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REVIEW PAPER

Dynamic mechanical behaviour of kenaf cellulosic fibre biocomposites: a comprehensive review on chemical treatments

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Abstract Natural cellulosic fibres, such as kenaf, have potential for use as replacement of man-made fibres in polymeric composites. The rapid depletion of synthetic resources, such as petroleum, and the growing consciousness of global environmental problems related to synthetic products push toward the acceptance of natural fibres as biocomposite components. Kenaf (*Hibiscus cannabinus* L.) is a multipurpose hibiscus species used to make engineered wood, clothing, packing material, rope and twine. Kenaf is essentially made up of cellulose (about 70%), predicting its excellent mechanical performance. Kenaf fibres are chemically treated before mixing with other polymer resins to enhance their fibre properties. Based on the previous literature, the effect of chemical treatment on the dynamic mechanical performance of kenaf cellulosic biocomposites remains unexplored. The present review focuses on the recent works on the influence of major chemical treatments used on kenaf fibre, such as alkaline, silane and acetylation on fabricated biocomposites. The present review also unveils other chemical treatments (e.g. zein and amino acid) and combined treatments on the fibre to improve the biocomposites' dynamic mechanical behaviour.

Keywords Kenaf fibre · Dynamic mechanical analysis · Storage modulus · Loss modulus · Tan δ · SEM

Introduction

Environmental pollution and destruction have become global issues. As such, most governments in the world are working on green technology for sustainable development and optimising the ecosystem and commercial benefits (Razman et al. 2011; Golar et al. 2019; Ali et al. 2020). Coir, hemp, jute, flax, sugar palm, kenaf and sisal are natural fibres used as natural resources. The growing popularity and the market demand towards natural fibre in the current scenario are due to their advantages of low price, lightweight and versatile application (Jaafar et al. 2018; Ilyas et al. 2020b, d). Furthermore, the resources from a myriad

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of natural fibres are widely available, and the energy consumption is low (Sanyang et al. 2016; Zainuddin et al. 2019). Considering that global natural resources, such as fossil fuel, are decreasing in number, the global community is focusing on substituting natural fibres as reinforcing or filler material in composite fabrication (Omran et al. 2021; Ilyas et al. 2021). Natural fibre is gaining interest and utilised in various fields due to its low price, good biodegradability, wide availability and low density (Ilyas et al. 2020c). As such, natural fibre is becoming the new substituent for conventional fibre, including glass, carbon and aramid (Mansor et al. 2015; Mastura et al. 2017). Moreover, the dependency on non-renewable resources can be reduced in the composite fabrication, and the use of natural fibre in polymeric composites through the efficient reuse of plant by-product can overcome the high price of composite products (Johari et al. 2020a, b; Asyraf et al. 2020f).

Kenaf is a widely grown industrial and commodity crop in Malaysia. The crop is classified as a substitute for common plant fibres, such as sugar palm and pineapple leaf (PALF) fibres, because of short harvestable time and various traditional applications as a multipurpose crop, including livestock, food and other fibre advantages (Saba et al. 2015b). Kenaf grows to a height of 4 to 5 m at only around 5 to 6 months, indicating a rapid growth maturation (Rashdi et al. 2009), high abundance of resources and fast cultivation of the crop for industrial-scale production. Kenaf is regarded as a renewable material, and its fibre is sustainable and biodegradable (Joseph et al. 1996; John and Thomas 2008). In this case, kenaf rapidly consumes carbon dioxide at around 1.4 times its own weight and has a high rate of photosynthesis, which is triple than that of common plants (Serizawa et al. 2006). This cellulosic fibre also has low density (1.3 g cm^{-3}). As such, kenaf is widely applied in various thermoplastic and thermoset composites to substitute current man-made fibres (Wambua et al. 2003; Bajuri et al. 2016). The kenaf fibre can reinforce any polymeric matrix via numerous methods, including pultrusion, hand lay-up, hot compression and filament winding (Eichhorn et al. 2001; Siqueira et al. 2010; Saba et al. 2015a). Therefore, in view of its versatility and advantages, the kenaf fibre may be an alternative to petroleum-based fibre and has attracted the interest of researchers, community and manufacturers.

Kenaf fibre composites are popular due to their manufacturability, cheap cost, sufficient energy requirements, abundance, renewability and safety (Davoodi et al. 2010) but have some drawbacks in terms of their physicomechanical and thermal performances. The main downside observed with the inclusion of kenaf fibre in polymeric resin is the lack of surface adhesion between two phases (Bajuri et al. 2016; Asyraf et al. 2020b), which results in the low properties of the final biocomposite laminate. According to Jaafar et al. (2018), the poor interfacial adhesion is due to the occurrence of impurities on the fibre surface morphology, thereby causing a rough topology, as shown in the red circle in Fig. 1. These impurities are made up of lignin, hemicellulose and pectin, which cause the poor compatibility of the fibre with the polymer matrix. Krishna and Kanny (2016) have mentioned that these impurities are made from hydroxyl groups that allow the kenaf fibre to have hydrophilic properties. Considering that most polymer resins, such as polyester (PE) and epoxy, are hydrophobic, the formation of good chemical bonding between kenaf fibres and their polymer is inhibited. Moreover, the inclusion of kenaf fibre in the matrix is frequently associated with agglomeration, which is attributed to insufficient dispersion because fibres tend to form covalent bonds (Liu et al. 2004a; Akil et al. 2011).

