resulted in a considerable increase in storage modulus.	[87]
MWCNTs/graphene	Jute fiber	Polyvinyl alcohol	PVA (20wt.%), G (0.25–20wt.) MWCNT (0–10%) and JF	Ball-milled, mechanical stirrer and hot press	The modulus of storage and durability of the PVA/G nanocomposites enhanced with the help of jute.	[88]

Graphene oxide	Piassava fiber	Epoxy	Neat and GO-coated Piassava fiber (10–50 vol%)	Mixing, compression molding	The DMA results revealed remarkable changes caused by the amount of fiber reinforcement in the epoxy matrix composites as well as the functionalization of such fibers by the GO-coating. The main DMA parameters such as storage and loss modulus as well as the damping factor showed to be sensitive to these changes.	[89]
Graphene Oxide	Curaua Fiber	Epoxy matrix (EM)	Epoxy matrix (20%), Graphene oxide coated curaua fiber (GOCF)/EM	Hand-laid up and compression molding	The DMA storage modulus (E′) of 50 GOCF/EM was significantly enhanced by more than 250% with respect to the neat EM and more than 90% to 20 GOCF/EM. The DMA loss modulus (E″) of 50GOCF/EM was enhanced by more than 600% compared to neat EM and 200% to 20GOCF/EM.	[90]

that the basalt fiber significantly improved the fiber-matrix interface interaction, as portrayed by the increment of storage modulus. The loss modulus of hybrid composites was lower than the single CQ fiber reinforced composites. Regarding the elasticity, the composite with a higher hybridized fiber loading exhibited lower damping factor, implying higher elastic property. The inclusion of hybrid fibers also increased the T_g of the composite by around 7 °C in comparison to the control.

Recently, Krishnasamy et al. [69] highlighted the effect of hemp and sisal fibers at various stacking patterns on the DMA properties of biodegradable epoxy hybrid composites. It has been demonstrated that the non-hybrid composites with single reinforcement have better DMA properties compared to the hemp/sisal fiber hybrid composites. This conclusion was made based on the detrimental reduction of storage modulus with the incorporation of both fibers in a single matrix. Besides, the calculated effectiveness coefficient also revealed the lower efficiency of fiber reinforcement with hybrid composites. Regarding the loss modulus, single reinforced composites exhibited higher loss modulus values, while hybrid composites portrayed lower values than the neat epoxy. The incorporation of single and hybrid fibers in the matrix also lowered the composite's damping factor, indicating the formation of strong fiber-matrix bonding that impedes molecular movement. T_g values derived from the loss modulus and tan δ showed insignificant differences for neat and hybrid composites.

4.3. *Hybrid bionanocomposite*

Bionanocomposite is a composite material that constitutes at least one type of nano-scale range particles and a biological sourced material or biopolymer [73]. Some of the published works on the DMA properties of hybrid bionanocomposites are summarized in Table 3. In accordance with the compilation of works, it appeared that most studied hybrid bionanocomposites are reinforced with multiple scale particles. Multi-scale composites are composite materials that comprise reinforcing elements from various length scales such as macro, micro, and nano scales [74]. Fig. 6 illustrates the preparation process of multi-scale graphene/carbon fiber reinforced copper matrix hybrid composite.

An impactful work by Jesuarockiam et al. [76] has explored the dynamic mechanical properties of Kevlar/*Cocos nucifera* sheath (CS)/epoxy composites with graphene nano platelets (GNP). It is well known that replacing synthetic fiber with natural fiber may negatively effects the composites' properties. Hence, the GNP was implemented to enhance the thermal and viscoelastic behavior. The Kevlar and CS fibers were layered at varying sequence, whereas the GNP was added at varying composition ranging from 0% to 0.75%. From the DMA, the storage maodulus and loss modulus of S3G3 composite (1:1 ratio of Kevlar/CS and 0.75 wt.% of GNP) exhibited almost similar storage and loss moduli to S1G0 (Kevlar/epoxy). It also exhibited better compatibility between the multi-scale filler and epoxy with lower tan δ. Overall, the hybrid nanocomposite at an optimum composition can effectively replace the Kevlar/epoxy composites.

