

## Review Article

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# Influence of filler material on properties of fiber-reinforced polymer composites: A review

<https://doi.org/10.1515/epoly-2022-0080>

received September 06, 2022; accepted October 17, 2022

**Abstract:** The current day target for material scientists and researchers is developing a wholesome material to satisfy the parameters such as durability, manufacturability, low cost, and lightweight. Extensive research studies are ongoing on the possible application of polymer matrix composites in engineering and technology, since these materials have an edge over conventional materials in terms of performance. Hybridization of reinforcements is considered to be a better option to enhance the efficiency and performance of composite materials. Accordingly, research studies focus on the surface treatment of natural fibers and the addition of nanofillers (natural or synthetic) by industry and academia to take the properties and application of composites to the next level. This review purely focuses on the influence of fillers on the properties of composites along with the probable application of filler-based polymer composites.

**Keywords:** natural fillers, natural fibers, polymer composites, mechanical properties, X-ray diffraction

## 1 Introduction

Polymer matrix composites (PMCs) are in prime research focus lately owing to their superior properties that are well-suited for many engineering applications. Composite materials are known to possess enhanced strength and durability with ease in processing and less expensive (1). Current-day researchers focus more on the development of PMCs reinforced with fibers (either natural or synthetic) and fillers (either organic or inorganic) owing to their vast spectrum of properties (2,3). Specifically, synthetic fibers are mostly preferred due to their higher strength, thermal retention, durability, performance with the lightweight, low investment, and the ability to produce any complex structure in marine, automotive, aerospace, and construction applications (4–6). At the same time, natural fiber-reinforced polymer (FRP) composites are also most commonly developed and used in various structural and non-structural applications with low or medium loads. Natural fiber composites are completely recyclable, renewable, and sustainable and also come at a relatively low cost of extraction and processing and are light in weight and low cost. Properties of natural fiber composites are dependent on various parameters including fiber content, fiber orientation, fiber dimension, and fiber placement (7,8). Yet, due to some shortfalls in natural and synthetic fibers, the properties of the attained composites were not as expected. Hence, researchers found various ways and means to improve the properties FRPs. In general, two common methods are widely adopted to improve the mechanical, physical, thermal, tribological, and optical properties of FRPs. The first method is the reinforcement of FRPs with different organic and inorganic fillers, which improves the toughness of matrix material and the second method is the hybridization of FRPs with another type of fibers or fillers in the same or different planes to enhance different composite properties (9–12).

Hybridization of composite is the method of combining FRPs with another type of natural or synthetic fibers or fillers to obtain the combined properties of

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both the materials in the resulting composites, which can be utilized in a wider application spectrum. If the fibers are hybridized, there are many methods including layer-by-layer stacking, arranging different fibers in a single layer, varying the fiber orientation, and placement of selective fibers in a layer (13–15). Hybridization can be carried out even at nanoscale by adding nanofillers, which results in hybrid nanocomposites. Properties of hybrid nanocomposites were governed by the choice of nanofiller, manufacturing method, their dispersion, and the interaction between the fillers, fibers, and the matrix (16,17). The addition of filler materials either into a polymer matrix or into FRPs is a common research work undertaken by various researchers. Generally, filler materials are considered to be inert material which are normally added to FRPs to enhance the material performance by improving the physical and mechanical properties of the composites (18,19). When fillers are added to the matrix, they render composites with a better surface finish retarding the formation of coarse structure and begetting better mechanical properties, which are impossible to obtain with coarse structure (20). Filler materials are either organic or inorganic. Inorganic fillers are used in polymer composites to obtain better thermal stability, tribological behavior, good interfacial characteristics, and higher mechanical properties. The influence of these fillers on the properties of the composites depends upon the size, shape, aspect ratio, surface area, and dispersion of fillers within the composite. In applications like tissue engineering and biomedical implants, the size, content, and geometry of the fillers play a significant role in determining the properties of the composites (21–23).

It was stated in many of the research works that the addition of fillers in the polymer matrix improves the properties, enhances the processability, and reduces the material cost. Usually, filler particles are added to the polymer matrix in micro or nano-size. Nanoparticles or nanofillers offer high surface area and improved adhesion at the interface between the filler and matrix when compared with micro-fillers. Due to this reason, nanofillers are considered to be the ideal particles to improve the thermal, mechanical, and physical properties of FRP composites as nanofillers are characterized by high aspect ratio and specific surface area. Nanocomposites are typically developed by adding either nanoparticles, nanotubes, or nanolayers on a weight basis into polymer matrices (24–27). The addition of nanofillers within the polymer matrix results in better mechanical properties and reduced water absorption in fiber-reinforced composites due to the homogenous dispersion of the nanofillers within the matrix accomplished by mechanical action. This results in good interfacial

adhesion between the matrix and the nanofillers resulting in an effective transfer of stresses between them during loading. It was stated in many research works that the addition of filler increases the fiber–matrix interaction and when the load is applied this promotes increased stress transfer between the fiber and matrix resulting in the improvement of mechanical and thermal properties of the composites (28,29). Filler addition may also reduce the presence of voids within the matrix, which results in increased composite stiffness. Improvement of mechanical properties of filler-incorporated fiber-reinforced composites depends on the content of fillers as the presence of a higher quantity of fillers promotes better particle–particle interaction and fiber–particle–matrix interaction, which may not be possible during the presence of optimal or sub-optimal presence of fillers (30,31).

On the other hand, when the content of nanofillers is increased beyond a certain limit, then the micro-spaces between the fillers increase resulting in agglomeration of nanofillers. This reduces the bonding strength between the matrix and fibers, reducing the mechanical strength of the laminated composites (32). The addition of nanofillers retards the water absorption of the fiber-reinforced composites by preventing the entry of water molecules through capillary action and acts as a water barrier to reduce the rate of absorption of water. Owing to the water-resistant (hydrophobic) nature of the nanofillers, the rate of water absorption reduces when the concentration of nanofillers increases in FRPs (33,34). Fillers present in the polymer matrices govern the properties of the polymer composites based on surface area and chemistry, shape, size, and distribution of fillers. Typically, fillers are of short fibers or particulates with their size varying from micro to nano-scale. Effective improvement in properties of the composites witness an increasing trend with the ratio of filler surface area to volume and witness a decreasing trend with the decreasing size of the fillers (35,36). Effective utilization of nanofillers within the polymer matrix is directly influenced by the particle size of the reinforcing fillers. When the particle size of the filler is small, then the surface area per unit weight of the filler is higher, which results in high contact of filler and polymer at the interface which improves the effectiveness of the filler reinforcement. On the other hand, micro-fillers have a smaller surface area per unit weight and the interaction of the filler with the polymer is retarded by the voids present in between the two filler particles thus reducing the effectiveness of the filler and reducing the performance of the laminated composites. Due to the high aspect ratio and low width of the nanofillers, they render better mechanical, physical, thermal, and optical

properties to the composite materials and promise the development of innovative materials for a wide range of applications (37–39).