These demerits, including the poor interfacial bonding and exhibition of the hydrophilic property of kenaf cellulosic fibre, can be avoided through various chemical treatments that alter the

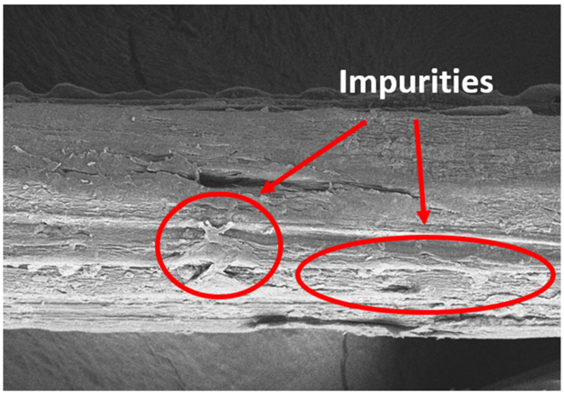


Fig. 1 Fibre surface topology of untreated kenaf fibre observed using SEM (Jaafar et al. 2018). Creative Common CC BY license

compatibility between the kenaf fibre and their matrix. These chemical treatments form fully cured and crosslinked kenaf cellulosic fibre that reinforces the polymeric matrix to attain improved mechanical strength (Jaafar et al. 2018) and thermal stability (Asim et al. 2018). A successful crosslinking between kenaf fibre and its matrix may occur when the hydroxyl group components, such as pectin, lignin and hemicellulose, are effectively washed from the fibre (Aziz and Ansell 2004). This phenomenon forms a strong chemical bonding between two phases' interfaces and a good network and can increase the applications of kenaf fibre, such as marine and automobile sectors, because the process increases the resistance to creep, wear and tear (Asyraf et al. 2019a, 2020a, c, d, e). The enhancement in these properties can improve the service life of the material especially when exposed to extreme conditions, such as elevated temperature, high humidity and acidic environment (Asyraf et al. 2019b, 2020b).

Thus, from the literature, the kenaf fibre is useful for modern industrial applications, such as flooring (Saba et al. 2015b), household products (Mazani et al. 2019) and automobile interior components (Mansor et al. 2014) due to its versatility and numerous advantages. However, some issues have become critical problems that hinder reaching its full potential due to their interfacial adhesion and hydrophilic behaviour. Several research scientists have proposed to modify the fibre topology and component via chemical treatments to grasp their research purposes. Thus, this article provides a state-of-the-art review on the influence of chemical modifications on the dynamic mechanical performance of kenaf biocomposites in various applications. Additionally, this review can be used as a guide to gather information on fibre treatments (e.g. alkali, silane and acetylation treatments) to extend the quality of the dynamic mechanical properties of kenaf biocomposites.

Kenaf fibre and its biocomposites

In general, kenaf has originated from West Africa, but around 75% of kenaf is produced by India and China (Bajuri et al. 2017). As such, the kenaf plant is one of the vital bast fibre resources for these nations. More than 40% of the stalks acquired from kenaf have higher versatility than those from other plants and are useful in many applications (White and Higgins 1964). As such, kenaf is more economical compared with other plants.

The kenaf fibre can be obtained from its bark (Fig. 2) and is lightweight, biodegradable and has good mechanical performance (Tawakkal et al. 2012). However, Nishino et al. (2003) have specified that the shape, size and strength of fibres vary depending on their cultivation condition. The physicomechanical properties of kenaf fibre and other natural fibres are shown in Table 1. Figure 3 shows that kenaf fibres are used in their nonwoven mat form and as woven fabric in textiles, fibreboard, civil, automotive and electronic industries. Moreover, the fibre is frequently implemented as reinforcement in various thermoplastic and thermoset plastics, including epoxy, PE and vinyl ester. In addition, around 15 MJ of energy is required to produce 1 kg kenaf fibre, whereas 54 MJ is needed to produce 1 kg glass fibre in terms of energy consumption (Akil et al. 2011). This remarkable findings shows the low energy in the kenaf fibre production, which subsequently reduces production cost. Hence, the kenaf fibre shows some promising characteristics that can benefit the ecological system and manufacturing industries towards the use of green technology.