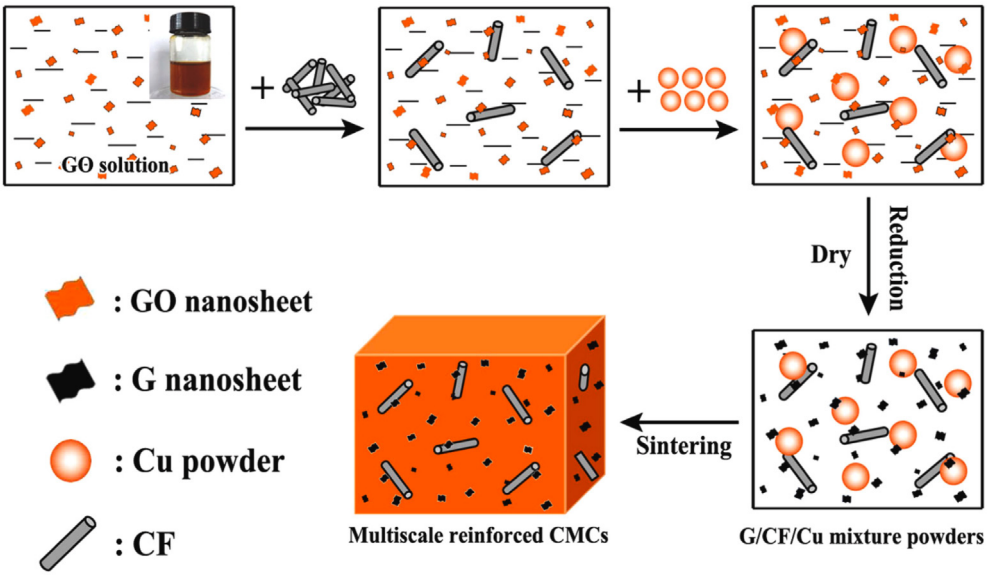

Fig. 6 – Schematic illustration of the construction of multiscale hybrid composite [75].

A detailed DMA study on the multi-scale hybridization of nano-reinforcement and kenaf fiber for an epoxy composite was also reported by Khan et al. [77]. Three different types of nano-reinforcements, namely cellulose nanofibers (CNF), montmorillonite (MMT) nanoclay, and organo-montmorillonite (OMMT) organoclay, were included separately in the polymer matrix at a constant loading of 0.75% and subsequently incorporated with 40% of kenaf fiber to form hybrid epoxy bionanocomposites. It was stated that the modification of MMT to OMMT was an attempt to address the incompatibility of MMT with a hydrophobic polymer such as epoxy. In accordance with the DMA results, it has conclusively been shown that the OMMT hybrid composite restricts the mobility of the polymeric chains relatively better than CNF and MMT hybrid composites, as observable by a higher value of storage modulus. The hybrid composites' loss modulus also portrayed the same trend as the storage modulus, with a maximum value exhibited by the OMMT hybrid composite. Similarly, the OMMT hybrid composite showed the most desired damping characteristic as observed by the damping factor's low magnitude. It appeared that the nanoclay modification had tremendously improved the interfacial adhesion between the reinforcement and the polymer matrix.

A recent study by Kamble et al. [78] assessed the DMA properties of reduced graphene oxide (rGO) nanoparticles/cotton fiber hybrid reinforced epoxy bionanocomposite. The total reinforcing particles were fixed at roughly 36%, whereas the rGO loading was varied from 0% to 1%. Based on the DMA finding, it was figured out that the rGO incorporation in the hybrid epoxy composite at 0.1% and 0.3% loadings had improved the stiffness as observed by the increment of the storage modulus. In contrast, the reduction of storage modulus was noticed when higher rGO loadings were included in the composite, suggesting possible aggregation of the nanoparticles. No substantial loss modulus change was observed with the inclusion of the rGO particles in the hybrid composite. In terms of the viscoelasticity, hybrid composites loaded with 0.1% and 0.3% rGO were lower in damping factor relative to the neat epoxy, while composites with higher ratios of rGO exhibited higher damping factors compared to the control. The increase of the damping factor suggests poor particles/matrix interface bonding at higher loadings of rGO.

Joseph et al. [88] performed compelling research that hybridizes more than two reinforcing particles. MWCNTs, graphene, and jute fiber were added into a single polyvinyl alcohol matrix in this particular work. The graphene loading was remained constant at 20 wt%, while the jute fiber loading was varied from 0 wt% to 20 wt%. The MWCNTs was added at 2.5 wt % loading in only one hybrid composite. It was hypothesized that the inclusion of MWCNTs in the hybrid composite can prevent the aggregation of the graphene particles. The presented DMA data showed that the incorporation of jute fiber in the composite at up to 5 wt% had a positive influence on the hybrid composite's stiffness, as portrayed by the increment of storage modulus value. Nevertheless, a higher concentration of jute fiber had caused poor dispersion and agglomeration of particles, resulting in a detrimental effect on the stiffness. It was also revealed that the hybridization of graphene and jute fiber had a synergistic effect on the composite's damping behavior, as portrayed by the significant reduction of tan δ value. The inclusion of MWCNTs marginally improved the hybrid composite's hardness by bridging the graphene particles with the polymer matrix.

A pioneering study of poly glycerol-sebacate (PGS)/gelatin copolymer reinforced with hybridized graphene oxide (GO) and MMT nanoclay was reported recently by Aghajan et al. [86]. Several composites were fabricated without and with nano-reinforcement with a total loading of 1.0%. Based on the DMA result, it was found that the inclusion of GO and MMT individually did not alter the composites' stiffness, as revealed by the unchanged storage modulus values. Unfortunately, the hybridization of two nano reinforcements had an antagonistic

Table 4 – Application of DMA.