Nanocomposites are developed by reinforcing nanofillers through some advanced manufacturing methods resulting in high-performance composite materials. By integrating nanotechnology principles in the development of nanocomposites with fibers, fillers, and matrices, an improvement in the overall performance of the composites can be witnessed (40). It was noted from various studies that the potential of nanofillers in improving the mechanical performance of the composites was very high to enable their use in different engineering applications. Many countries have started to utilize natural filler-based composites in various applications like rail and decking products, car dashboards, interior roofs, seat panels, head liners, doors, architectural moldings, and parcel shelves (41,42). Fillers are typically organic or inorganic. Inorganic fillers include zinc oxide, alumina, calcium carbonate, magnesia, zirconia, titanium dioxide, and silica, while some studies were also conducted on char, carbon black (CB), nanoclay, carbon fibers, graphene, and carbon nanotubes (CNTs). Organic fillers include particulates derived from various plants and animal fibers or sources, bioceramics, wheat gluten, chitosan, and so on (43–45). The quantity of nanofiller to be added to the polymer matrix depends on the type of filler and the type of matrix used to manufacture the composites. In most of the studies, the quantity of filler varies between 4% and 5% by weight (46,47). Table 1 enlists the usage of filler materials in various polymer composites along with the fiber and matrix used in the composite. This review keenly focuses on the influence of filler materials' addition on the properties of fiber-reinforced PMCs. A few aspects regarding the characterization of fillers and the applications of filler-reinforced composites are also discussed in the current review.

## 2 Characterization of fillers

Characterization of the fillers for their surface charge, shape, size, and morphology is usually done by techniques like scanning electron microscopy (SEM), wide-angle X-ray scattering, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy, X-ray photoelectron spectroscopy, and atomic force microscopy. The crystallinity index (CI) and crystallite size (CS) of the fillers were usually determined by using XRD and the functional groups present in the fillers are

determined by FTIR. In most of the studies, XRD analysis was performed with monochromatic X-ray waves with Bragg's angle ( $2\theta$ ) varying between  $10^\circ$  and  $80^\circ$  with a scan rate of  $2^\circ\text{-min}^{-1}$ . The wavelength ( $\lambda$ ) of Cu-K $\alpha$  radiation is 0.154 nm and the X-ray diffractometer will be operated between 30–40 kV and 30–40 mA. During FTIR analysis, the spectral peaks were recorded between wavenumbers of 4,000 and  $400\text{ cm}^{-1}$  with a scan resolution of  $2\text{ cm}^{-1}$  using the KBr disc method (79–82). In most of the studies, CI and CS can be determined using the following equations:

$$\text{CI} = \frac{I_c - I_{\text{am}}}{I_{\text{am}}} \times 100 \quad (1)$$

$$\text{CS} = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

where  $I_c$  denotes the maximum intensity peak at (2 0 0) corresponding to the crystalline fraction and  $I_{\text{am}}$  denotes the minimum intensity peak at (1 1 0) or (1 0 0) specifying the amorphous contents present in the filler. Similarly,  $K$  is a constant with a value of 0.9,  $\beta$  is the full width half maximum value of the diffraction peak (83,84).

Various studies reported these characterization techniques on different natural or synthetic fillers. Few authors determined the CI of chitosan particles, which were reinforced in an epoxy matrix and the mechanical properties of the composites were determined using appropriate tests. From XRD results of chitosan particles, it was noted that the crystalline peaks were identified at  $2\theta = 15^\circ$  and the CI of the particles was calculated to be 65% (85). In some other experimental works, bamboo micro-fillers were reinforced in an epoxy matrix, and the mechanical properties of the composites were determined. XRD analysis was carried out for untreated and 5% NaOH-treated fillers individually. From the results, it could be noted that the crystallinity of the untreated and treated bamboo fibers was 65% and 86%, respectively. The values of CI of untreated and treated fibers were 0.46 and 0.85, respectively. The maximum intensity peak was obtained at an angle of  $22.3^\circ$  denoting the presence of crystalline materials (86). Few experiments were carried out on XRD analysis of multi-walled CNTs (MWCNTs) and micro-glass fiber-reinforced polypropylene (PP) composites. Results stated that neat PP composites exhibited wider peaks when compared with the other composites. Specifically, owing to the addition of MWCNTs, the CS of the composites was found to be reduced. On the other hand, including glass microfibers in PP/MWCNT increased the CS of the composites. As the content of glass microfibers increased, the CS of the composites also increased due to the increase in the content of nano-agglomerates resulting in the formation of coarse-grained composites (87).

**Table 1:** Fillers used in polymer composites

S. no	Matrix material	Fiber reinforcement	Fillers used	Filler (wt%/vol%)	Composite manufacturing method	Ref.
1.	Epoxy	Glass fiber	Titanium carbide	0–20	Hand layup	(48)
2.	Epoxy	Glass fiber	Alumina powder	1–5	Stir casting	(49)
3.	Epoxy	Glass fiber	Silicon carbide	5–10	Hand layup	(50)
4.	Epoxy	Jute fibers	particulates and graphite			
5.	Epoxy	Glass fiber	Aluminum oxide powder	0–10	Hand layup	(51)
6.	Epoxy	—	Silica and carbon powder	0–1	Hand layup	(52)
7.	Epoxy	—	MWCNT and nano-diamond	0.05–0.2	Stir casting	(53)
8.	Epoxy	—	Nanoclay	0–10	Stir casting	(54)
9.	Epoxy	Glass fiber	CNT	1–5	Hand pultrusion	(55)
10.	Epoxy	Glass fiber	MWCNT and nano-clay	0.005	Vacuum bag molding	(56)
11.	Epoxy	—	Coal mine waste particles	0–40	Stir casting	(57)
12.	Epoxy	Glass fiber	Graphene oxide (GO)	0.1–0.7	Hand layup	(58)
13.	Epoxy	—	Core shell rubber particles	0–38	Stir casting	(59)
14.	Epoxy	Glass fiber	Fly ash particles	10	Hand layup	(60)
15.	HDPE and polybond	—	Rice husk powder	20–70	Compression molding	(61)
16.	Polyester	Roselle fibers	Coconut shell particles	0–10	Hand layup	(62)
17.	Recycled PP	—	Peanut shell powder	0–40	Compression molding	(63)
18.	Polyester	Glass fiber	Egg-shell powder	5–10	Hand layup	(64)
19.	PP	—	Wood saw-dust	10–25	Compression molding	(65)
20.	PP	Sisal fiber	Nano-clay	0, 1, 3, 5	Vacuum bag molding	(66)
21.	HDPE	Sugarcane bagasse	Nano-clay	0–4	Injection molding	(67)
22.	PP	Sugarcane bagasse	Nano-clay	1–4	Melt compounding and compression molding	(68)
23.	Epoxy	Sisal fibers	Nano-clay	0–4	Hand layup	(69)
24.	Acrylated soybean oil	Hemp and sisal fibers	Bacterial cellulose	—	Compression molding	(70)
25.	Epoxy	Flax fibers	CNTs	0–4	Spray drying	(71)
26.	Epoxy	Palm oil	Graphene	0.01–0.04	Vacuum resin infiltration technique	(72)
27.	Epoxidized soybean oil	Flax fiber	Nanoclay	0–10	Vacuum infiltration technique	(73)
28.	PP	Bagasse	Graphene	—	Melt compounding and injection molding	(74)
29.	PP	Kenaf	Graphene	0–5	Melt processing technique	(75)
30.	Epoxy	Flax fiber and PLA	Alumina	0–3	Compression molding	(76)
31.	Epoxy	Ramie fibers	MWCNTs	0–0.6	Compression molding	(77)
32.	Epoxy	Bamboo fibers	CNTs	0.15	Hand layup	(78)