Fig. 2 Ripened kenaf plant (Karthikeyan and Kumar 2016). Creative Common CC BY license

Table 1 Physicomechanical properties of kenaf fibre and other natural fibres (Rouison et al. 2004)

Fibres	Diameter (μm)	Micro-fibrillar angle (°)	Density (g/cm^3)	Moisture absorption (wt. %)	Strength (MPa)	Young's modulus (GPa)	Elongation (until break), (%)
Abaca	–	–	–	–	430–760	–	–
Coir	100–460	30–49	1.15–1.46	8	131–220	4–6	15–40
Cotton	12–38	–	1.5–1.6	7.85–8.5	131–220	5.5–12.6	7–8
Flax	40–600	5–10	1.5	8–12	345–1500	27.6	2.7–3.2
Hemp	25–500	2–6.2	1.47	8–12	690	70	1.6
Jute	25–200	8	1.3–1.49	12.5–13.7	393–800	13–26.5	1.16–1.5
Kenaf	–	–	–	–	930	53	1.6
Oil Palm Empty Fruit Bunch	150–500	42	0.7–1.55	–	248	32	25
PALF	20–80	1.4	–	11.8	413–1627	34.5–82.5	1.6
Sisal	50–200	10–22	1.45	10–22	468–700	9.4–22	3–7

Fig. 3 Kenaf fibre mats and unidirectional fabrics (Fiore et al. 2015). Reproduced with permission from Fiore et al. 2015. Copyright 2015 Elsevier

Issues regarding kenaf cellulosic fibre biocomposite

The kenaf fibre consists of various plant components, which provide a rigid, strong and biodegradable fibre. However, several major defects are observed in terms of cellulosic fibre compatibility with polymeric resin to form a good composite laminate. This phenomenon is due to the influence of the high moisture uptake of kenaf fibre that creates a low-quality composite (Joseph et al. 1996, 2002). The high intake of water within the fibre is because of the hydrophilic behaviour induced from the chemical components of the plant fibre (Sreekala et al. 2000; Sreekala and Thomas 2003). In addition, wood fibres, such as kenaf, are composed of highly concentrated hydroxyl group components, which contribute to the properties of the hydrophilic fibre (Makarona et al. 2017; Yallappa et al. 2017; Deeraj et al. 2020). Therefore, a weak fibre–matrix bonding is due to the strong polar and hydrophilic properties of fibre and the polarity and hydrophobicity of polymer materials (John and Anandjiwala 2008; John et al. 2008; Zainuddin et al. 2017). Hence, the low fibre–matrix bonding causes a low ability of the stress transfer of fibre–matrix, which induces changes in the dimensions of the composite and propagates to the microcracks because of the low interlocking effect between two distinct phases of the composite (Paul et al. 2007, 2010; Farahani et al. 2012). The chemical modification of cellulosic fibre contributes to the improvement of the overall material properties, including mechanical, physical, chemical and electrical performances (Pottathara et al. 2016, 2018, 2019).

As a solution, chemical modifications, such as fibre treatments, are introduced to enhance the overall performances of the kenaf fibre as composite and bridge the gap of compatibility amongst hydrophilic fibre and hydrophobic resin features (Liu et al. 2019). Fibre treatments, such as acetylation, silane treatment and alkalisation, eliminate hemicellulose, wax, lignin and oil (Gopakumar et al. 2018; Saba et al. 2019) and reduce the fibre diameter (Bledzki and Gassan 1999), thereby assisting in the adhesion bonding strength between lignocellulosic fillers and resins.

Chemical treatments on the kenaf cellulosic fibre

The modification of kenaf fibres is commonly conducted using chemical reagents or substances. These substances allow the activation or the removal of certain components inside the kenaf fibre to bond the hydroxyl group and the resin inside the composite. Some types of chemical treatments have been stated in the previous literature. A common structure for untreated fibre conditions seems to be a pool of cementing material–hydroxyl group components. A common untreated cellulosic fibre is exposed to these cementing materials, including hemicellulose, lignin, wax and oils (Zhu et al. 2013), as shown in Fig. 4a. The chemically modified fibre has a clean and rough structure because the partial cement structure components are broken and washed away (Guduri et al. 2009), as displayed in Fig. 4b.

Alkalinisation

Alkalinisation or alkali treatment is a chemical treatment that utilises different concentrations of sodium hydroxide and removes the impurities on the fibre to increase the compatibility during reinforcement inside and thermoplastics (Alemdar and Sain 2008; Bachtiar et al. 2011). Generally, the process involves immersing the untreated fibre into the sodium hydroxide solution (Ray et al. 2001; Hassan et al. 2003; Ndazi et al. 2007). The advantage of this chemical treatment is the interruption of the hydrogen bonding in the chemical structure, thereby creating a rough fibre surface. Han et al. (2007) have proven this finding and shown a decline in the absorption peak intensity at the 1640 cm^{-1} band (red circle in Fig. 5), which is due to the removal of OH bending in the absorbed water molecules by alkali treatment. Mat Taib et al. (2016) have used X-ray photoelectron spectroscopy (XPS) and established shown an alkali-treated kenaf oxygen–carbon ratio of 0.59, which is higher than that of untreated kenaf fibre (0.37) and displays increased in the quantity of oxygen relative to carbon. This phenomenon occurs due to the increase in the proportion of carbon element, which may be attributed to the presence of carbohydrates on the fibre surface. Moreover, this observation has established that some polar groups are present on the surface of fibre after alkali treatment due to partial removal of the surface lignin. The removal of lignin and other materials (which make up the noncellulosic layer) from the fibre surface has exposed the underlying cellulose, which has high hydroxyl group content. Mohd Radzuan et al. (2020) reveal that the contact angle of the untreated kenaf/polypropylene (PP) composite (92°) is slightly higher than that of the treated composite (83.1°), as shown in Fig. 6. These attributes explain that the decreased wettability and hydrophilicity of the treated kenaf composite (Product T), which subsequently improves the overall mechanical property. Hence, the modification of the kenaf