Polymer properties & characterization	Polymer composite characterization	Industrial applications
• Polymer blends and phase morphology	• Storage and loss moduli of polymer composite	• Chemical industry
• Polymer–polymer compatibility	• Evaluation of the interfacial bonding in polymer composites	• Melting point, dynamic modulus, T_g of chemicals
• Polymer rheological and thermal properties	• Investigation of an ideal curing schedule of fiber reinforced polymer composites	• Paints and lacquers industry
• Effect of orientation on the mechanical properties of solid polymers	• Sol gel transformation in polymer composite	• The curing reactions and T_g of the materials
• Rate and extent of curing properties of thermoset resins	• Characterization of the thermo-rheological properties of gel systems	• Oil and gas industry
• Melting point of semi-crystalline polymers	• Mechanical, viscoelastic properties, melting point, vulcanization in elastomeric polymer composite	• Structural pipeline repair
• Polymer glass T_g	• Evaluation of composite structure and performance	• Pharmaceutical and biomedical science
• Polymer damping properties		• Optimization of the formulation of pharmaceutical drug delivery systems
• Polymer storage and loss moduli		• Food industry
		• Glass transition and gelation point
		• Automotive industry
		• Curing reactions, damping behavior, dynamic modulus of auto and aerospace components

effect on the stiffness, which was claimed to be caused by the formation of a separated filler network that restricts the PGS chains' movement. Besides, it was also found that the non-hybrid inclusion of nanoparticles had reduced the composites' elastomeric behavior, as observable by the increment of the loss modulus. The tan δ of the hybrid composite was higher than the control, indicating poor nano-scale filler distribution in the matrix.

5. Applications of DMA

DMA can be utilized for various applications in research and industrial fields. The summarized applications of DMA are depicted in Table 4. Despite being initially invented for studying metals' deformation, DMA is handy for polymer and polymer composite characterizations due to its ability to provide remarkable insights into the polymer molecular structure and stiffness [91]. Owing to its outstanding sensitivity and accuracy to detect molecular relaxation and macroscopic process, researchers often opt for the DMA technique instead of temperature-only based methods. DMA is applied to perform various tests in material engineering research such as thermomechanical analysis, static and dynamic stress–strain, creep recovery, stress relaxation, and dynamic temperature, frequency, and time scans. These techniques revealed numerous material properties such as T_g, thermal expansivity, Young's, loss, and storage moduli, load capacity, ductility, toughness, stiffness, damping, and crosslink density. Based on the recently reported works [92–94], fabricated materials characterized via DMA are primarily designed for biomedical, military, packaging, electronic, and chemical industry applications.

6. Conclusions and future insights

Based on the review, it can be generalized that the DMA technique is a vital method to elucidate the properties of natural fiber hybrid reinforced composites.

- DMA method enables a highly accurate and sensitive detection of storage and loss moduli, damping factor and T_g of heterogeneous polymeric composites.
- The hybridization of two or more reinforcing agents in a single matrix may and may not positively influence the fabricated hybrid composites' DMA properties and T_g.
- Various hybrid combinations; natural/natural, synthetic/natural, single scale, and multi-scale hybridizations to reinforce polymer composites were reported in the literature.
- The loading ratios of the reinforcing particles affect the DMA results tremendously. Hence, it is crucial to optimize the formulation of the composite to obtain optimum DMA properties.
- For the case of nano-scale reinforcements, particularly for bionanocomposites incorporated with graphene derivatives, higher loading often causes particles agglomeration that negatively affects the composites' characteristics.

Overall, this paper has discussed and reviewed some significant works on the DMA properties of hybrid reinforced polymer composites. This review has provided comprehensive information that can be used as a foundation to perform further investigations related to hybrid composites. On the basis of the reviewed research works, several recommendations are proposed for future endeavors.

- The demand for thermoplastics and biopolymers is currently increasing as the human standard of living improves, both in terms of development and sustainability. In comparison to hybrid thermoset composites, there has been a paucity of information on hybrid thermoplastic and bio-polymer composites. More DMA studies on both types of composites should be conducted to obtain better insights into the behavior and characteristics upon the incorporation of hybrid reinforcements.
- In some cases, the addition of natural fibers in the composites have negatively affected the DMA properties. Some researchers have demonstrated the use of nanofillers to counterbalance the negative effect. Future research should look into adding nano-reinforcements to existing hybrid composites to improve their properties while keeping them cost-effective and environmentally sustainable.
- Nano reinforcements have been reported to agglomerate at higher loading ratios, limiting their use to low concentrations. Future research should look into ways to prevent agglomeration, such as chemical treatment or grafting, in order to uncover the previously unseen possibility of nano reinforcements in composites at higher loadings.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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