Few experiments focused on performing XRD analysis for Polyalthia seed powder filler (PLSF)-reinforced vinyl ester composites. From the results, it was observed that the minimum intensity peak attributing to the amorphous fraction was obtained at  $15.93^\circ$ , while the maximum intensity peak attributing to the crystalline fraction was obtained at  $22.52^\circ$ . The CI of PLSF was calculated to be 65.5% and the CS of the filler was calculated to be 4.44 nm indicating the good chemical and water resistivity of the filler (88). In some other experiments, NiO powder was hybridized with oil palm empty fruit bunch

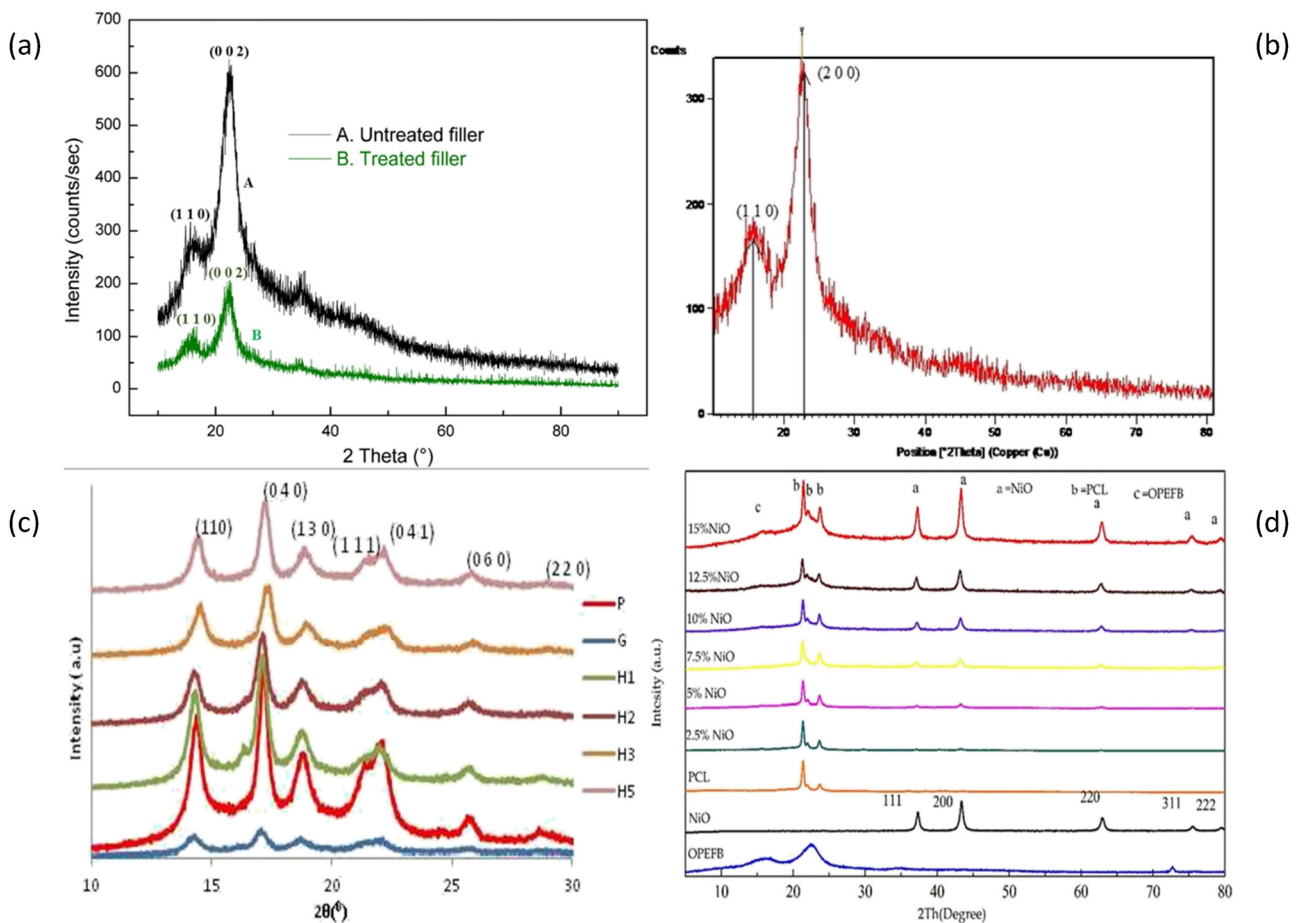
fiber (OPEFB) and reinforced in polycaprolactone in various weight fractions to determine the dielectric properties of the composites. All the precursors and final composites were separately subjected to XRD analysis. It was found from the results that OPEFB and NiO filler powders exhibited higher crystallinity and lower amorphous nature. From the XRD peaks of the composites, it was found that the sharp peaks of polycaprolactone (PCL) and OPEFB were reduced while increasing the weight fraction of NiO fillers. This indicated a good crystallinity of the compounded materials and these materials were recommended to be used in

various dielectric applications (89). Iron fillings were used as reinforcement in polystyrene-based resin in some other experiments and the XRD analysis was performed for iron fillings and composites. It was found from the XRD results that broad peaks of maximum intensity were found between  $16$  and  $28^\circ$  and narrow low-intensity peaks were found between  $10$  and  $16^\circ$ . It was found that the addition of iron fillings increased the crystallinity of the polystyrene composites along with the increase in the weight fraction of the reinforcement (90). From all the aforementioned studies, it was noted that the fillers increased the crystallinity of the composites. Crystalline composites were attributed with cellulose as their major microconstituent, whereas other constituents like hemicellulose, lignin, pectin, and so on were not present. Figure 1 shows the XRD peaks of various types of fillers from different experiments.

### 3 Properties of filler-reinforced composites

#### 3.1 Physical properties

The high-affinity pattern established in thermally kneaded microcrystalline cellulose (MCC) and maleic-anhydride polypropylene (MAPP) binary composites can be clarified by spectroscopy. Cellulase digestion and hot xylene extraction were used to eliminate excess cellulosic and MAPP components not engaged in the binding sites, which allowed for the compacting of the MCC and MAPP binding sites. As a result of enzymatic degradation it was able to diagnose the existence of ester adhesion between MCC along with MAPP using FTIR spectroscopy. Solid-state



**Figure 1:** XRD peaks of filler materials (86–89). Copyright © 2019, Springer Nature; Copyright © 2019, 2020, Elsevier; Copyright © 2018, John Wiley and Sons.

nuclear magnetic resonance (NMR) spectroscopy experiments further corroborated the presence of ester bonding (91). However, it used a DMSO-d<sub>6</sub>/pyridine-d<sub>5</sub> solvent system, “swollen-state” NMR provided clear signals for this with the hydrophilic moieties including cellulose and acid moieties in MAPP, despite the fact that these components seem to be difficult to dissolve in the raw material organic solvents. New signals were discovered and a widening was carried out for 1 h compiled by vigorous kneading, which was measured by the experiments. Both these findings are consistent with the components bonding together via covalent forces. Additionally, 2D Heteronuclear Single Quantum Coherence NMR inside the swollen state provided evidence for the creation of covalent bonds among MCC and MAPP. Thus, the set of cautious extraction treatments and sufficient spectroscopic techniques has successfully detected the enforceable sites for cellulose and MAPP. Swollen-state NMR could be generalized to ternary systems with matrix polymers, potentially allowing for the quantification of highly efficient acid component sites in a compatibilizer. The findings as a whole are crucial to comprehending compatibilizer effects and being applicable to enhance the composite materials’ material characteristics (92). The idea of a percolation transition has been around for a while and has solid groundwork. As a result, getting the composites to the right microstructure is the main challenge when looking into their properties.