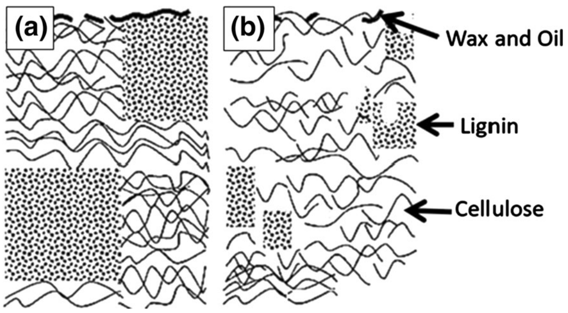


Fig. 4 Schematic structures of **a** unmodified and **b** modified cellulosic fibres (Chandrasekar et al. 2017). Reproduced with permission from Chandrasekar et al. (2017). Copyright 2017 Taylor and Francis

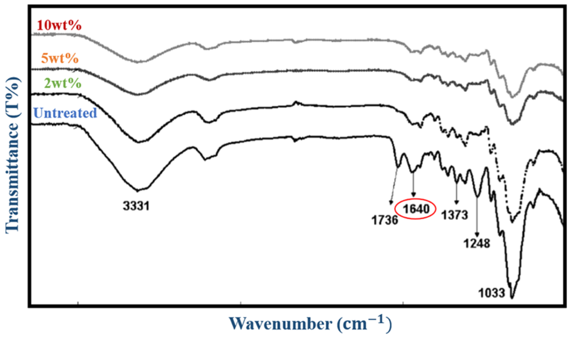


Fig. 5 FTIR spectra of alkali-treated kenaf fibres

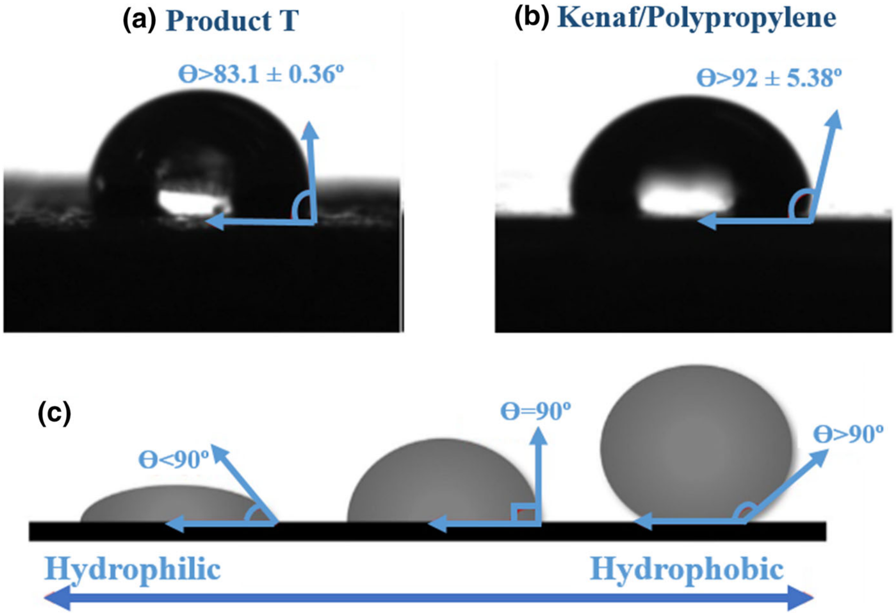


Fig. 6 Contact angles of **a** modified and **b** unmodified kenaf biocomposite

fibre via alkali treatment is a highly significant technique to enhance the fibre–matrix compatibility (Fig. 7).

The sodium hydroxide solution aids in removing plant cellular components, such as hemicellulose, wax, lignin and oils, which inhibit the reinforcement of the polymeric resin by the fibre. Alkaline treatment fibrillates fibre bundles into small fibres, as shown in Fig. 8 (Liu et al. 2004b). The modification shortens the diameter of fibre and increases the aspect ratio and influences the crystalline properties to convert from cellulose I into cellulose II.

Equation (1) displays the alkali treatment reaction of cellulose-based fibres with sodium hydroxide solution (Rashdi et al. 2009):

$$Fibre - OH + Na^{+}OH^{-} \rightarrow Fibre - O^{-}Na^{+} + H_2O \quad (1)$$

Silane treatment

The silane treatment is a well-known fibre surface modification method used to boost the fibre–matrix compatibility in a composite. Silane, a coupling agent, is usually applied to stabilise the composite material. The chemical treatment is useful in reducing the amount of cellulose of hydroxyl groups, which subsequently escalate the fibre–matrix compatibility. When moisture is present, the hydrolysable alkoxyl group leads to the formation of chemical compounds,