When creating percolative composites, the fillers must be crucial that the fillers be evenly distributed throughout the matrix. Physical properties close to the percolation threshold are largely independent of the components. It is theoretically possible to achieve a similar improvement through electrical conductivity ( $k$ ) rather than dielectric constant by combining conductive fillers with an insulating matrix. This observation has not been independently verified for accuracy. It is difficult, if not impossible, to directly compare the physical characteristics of the composite materials that have the same matrix and yet distinct fillers (or *vice versa*) because of the restrictions of current processing techniques. The filler/matrix interfaces in percolative composites have an entirely new impact on the material’s electrical and dielectric properties. New developments in surface chemistry have made it easier to disperse fillers in matrices, allowing for the preparation of percolative composites with significant electrical conductivity at incredibly low values of filler volume fraction or obtaining high electrical conductivity, low-loss percolative composite materials loaded with just a tiny portion of core-shell-structured fillers. By ensuring a more uniform distribution, processing flaws are negated. This allows for the experimental confirmation that the terms of quality

and quantity of fillers influence percolation (93). These materials can exhibit different chemical and physical characteristics compared to a similar chemical compound in the tiny or macroscale spectrum due to their small size in the microscopic or macroscopic spectrum (94) because of their extremely small size. Nanostructured and microstructured 50 wt% tungsten trioxide (WO<sub>3</sub>)/E44 epoxy composites were especially in comparison for their linear attenuation coefficient. The Monte Carlo N-particle code is used to explore the radiation shielding properties of this composite through micro and nano-sizes of WO<sub>3</sub> granules at gamma energies ranging from 50 keV to 1.33 MeV, and the results are compared to experimental data for accuracy. There is a good agreement between the results and the experimental work, which shows that WO<sub>3</sub> nanoparticles with a diameter of 50 nm increase the linear attenuation coefficient more than WO<sub>3</sub> microparticles with a diameter of 1 μm. Moreover, it improves the material’s physical characteristics (95).

## 3.2 Mechanical properties

### 3.2.1 Tensile properties

Wood fiber (ponderosa pine) characteristics were found to have an influence on the mechanical characteristics of wood or PP composite materials by Stark and Rowlands (96). Twin-screw extrusion and injection molding were used to create the composites. Wood flour (particle size equal to 64, 128, 215, and 513 μm) has been compounded at 40% weight with PP and then subjected to a tensile test with ASTM D638 standards. Finer particles had higher tensile strengths. In comparison to 513 μm, 21.7 MPa, the tensile strength of 215, 128, and 64 μm increased by 15%, 13%, and 11%, respectively, while the tensile modulus increased by 11%, 215, 128, and 64 μm by 8%, and by 11%, 513 μm, 3.2 GPa. The length-to-width ratio (aspect ratio) of the nanofiller is typically 3.3–4.5. The highest tensile strength and modulus were achieved with an aspect ratio of 215 μm. The results indicated that the aspect ratio had a more significant impact than particle size. It was assumed that a large aspect ratio would indicate sturdiness (97). Maximum stress transmission between fiber and matrix prior to composite failure requires an aspect ratio greater than its critical value (98). Due to inadequate stress transfer to the fiber and consequently improper reinforcement, fibers with aspect ratios below their critical value merely served as fillers (97,99).

However, once the optimal aspect ratio is attained, fibers are more likely to tangle and form bundles. As a result, its mechanical characteristics deteriorated, and it became more difficult to disperse. On the basis of their investigation, Ryu and Lee (100) advocated for an aspect ratio of 300 for fiber-reinforced rubber. As has been noted by other authors, increasing the aspect ratio tends to improve the mechanical properties (101–103). Microstructure and mechanical characteristics of sisal aggregates (150 and 300 mm) reinforced PP composite were investigated by Durowaye and co-researchers (104). Compression molding was used after a two-roll rheomixer combined sisal powder with ground PP. ASTM D412 1983 was used for the tensile tests. According to the data, the tensile strength of 150 mm is greater than that of 300 mm. Particles' surface area significantly increased with their substantial reduction. This occurred because the particles were evenly distributed throughout the matrix, which enhanced the strength of the particle–matrix interaction and, in turn, the particles' ability to prevent the matrix from deforming too drastically. Wood flour-reinforced PP composite research by Maiti and Singh (105) and also hardwood-reinforced high density polyethylene (HDPE) composite research by Facca and co-researchers (106,107) yielded similar findings. Using just a twin-screw extrusion process rheomix and thereafter injection molding, Rimdusit *et al.* (108) revealed the fabrication of 60 wt% filled PP rubber wood flour polymers containing various particle sizes (165, 90, 215, 275, 49, 362, along with 512 mm). Particle size had an inverse relationship with the tensile strength and modulus, with the sweet spot being reached at 275 mm. As the particle size increased, even more, the tensile strength and modulus both decreased. The composites' tensile moduli at 275 mm were 250% higher than those of pure PP.

Composites reinforced with bamboo sawdust showed a similar pattern. This is because the optimal particle size, 275 mm, provides a greater surface region to transmit the load between the matrix and the filler than do the larger particle sizes. Particles smaller than 275  $\mu\text{m}$  in diameter tended to aggregate into larger ones (109). The larger particle-size fillers have been having trouble wetting with the PP macromolecules. In addition, the high surface area of small particles means that the PP matrix might not be thick enough to completely encase the wood flour. Thus, there were gaps between the filler and the matrix, which reduced Young's modulus (110). The authors found that a particle size between 200 and 300 mm yielded the best results in terms of enhanced mechanical properties. Mechanical characteristics of polyethylene-oil palm empty fruit brunch (EFB) polymer composite were determined

experimentally by Rozman *et al.* (111) depending on the particle size, particle loading, and treatments. Composites are assembled using a hot-pressed technique after being made with a single-screw extruder. The length of the EFB fibers was measured and categorized into three categories: 75–180, 180–270, and 270–500 mm. As seen in EFB, the fillers' capacity to withstand tensile strength transmitted from either the polymer matrix is greatly diminished when they are characterized by abnormal and have a low aspect ratio (112). The tensile modulus significantly increased as the size of particles of EFB shrank. Filler dispersion and also the connection with polymer matrix might well be improved with finer particles having a larger specific area. It was clear from this that EFB fillers could be used to strengthen HDPE composites. Similar findings were found in an analysis of the material characteristics of polyethylene-oil palm frond polymer composite initiated by Rozman *et al.* (113) and Essabir *et al.* (114).

Composites made of corn husk fiber-reinforced recyclable materials with low-density polyethylene were the subject of research by Youssef *et al.* (115). Melt compounding and compression molding was used to make the composites. The composites have been made with varying amounts of corn husk powder (particle size: 125 mm) at a variety of concentrations (5%, 10%, 15%, and 20%). With 10% particle loading, the tensile strength increased by 44%; with 15% particle loading, it increased by 23–25%; and with 20% particle loading, it decreased by 5%. With 10% particle loading, an increase of 64% in Young's modulus was measured. The increased tensile characteristics can be traced back to the R-flow LDPE's and film formation within the composite structure, which strengthened the intrinsic bond strength and composite strength. Stronger internal bonds were found to correlate positively with higher densities and composite strengths (116–118). As reported by Singha *et al.* (117), biocomposites predicated on an unsaturated polyester matrix were strengthened by the addition of untreated and amended *Grewia optiva* fibers (90 mm) manufactured through a mixture of hand layout and compression molding, which improved their mechanical properties. The results of particle loading experiments (0%, 10%, 20%, 30%, and 40%) on untreated *G. optiva* fiber were analyzed. At a particle loading of 30%, the tensile intensity improved by 25%, representing an optimal condition. The improved load transfer here between the matrix and the fiber interface has been responsible for this.

However, as the fibers in composites began to clump together, the materials' tensile strength decreased. The tensile strength was reduced because of the agglomeration-caused defects and voids between the fiber and

matrix. The effect of fiber volume (30%, 40%, and 50%) on the mechanical behavior of luffa (300 mm)-reinforced epoxy composite materials was studied by Anbukarasi and Kalaiselvam (119). All of the unprocessed luffa composites had tensile strengths that were inferior to that of pure epoxy. At 40% particle loading, the tensile strength was at its maximum. Tensile strength decreased as particle loading increased because the fibers and resin were not mixed well enough. Mechanical characteristics of polyethylene-oil palm EFB composite material were examined in experiments by Rozman et al. (111) using particle loadings of 30%, 40%, 50%, and 60%. With expanding particle loading, the EFB composites' tensile strength steadily declined, while the tensile modulus dramatically improved. Due to their typical geometry, fibers failed to resist matrix-induced strain (120–122). Due to the polarity difference, stress transfer inside this composite is hampered, leading to poor interfacial adhesion between the fiber and the matrix (104,123,124). Adding filler led to an increase in Young's modulus because of the agent's inherent rigidity, which manifested as high stiffness. An elevated elastic modulus has been observed in natural lignocellulosic fillers than in polyethylene, PP, and other polymer materials. Consequently, the incorporation of these fillers into its composites enhanced their rigidity. Composites made from PP-reinforced wood flour, palm wood flour with LDPE as a matrix, and peanut husk reinforcement poly(butylene adipate-co-terephthalate) exhibited a similar pattern (125,126).

### 3.2.2 Flexural properties

Wood flour composites with a thickness of 215 mm were found to have greater flexural strength and modulus than those with a thickness of 513 mm, as reported by Stark and Rowlands (96). Strength and modulus of flexure are improved with the use of 215 mm wood flour, which has the greatest aspect ratio. It was found that the aspect ratio had a more significant impact than the particle size. Increasing the aspect ratio improved stress transfer again from the matrix to the fibers (127). Consequently, the material's mechanical properties were enhanced. Flexural modulus for PP-reinforced oil palm wood flour composite material was reported to increase with increasing aspect ratio (128). The trend that increasing the aspect ratio improves mechanical characteristics has been reported by other authors (100,129,130). Polyethylene and oil palm EFB were used in an experiment by Rozman et al. (112). The flexural modulus and strength improved as the particle size decreased (75–180, 180–270, and 270–500 mm). The

smaller particles can withstand more pressure than the larger ones. When comparing EFB composites with varying particle sizes, the ones with filler sizes between 75 and 180 mm showed the least flexural strength reduction. The reason for this was that the smaller EFB particles interacted and spread more widely throughout the polyethylene matrix. The findings indicate that the toughness increased with decreasing particle size (131). So, smaller particles require more force to shatter than larger ones.

About 60 wt% engaging PP rubber wood flour composite materials of varying particle sizes were reported (215, 90, 275, 165, 362, 49, 512 mm) (115). Similarly, the results for flexural strength and modulus have been following the same pattern as the tensile characteristics. The use of a particle size between 49 and 275 mm led to an increase in flexural strength and modulus. The flexural strength and modulus decreased as the particle size increased. Particle loading (30%, 40%, 50%, and 60%) was used in an experiment by Rozman et al. (112) to ascertain the effect on the mechanical characteristics of polyethylene-oil palm EFB composites. The particles weakened the material's flexural strength. Surface hydroxyl groups of cellulose, hemicellulose, and also lignin in EFB act as a shield, making the interface between hydrophilic EFB and hydrophobic polyethylene very weak. There was a rise in interface incompatibility as particle loading enhanced due to a decrease in wetting. The EFB composite materials displayed a rise in flexural modulus with increasing filler loading. More rigidity can be introduced into the composite through the use of filler (132). However, trying to load the specimens with untreated EFB particles led to a decline in toughness. Oil palm wood fiber PP composites (76), untreated curaua fiber-reinforced bio-polyethylene composites (133), and LDPE composite materials with palm wood flour as matrix all showed this same trend.

Flexural characteristics of *G. optiva* fiber-reinforced unsaturated polyester matrices (90 mm) were reported (119). The impact of particle load carrying capacity (0%, 10%, 20%, 30%, and 40%) on untreated *G. optiva* fiber was investigated. With a 30% particle loading, flexural strength increased by 22%. Enhancing the fiber-matrix adhesion would augment stress transfer here between the interphase of fiber-matrix (134), while enhancing the stress needed to deform a greater concentration of fibers would enhance the stress needed to deform the matrix. Wang et al. (135) also found a similar pattern. The flexural behavior of luffa (300 mm)-reinforced epoxy composite materials was investigated by Anbukarasi and Kalaiselvam (119), who looked at the impact of fiber volume at 30%, 40%, and 50%. The material was subjected to



flexure tests in accordance with ASTM D790. For all particle loadings of unprocessed luffa, the flexural strength was below that of unfilled epoxy. In contrast, the flexural strength is enhanced by 25% for a particle concentration of 40%, while staying constant at 30 MPa for particle loadings of 30% and 50%. Reduced bending stress is a direct result of the weakening flexural strength. This was because of the matrix's insufficient assistance for bending stress caused by the reinforcement's non-homogeneous filling. Huge voids may form as a result of particle debonding whenever the composite is loaded (136–140).

### 3.2.3 Impact properties

The energy needed for the dynamic failure of wood flour composite was measured by Stark and Rowlands (96) using the notched and unnotched izod impact test strength (ASTM D256). A notched izod specimen was used to determine the amount of energy necessary for crack propagation, while an unnotched izod specimen was used to determine the amount of energy necessary for crack initiation. The intensity of a notch's impact grew as particle size enhanced (64–513 mm). When improved by 28%, it was at its best. Meanwhile, unnotched impacts lost energy as particles grew larger. The increase was 41%, the study found. The crack spread due to a poor interface between the wood flour and the PP. The crucial crack propagation energy of composites made from larger particle sizes was higher because of the greater fracture surface area (103). Conversely, the unnotched impact energy (the minimum energy required to trigger a crack) is reduced with rising particle size. The wood flour in the PP matrix concentrated the stress, leading to potential cracking locations. As the particle size of wood flour increases, the stress concentrations anywhere along the weak interface between the wood flour and PP also increase (141–145), which also reduces the force of the unnotched impact. Particle size comparisons between 150 and 300 mm at volume fraction 0–25% utilizing the charpy impact test showed a similar trend, as disclosed by Duro-waye *et al.* (104). When loading the particles at 10%, the maximum impact energy has been 4.5 J. An improvement in impact energy of 58% was observed when using 300 mm particles instead of 150 mm ones.