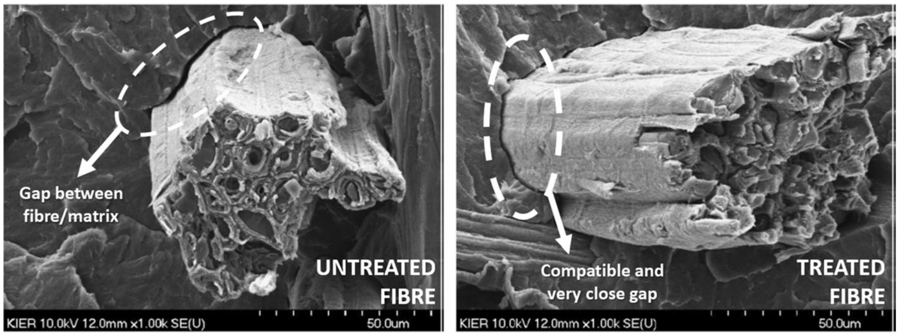


Fig. 7 Raw and 5 wt % of alkali-treated kenaf fibre breakages from the polypropylene matrix (Han et al. 2007)

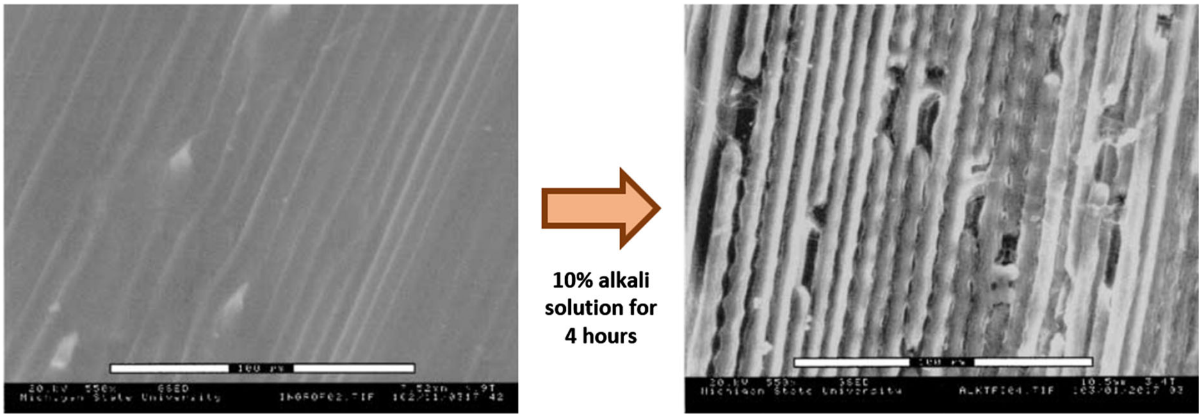


Fig. 8 Formation of small fibres from alkali treatment

namely, silanols. The silanol reacts with the hydroxyl group in the fibre to covalently bond to the cell wall. Hence, hydrocarbon chains are confined from the swelling of the fibre, making an intertwined network from the diffusion of hydrocarbon chain with resin (Dhakal et al. 2007; Seki 2009). Cho et al. (2009) have supported the above statement and found that the Fourier Transform Infrared (FTIR) spectra (Fig. 9) in the upper band has established that the silane treatment forms the asymmetric stretching of either Si–O–C or Si–O–Si band on the kenaf surface in the peak around 1050 cm^{-1}. Furthermore, the Si–C symmetric stretching bands can be found at 765 cm^{-1} (Herrera-Franco and Valadez-González 2005). Jacob et al. (2005) have shown the ^{29}Si NMR through the presence of a signal at − 56.8 ppm chemical shift. This phenomenon can provide a justification that the fibre modification via the silane coupling agent establishes the connectivity of hydroxyl groups on natural fibre with silane reagent. This reputable finding admits that this coupling agent can increase the mechanical interlocking, which causes the condensation between silane and kenaf fibre surface.

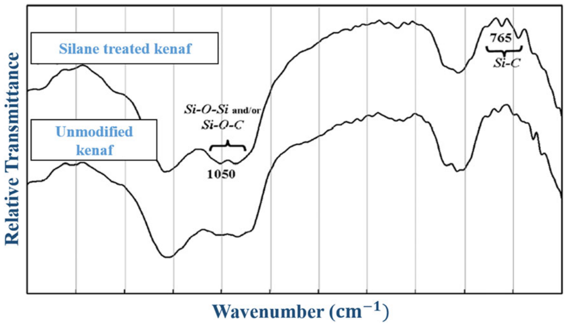


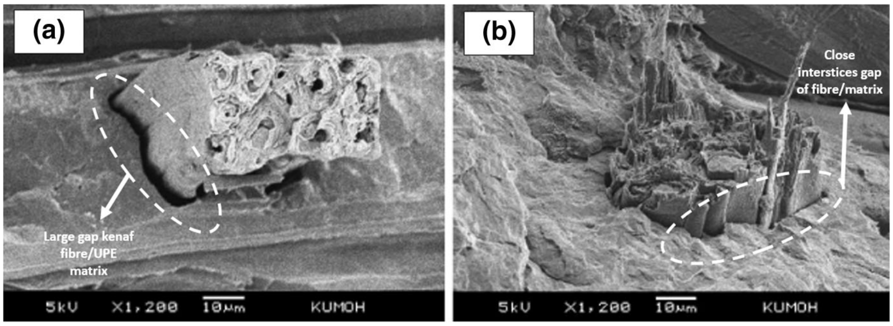
Fig. 9 FTIR spectra of pure and silane-treated kenaf fibres