Fibers with a greater cross-sectional area were more effective at absorbing and dissipating impact energy than those with a smaller diameter. The general pattern of findings was also reported by other authors (106,119,146–150). On the other hand, a notched impact test revealed an inverse relationship between industrial wood particle size and the mechanical characteristics of wood–polyvinyl

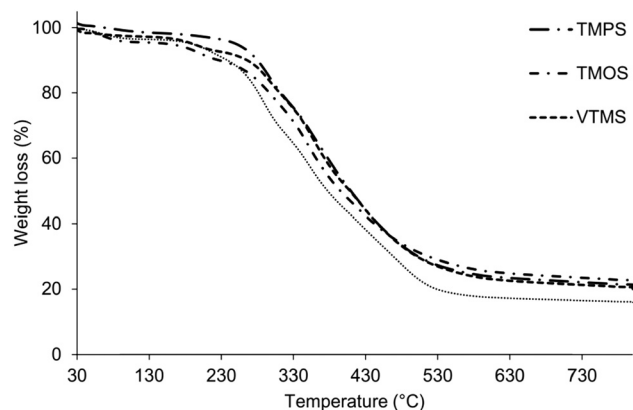
chloride composites (102). Particle loading was analyzed to determine how impact resistance in polyethylene-*oil palm EFB* composites varied, as conducted by Rozman *et al.* (113). As the particle loading increased, the impact strength decreased. The composites' original energy-absorbing capacity was diminished due to the presence of particle fibers. Inadequate bonding or adhesion between the reinforcement and the matrix led to a fragile interfacial region. As a result, the composite system's ability to accumulate energy all through fracture propagation was diminished, and debonding and frictional pull-out of fiber bundles occurred. Using a PP composite filled with rice husk flour, a similar pattern was observed. Untreated luffa (300 mm)-reinforced epoxy composites, Anbukarasi and Kalaiselvam (119) investigated the impact behavior as a function of fiber volume fraction (30%, 40%, and 50%). The ASTM D256 standard was used for the impact tests. It was determined that epoxy matrixes were more robust than luffa-reinforced composites. At its strongest, however, with 40% particle loading, the impact strength was overall increased by close to 30%. For filler concentrations above 40%, this was thought to be due to the fact that there was an insufficient interfacial interplay between the filler and matrix material. In addition to this, agglomeration caused a disruption in stress concentration (151–155).

High-density biopolyethylene (HDBPE) composites reinforced with curaua fiber were investigated by Castro and co-researcher (151), who used liquid hydroxylated polybutadiene as a compatibilizer (LHPB). An internal mixer was used, followed by thermopress molding or injection molding and an intermeshing twin-screw machine were used to compound the mixture. Compared to internal mixing, extrusion and injection molding produce composites with higher impact strength. The composites were made with different particle loadings of fiber, from 5% to 20%. The HDBPE in its pure form was  $234 \text{ J}\cdot\text{m}^{-1}$  resistant to impact. Without LHPB, the impact strength dropped from particle loading 5–15%, only to rise marginally to 20%. The impact strength of composites was significantly improved by adding 5% LHPB as a compatibilizer during fabrication. They formed a more robust interaction because of the enhanced adhesion at the interface and the enhanced load transfer between the matrix and the fiber. Since the polar cluster of the LHPE/fibers and the nonpolar shackles of the LHPB/HDBPE interacted, LHPE rubber was able to impart a toughening influence. This meant that the rubber was more evenly distributed throughout the material than it would have been with the LHPB/HDBPE blend. Nevertheless, particle loadings of 15% and 20%, with the addition of 5% LHPB to each composite, resulted in a slight decrease in

impact strength. As particle loading enhanced, a smaller fraction of the total was made up of LHPB compared to fiber. This made it harder for a greater quantity of fiber to evenly distribute throughout the mixture. Several research studies demonstrating that a composite reinforced with synthetic and natural fibers in addition of filler/modifier had impact strength greater than that of a commercially available HDPE composite (156–159).

### 3.2.4 Thermal properties

Thermo-gravimetric analysis (TGA) thermo-graphs of a kenaf core-polyurethane (KCP) composite and without silane treatment is shown in Figure 2. Weight as a component of temperature for all of these specimens exhibits the same pattern as the temperature is raised to 800°C. Out of the four tests performed (control TMPS, VTMS, and TMOS), it is clear that the TMPS specimen exhibited the most rapid and steady thermal decomposition. This is feasible since the Si within every silane can soak up some of the heat, slowing down the degradation method while simultaneously boosting the establishment of char impurities. Achieved through the development of the siloxane (Si–O) bond, prominent flexibility of the  $-\text{[Si-O]}_x-$  chain segments, and entropically long-term stability of low molecular mass cyclic siloxanes, particularly in comparison to their increased molecular weight throughout against thermal degradation all, seem to be architectural components of silane (polysiloxanes) that have a direct or indirect influence on their stability at various temperatures (160–162). Furthermore, end-group influence resulted in enhanced bonding of the KCP composite material. The char sediment from untreated KCP (19%) is higher than that from TMPS-treated KCP (27%) (163).

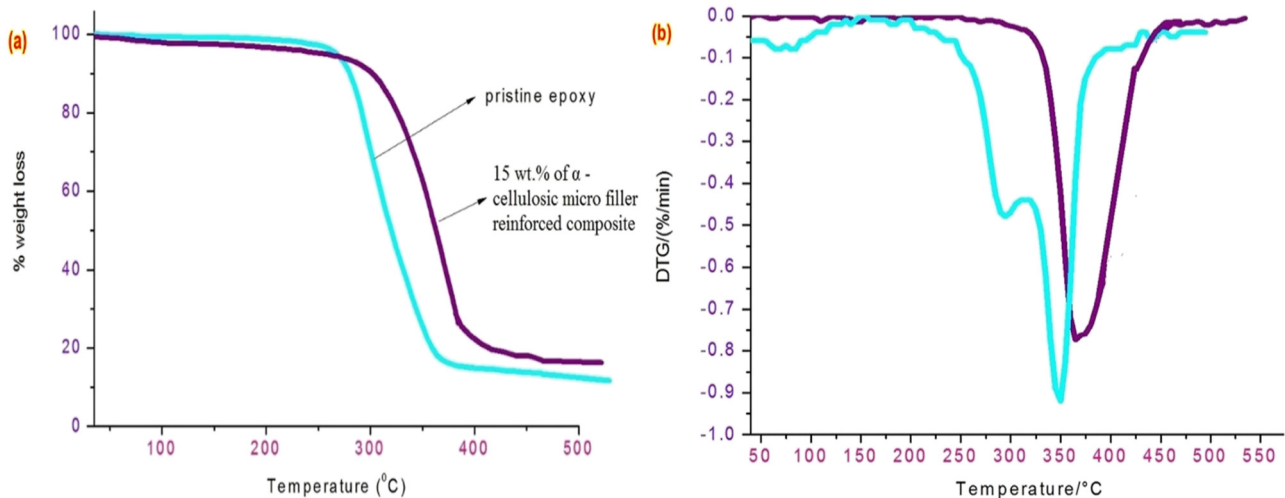


**Figure 2:** TGA without and with silane (163). Copyright © 2019, Springer Nature.

Sheets of a composite material made from low-density polyethylene (LDPE), CB, and OPEFB fiber were made by melt blending and compression molding. Fiber incorporation into composites was studied for its consequences on electrical conductivity, thermodynamic conductivity, tensile characteristics, and thermal degradation. FTIR findings indicate that the OPEFB fibers communicate inadequately well with polymer matrix and contribute to a reduction in mechanical properties. As the percentage of OPEFB fiber in the composites increased, the composites became less conductive electrically. Despite the mild decrease in permeability, the composites even now have been adequately conductive compared to applications of those sensors and electromagnetic shielding due to the fiber addition. The incorporation of 20% OPEFB fiber into LDPE/CB composites resulted in a reduction of thermal permeability by 10.9%. Since natural fiber has low thermal stability, the thermal reliability of LDPE/CB/OPEFB fiber composites is reduced with expanding fiber content (164).