The silane treatment, which causes the restructuring of the hydrocarbon chain, can affect the wettability of fibre. Hence, this treatment improves the chemical affinity of the matrix and is helpful in enhancing the tensile strength by reducing the effect of moisture to material properties, subsequently increasing adhesion (George et al. 2001; Huda et al. 2008). This argument is supported by Cho et al. (2009), who have reported the close interfacial contact of 0.5 wt. % silane-treated kenaf with its matrix (Fig. 10). Additionally, the pullout length of the treated fibre is comparatively shorter than that of the untreated fibre. Furthermore, Paul et al. (2008) and Pothan et al. (2000) have performed the solvatochromism analysis and discovered that the hydrogen bond-donating ability (α) of fibrils chemically treated with silane is lower than that of untreated fibrils, offering increased surface area due to the dissolution of hemicellulose and lignin and contributing to improved fibre–matrix interlocking. Overall, these findings from the previous literature indicate that the strongest fibre–matrix adhesion improves the stiffness and the strength of the biocomposite via the silane treatment.

The chemical reaction formula can be deduced from the silane reaction:

$$Fibre - OH + R - Si(OH)_3 \rightarrow Fibre - O - Si(OH)_2 - R \quad (2)$$

Fig. 10 SEM surface of kenaf/unsaturated polyester at **a** 0.0 wt. % and **b** 0.5 wt. % silane



Acetylation

Apart from alkaline and silane treatments, acetylation is another famous treatment used by many researchers (Melo et al. 2012; Alsagayar et al. 2015). The process is a well-known chemical modification of esterification, which presents the plasticisation of cellulosic fibre. Chung et al. (2018) have supported this claim, and Fig. 11 displays that the peak (red circle) located at 1740 cm^{-1} is ascribed to the carbonyl stretching bond C=O within the ester bond. The chemical reaction takes place on the cell wall of the hydroxyl groups for lignocellulosic materials by using acetic compound at high temperatures (Dhakal et al. 2007). Chung et al. (2018) have confirmed both arguments by using FTIR analysis (Fig. 11) and found that two peaks at 1369 and 1222 cm^{-1} (blue circle) are attributed to the C–O and C–CH_3 stretching bends, respectively, of acetyl group (Sun and Sun 2002), confirming the acetylation of kenaf. Zafeiropoulos et al. (2003) have found that the acetyl ester bonds are formed on the surface of acetylated flax fibres even though the coverage is heterogeneous based on the XPS and ToR–SIMS approaches. This finding shows a reduction in the amount of hydroxyl groups available for binding with water molecule. Thus, the sufficient acetylation remarkably reduces the amount of hydroxyl to improve the fibre–matrix compatibility. Overall, improvements can be attained using this chemical treatment by using acetic acid to break down the hydrogen bonding, which holds the whole chemical structure together. Subsequently, the surface roughness increases to activate the contact surface of the fibre–matrix (Wibowo et al. 2004; Ifuku et al. 2010) The acetylated kenaf fibre is cleaned from hydroxyl component impurities, which inhibit the interfacial adhesion of the fibre/matrix (Fig. 12).

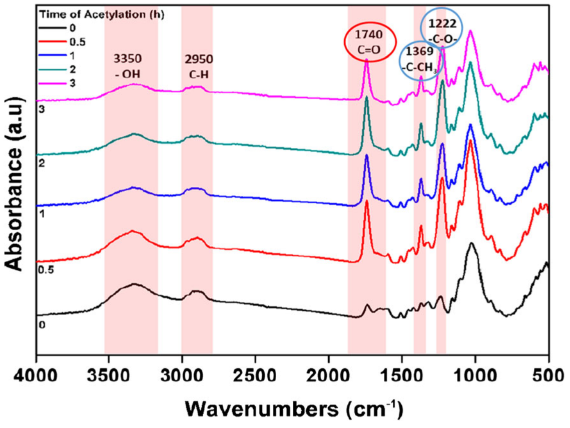


Fig. 11 FTIR spectra for untreated and acetylated kenaf cellulosic fibre with four consecutive times of immersion: 0.5, 1, 2 and 3 h (Chung et al. 2018). Creative Common CC BY license

The reaction order can be found in Eq. (3).

$$Fibre - OH + (H_3C - CO)_2O \rightarrow Fibre - O - CO - CH_3 + CH_3COOH \quad (3)$$

Other chemical treatments

Another chemical modification technique conducted by researchers to improve the interfacial surface of kenaf fibre is through the grafting technique via the polymethyl methacrylate (PMMA) and bleaching. The process aids in lowering the amount of the hydroxyl group in the kenaf fibre, which contributes to the improvement of the crosslinking with polymer resin and encourages the fibre to have a hydrophobic interface because the functional groups of PMMA alter the polar hydroxyl group to form covalent bonds. The functional groups of PMMA include isocyanates [–N=C=O], maleic anhydride (A–$[CO]_2$–O–) and dichlorotriazine (–Cl–), forming chemical bonds to

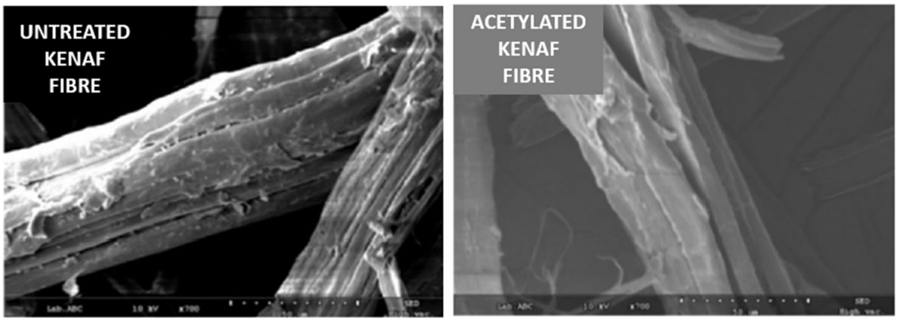


Fig. 12 Structural illustration of untreated and acetylated kenaf fibre in SEM micrographs (Chung et al. 2018). Creative Common CC BY license

establish the stability of the composite (Bakar et al. 2015). According Bakar et al. (2015), a new sharp peak around 1724 cm^{-1} due to an ester group is observed (Fig. 13). This phenomenon can be because the kenaf fibre grafted by the methyl methacrylate by forming a covalent linkage subsequently enhances the compatibility between the kenaf and the PVC/EVA.

The bleaching treatment requires the application of hydrogen peroxide as bleaching agent to eliminate the remaining lignin components from the fibre (Jonoobi et al. 2009). For instance, Sarkawi and Eichhorn (2010) have conducted a study on the bleached kenaf fibre composite by using Raman spectrometry to evaluate the micromechanics deformation behaviours of kenaf/epoxy composite and found that the Raman peak position (1095 cm^{-1}) linearly decreases as the strain and stress increase and reverts to its original position after the fibre is deformed. This finding illustrates that the stress is transferred evenly from fibre to matrix due to improved interfacial adhesion from bleaching.

Thermal characterisation techniques

Material characterisation is one of the main parameters in understanding the properties of plant-based fibre in various conditions. Generally, material characterisation, including tensile, compression and flexural modes, is involved in mechanical testing with respect to an international standard, i.e. American Society for Testing and Materials (ASTM) standards.

The dynamic mechanical analysis (DMA) is a vital laboratory method especially for foods, pharmaceuticals, organic and inorganic chemicals, polymers and biocomposites. The DMA is crucial in examining

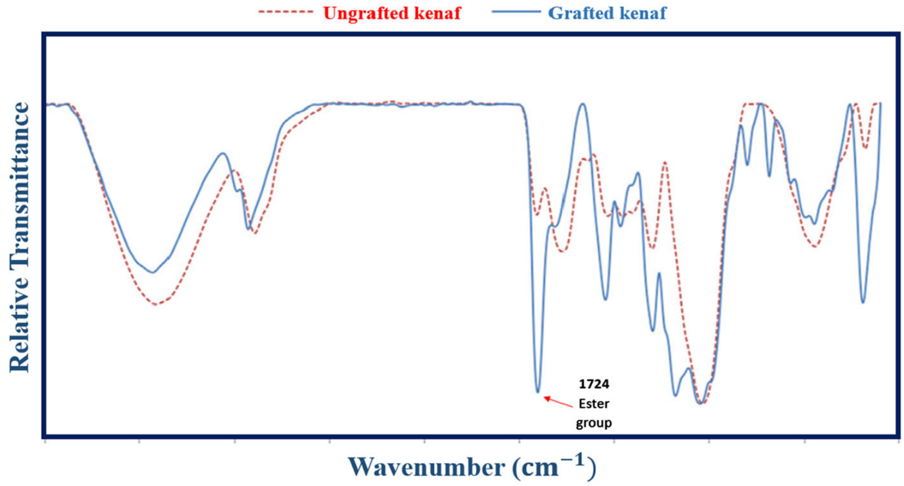


Fig. 13 FTIR spectra of kenaf fibre reinforced in PVC/EVA matrix composites

weight loss, stiffness and strength, dimensional changes and heat flow in terms of temperature (Pothan et al. 2003). The analysis also portrays the behaviours of materials, such as crystallisation, molecular structure, decomposition, viscoelastic property, oxidation, stability, mobility, glass transition temperature (T_g), volatilisation, crosslinking, tensile properties and composition. In general, various thermal analysis approaches and methods, such as DMA, differential scanning calorimetry (DSC), differential thermal analysis, thermogravimetric analysis, thermomechanical analysis (TMA) and pyrolysis combustion flow calorimetry, are available. Thermal analysis methods are summarised in Table 2.