Deforestation is a major threat to the environment, so efforts are underway to reduce it. In this enterprise, agricultural byproducts are used as a substitute for timber in building construction. In this direction, cellulosic micro-filler, which would be separated from *Cocos nucifera* var *aurantiaca* peduncle (CAP) through the chemical treatment procedure, used as a reinforcing material instead of human-made carbon, ceramic fillers, but rather wood-derived products. According to the finding of the tensile, flexural, and impact tests, the mechanical characteristics of the cellulosic micro-filler-reinforced epoxy composites were enhanced in a linear fashion from 3 to 15 wt% of filler loading, with the latter showing superior behavior. Field emission SEM is utilized to probe the broken mechanical test specimens' internal structure. Moreover, visco-elastic behavior and thermal stability of the wt% of  $\alpha$ -cellulosic mini-filler-reinforced epoxy composite was analyzed through dynamic mechanical and TGA and compared with pristine epoxy. Using a TGA, the thermal stability of both pure epoxy and epoxy composites reinforced with cellulosic micro-filler was determined. The sample was heated from space temperature to 720°C at a rate of  $10^\circ\text{C}\cdot\text{min}^{-1}$  in an atmosphere of nitrogen ( $20\text{ mL}\cdot\text{min}^{-1}$ ) (165).

Figure 3 shows the results of the TGA performed on both pure epoxy and epoxy composites reinforced with 15 wt% cellulosic micro-filler. At temperatures up to 120°C, the preliminary weight loss of wt% was caused by the evaporation of mechanically weak humidity. For a mix containing 15 wt% cellulosic micro-filler and virgin epoxy, the outer layer is only loosely bounded by the composite and dehydration of supplementary alcoholic groups. Second-stage thermodynamic decay up to 290°C involved the decomposition and pyrolysis of aromatic clusters for



**Figure 3:** (a) TGA curve and (b) DTG curve of the 15 wt% of  $\alpha$ -cellulosic micro-filler composite along with pristine epoxy (165). Copyright ©2020, Elsevier.

the epoxy system, which also affected the reduction in the curing material of aliphatic amine due to breaking the CN bond to low energy (166,167). There is almost no difference in the weight between the epoxy composite with wt% of cellulose fiber micro-filler and the unreinforced epoxy at this point. Weight reduction of about 65% points to 74% points here between temperate zones of 290°C and 350°C for pristine adhesive and 290°C and also 372°C for 15 wt% epoxy-reinforced composite including both suggests that the incidence of major deterioration may be because of the decay of the epoxy network. Figure 3a and b displays a TGA and differential thermal analysis (DTG) graph showing that the thermal stability and thermodynamic decomposition values for the pristine epoxy are 290°C and 350°C, respectively, while the values for the 15 wt% cellulose fiber micro-filler-reinforced resin polymers are 290°C and 365°C. Consequently, the initiation of carbon char material in cellulose fiber and micro-filler-reinforced polymer matrix transitioned the heat consistency and thermodynamic decomposition of the composite to an elevated temperature, so it behaves like an outer material from that further thermal decomposition of the composite occurred (165,167).

Natural composites have been gradually substituting plastic waste in an effort to achieve nature conservation. The purpose of this investigation was to investigate the feasibility of incorporating waste tamarind seed powder (TP) as nothing more than bio-filler reinforcements through an epoxy-based polymer matrix to produce composites. The filler percent was adjusted from 10% to 20% by the weight of the epoxy matrix to create the epoxy-based polymer. The effect of reinforced TP substance on the mechanical

properties (tensile, flexural, and impact strength) of polymer composites was evaluated using a computerized universal testing machine. Experiments showed that as the percentage of bio-filler in the polymer composite rose from 10% to 40%, the mechanical properties of the composite improved, but then unexpectedly fell when the TP content reached 50%. It was observed that bio-filler-reinforced composite materials made with 40% TP in epoxy had excessive tensile, flexural, and impact strengths of 15, 20, and 25 MPa, including both. Moreover, the thermal properties of the composite are enhanced (168).

### 3.2.5 Sound absorption properties

Composites reinforced with natural fibers are used in place of metals because of their low density, high strength-to-weight ratio, resistance to corrosion, and rigidity. Natural fibers have potential benefits, but their application is constrained by issues like poor interfacial adhesion among fiber and matrix, a low melting point, and absence of resistance to moisture. Thanks to hybridization, the characteristics of composites can be fine-tuned to meet specific needs and optimize the performance. There has been an uptick in research into the potential of using natural fibers as a replacement in fiber-reinforced composites, which has led to new applications in the industry. For this reason, many companies in the auto industry are adopting a “Green outlook” right now. Glass fiber-reinforced polymers (GFRPs) that incorporate natural fibers are increasingly used in the automotive industry. Sisal and waste tea fibers are chosen for their eco-friendliness and acoustic absorption characteristics,

respectively, for experimentation. Hydrophilic components such as hemicellulose, waxes, and lignin are present in both types of fiber. As a result, an alkaline (NaOH) treatment of 5% is applied to the fibers and an epoxy matrix hybridizes them with GFRP. Tensile strength, impact strength, and also flexural strength were among the mechanical characteristics tested. It also has moisture-absorbing qualities. FTIR assessment was used to investigate the chemical makeup of the composite. As an additional experimental technique, the impedance test tube technique is used to examine the sound-absorbing qualities. Data demonstrate that sisal-glass hybrids exhibit improved flexural and impact characteristics. It has been found that tea fiber, when used as filler, is significantly more effective at dampening noise than alternative fibers. SEM analysis was additionally utilized to look at the interfacial properties like internal cracks, blow holes, and fiber pullouts (169–172).

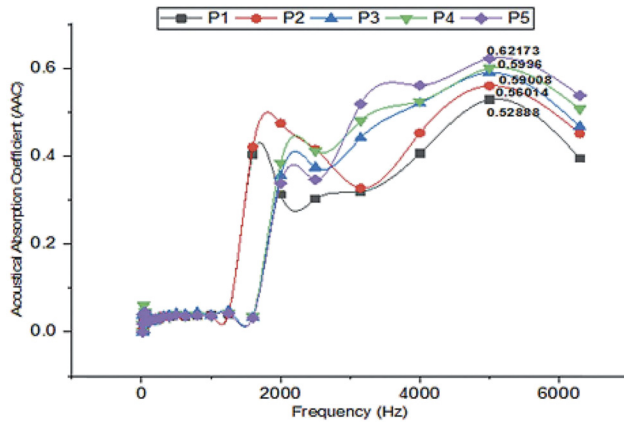
To test whether or not the addition of biochar like unreactive filler to concrete could enhance its efficiency properties and provide a chance for carbon sequestration, researchers swapped out the sand and also coarse aggregate typically used in concrete with biochar. A linear relationship between biochar loading and density reduction in concrete was found. Adding 15% biochar by weight reduced the density of the sample to  $1,454 \text{ kg}\cdot\text{m}^{-3}$ , placing it in the lightweight concrete classification, at the expense of increased brittleness. Biochar additions of up to 12% by weight were found to be safe for concrete. Up to 30% by weight of activated carbon was finally updated, but this had no effect on the density, which remained at  $1,370 \text{ kg}\cdot\text{m}^{-3}$ . Sound absorption coefficients were improved by the addition of biochar and activated carbon over a wide range of frequencies (200–2,000 Hz). The coefficient was the same for both groups of carbon materials despite the variations in concentration. The concrete mixes containing 10% and 15% biochar by weight had a noise reduction coefficient (NRC) of 0.45, putting them in the category of materials with good sound absorption properties. When compared to regular concrete, the addition of biochar improved the thermal insulation characteristics. We found that the thermal conductivity was reduced by  $0.208\text{--}0.230 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  with 1% biochar by weight and by  $0.192\text{--}0.197 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  with 2% biochar by weight.

The lower thermal conductivity enhances the insulating characteristics of the concrete and by extension, the energy consumption of building structures that would use such concrete, but this would not classify the material as a building's electrical insulation. Although there was no discernible pattern in the concrete's compressive capacity as a result of biochar loading, an improvement in compressive strength relative to control samples was noted. The

most vital realization is that the compressive intensity of the concrete is unaffected by the addition of biochar, and the other benefits can be pursued without jeopardizing the structure's integrity. Almost 1 L of additional water was needed for per kilogram of biochar added to acquire a workable paste, which has been a consistent drawback demonstrated across all the experimentations. Therefore, it might be preferable to use plasticizers while adding biochar in order to enhance water efficiency. According to the findings of the multiple experiments conducted on the concrete, it appears that using low concentrations of biochar in concrete can result in desirable material characteristics. While still conserving water, the heat transfer of something like concrete improved between 1% and 2% by weight. At 3% biochar by weight, there was no decrease in compressive strength. The sound absorption coefficient plateaued at around 7% carbon content, concluding that further increase in carbon content had no effect (173,174).

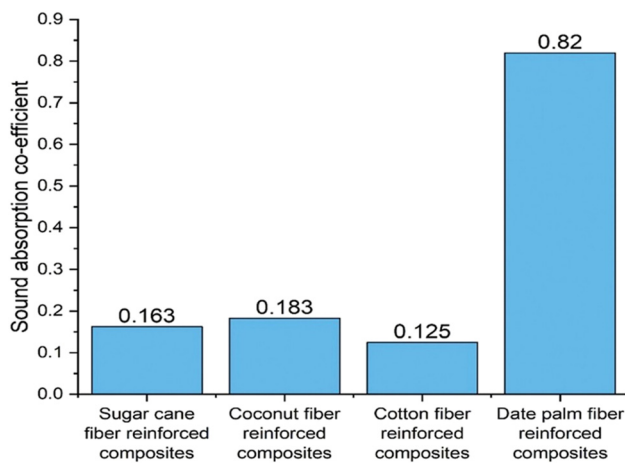
In some other studies, experimental tests were carried out to learn more about the mechanical and sound-absorbing properties of hybrid epoxy composites made from waste tea-leaf fiber (WTLF), kenaf, and E-glass fiber. In the first step, 5% NaOH was applied to the WTLF and kenaf fibers. Compression molding was used to create hybrid composites that were 40% fiber and 60% matrix. Mechanical and sound absorption tests were conducted on the manufactured hybrid composites in accordance with ASTM criteria. The results showed that the composites containing 25% kenaf and 5% WTLF had better mechanical properties, while the composites containing 25% WTLF and 5% kenaf fiber had better sound absorption attributes. SEM was used to analyze the fractured specimens' surface morphology, including fiber pullout and matrix crack. Spectral analysis of hybrid composites treated with alkali demonstrated superior interfacial bonding here between polymer and fiber compared to the untreated fiber (175). Standardized acoustic tests (SACs) were performed on hybrid composite samples across the frequency spectrum. At first, there was no reduction of noise between 450 and 500 Hz. The sweet spot for soundproofing was found to be between 950 and 1,000 Hz. The absorption coefficient values were most noticeably elevated for the range of frequencies between 3,150 and 6,300 Hz. It follows that there is less loss of energy due to vibration at higher frequencies and more at lower ones.

Figures 4 and 5 display the SAC of the bio-composites at varying frequency ranges and SAC values. It was found that the composites have higher SAC across the board. Enhanced frictional losses and lower transmitted sound energy result from sound propagating through a hybrid composite with a greater concentration of particulates.



**Figure 4:** Sound absorption coefficient at various ranges of frequencies (179). Copyright © 2021, Taylor & Francis.

Cross-sectional microscopy of kenaf and WTLF reveals a network of hollow sub-fibers connected by a central lumen. Meanwhile, pure glass fiber seems to have a typical crystal structure (176), and it is a fibrous material. Fiber surface lignin, hemicellulose, and wax are all removed during alkali treatment, while surface roughness and voids are enhanced. Natural fiber composites benefit further from this because of their ability to dampen noise (177). As a result of the increased friction caused by the roughness of the fiber, the sound absorption coefficient is increased (178). Suitable conditions and the prospect to substitute all synthetic fiber systems mean that it can be used in cars, theatres, and homes. When compared to epoxy hybrid natural fiber composites, the SAC had a higher sound absorption rate, ranging from 0.34 to 0.56 (179,180).



**Figure 5:** Sound absorption coefficient of hybrid composites (180).

## 4 Conclusion

The utilization of fiber-reinforced composites is at its peak lately in most of the engineering applications. Based on the requirements, the fiber-reinforced composites were filled with either synthetic or natural filler materials. This article comprehensively dealt with the characterization and properties of fillers reinforced hybrid fiber-reinforced composites from which some salient conclusions could be obtained. In many cases, fillers aid in obtaining higher mechanical and thermal properties for the composites which could not be obtained by the fiber-reinforced composites without fillers. In order to develop nano-composites, natural or synthetic fillers are usually reinforced in nano-size according to the requirement. Nano-fillers are noted to enhance the properties to a certain extent, but beyond that, the properties of the composites were found to decrease due to their agglomeration. This is purely due to the strong vander Waals forces inducing the adhesion of filler particles with one another. This results in a decrease in mechanical properties and poor interfacial adhesion, which is directly governed by the geometry and concentration of the filler. But if natural fillers are used in natural fiber-reinforced composites, they enhance the renewability and sustainability of the composites. On the other hand, fillers increase the adhesion between fiber and matrix and thus enhance the water resistance of the composites by retarding the penetration of water. Fillers are usually added to the composites based on the applications, materials used, and required properties. On the other hand, synthetic fillers enhance the tribological properties of the composites, which are also dependent on the compatibility of the filler with the matrix material. The addition of fillers increases the sound absorption coefficient of a composite material due to the porosity of the fillers. Thermal resistance decreases and thermal conductivity increases for a composite material reinforced with natural filler material owing to the compatible thermal interface generated between the filler, fiber, and matrix. Improving the interfacial adhesion with the matrix, controlling the particle size of the filler, governing the volume/weight fraction of the filler, and choice of matrix render better filler-reinforced polymer composites with enhanced properties. Such filler-reinforced composites find their applications in many areas requiring electrical conductivity, dielectric medium, and in electrical and electronics applications due to the formation of dense percolation networks.

**Funding information:** Authors state no funding involved.

**Author contributions:** Manickam Ramesh: writing – original draft, writing – review and editing, methodology; Lakshmi Narasimhan Rajeshkumar: data curation, validation – verification, formal analysis; Nagarajan Srinivasan: writing – original draft, writing – review and editing, supervision, project administration; Damodaran Vasanth Kumar: project administration, supervision; and Devarajan Balaji: visualization, resources.

**Conflict of interest:** Authors state no conflict of interest.

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