Dynamic mechanical analysis

The rise of modern DMA approaches carries a renaissance in the last few decades of thermal analytical techniques. The DMA is implemented for quality control applications and to compare material performance. Interestingly, the DMA is still one of the most essential tools for material engineering and characterisation fields because these tools effectively sense any possible dimensional change even at the nano level for any solid at specific loads (Gaisford et al. 2002). This test is usually conducted in various conditions depending on the purpose. The common standards applied to evaluate the dynamic mechanical behaviours of polymeric composites as a function of temperature are ASTM D4065, ASTM D4440 and ASTM D5279 (Dynamic Mechanical Analysis 2020). Typically, the DMA is conducted to assess differences in stiffness, damping and T_g of the polymeric composites during curing (Anuar and Zuraida 2011).

The DMA exhibits the outcomes on storage modulus (E'), which is related to the Young's modulus of the composite. The storage modulus or E' is exploited by material researchers to identify the stiffness of the composite. In general, the E' describes the ability of a material/composite to store energy for the upcoming application (Majhi et al. 2010). A viscous response of a material/composite is referred to as loss modulus (E'') or dynamic loss modulus (Jawaid et al. 2013; Ilyas et al. 2020a). E'' establishes the output data on the tendency of material/composites to release the applied energy and is frequently linked with the term internal friction. E'' is sensitive to distinct types of relaxation processes, morphology, transitions, molecular motions and other heterogeneities of the material structure. The DMA aids material engineers and researchers to estimate the amount of polymer chains immobilised by the filler surface in accordance with that reported by Joy et al. (2017). Equations (4) and (5) exhibit the general equations used to attain E' and E''.

$$\text{Storage modulus: } E' = \frac{\sigma_0}{\varepsilon_0}\cos\delta \tag{4}$$

$$\text{Loss modulus: } E'' = \frac{\sigma_0}{\varepsilon_0}\sin\delta \tag{5}$$

The mechanical damping factor (tan δ) can be acquired from the ratio of E' and E'' (Eq. 6). Figure 14 demonstrates the correlation amongst E', E and tan δ in terms of trigonometry view. The resultant component from Fig. 14 is denoted as $E*$ and called as shear modulus (complex modulus). In the analysis, high tan δ values usually indicate that the material/composite is a nonelastic strain component, whereas low tan δ values display high elasticity. A reduction in the tan δ of the composite is due to good fibre–matrix compatibility because the mobility of molecular chains between phase interfaces decreases. The molecular movement and the viscoelasticity influence mechanical damping factor, tan δ. Other minor defects, including dislocations, phase, grain boundaries and various interfaces, affect tan δ (Zhang et al. 2012). Figure 15 shows the example of dynamic mechanical properties (E', E and tan δ) in a graph.

Table 2 Thermal analysis approach with their evaluations and measuring units

Abbreviation	Thermal measuring tests	Measuring unit	Property evaluated
DMA	Dynamic mechanical analysis	Metre	Elasticity
DTA	Differential thermal analysis	°C	Temperature
DSC	Differential scanning calorimetry	J/s	Enthalpy
TGA	Thermogravimetric anaysis	Gram	Mass
TMA	Thermomechanical analysis	Pascal	Deformation

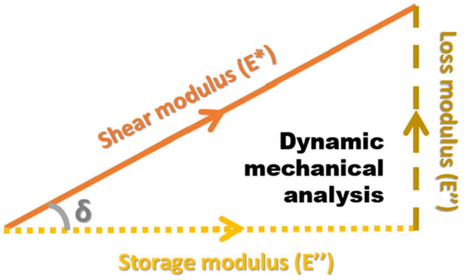


Fig. 14 Relationship of storage and loss moduli and mechanical damping factor

$$\text{Damping factor: } \tan\delta = \frac{E''}{E'} = \frac{\text{loss modulus}}{\text{storage modulus}} \qquad (6)$$

Glass transition temperature

Typically, the T_g is an essential physical behaviour for composite materials especially thermoset polymer composites. The T_g can be used to indicate changes in mechanical behaviour or beginning of phase changes. The T_g identification is applied in DMA to compute the temperature region of the matrix from the glassy state (hard material) to the rubbery state (soft and pliable material) (Kosbar and Wenzel 2017), as shown in Fig. 16.

Overall, the T_g can indicate the state of a material. A low T_g denotes a glassy (brittle) state, whereas a high T_g denotes a rubbery (elastic) state. The glassy region is noticeably high in interlocking bonds, stiffness and region-restricted molecular movement. The rubbery region manifests the mobility of molecular chains when exposed to elevated temperature (Mahdi and Tan 2016). However, several polymers have applications above the specific T_g, and other polymers are useful below the T_g. For instance, rubber elastomers, such as polyisoprene and polyisobutylene, are applied and used due to their flexibility and softness in the rubbery state. Hard plastics (i.e. polystyrene and PMMA) are used in their glassy states (below their T_g, which is approximately at 100 °C above room temperature. A recent study has shown that T_g is notably different from the melting temperature (T_m). At T_m, the sample begins to melt, whereas at T_g, the sample becomes soft (Saba et al. 2016). The T_g can be examined using other thermal analysis methods, including DSC and TMA. Figure 17 illustrates the comparative identification of T_g from DMA, TMA and DSC.

Thermoset composites are present as either highly viscous liquid or ductile solid at temperatures above the T_g and as hard and brittle materials at temperatures below the T_g. Amongst various thermosets, the epoxy is the most crucial thermosets and highly crosslinked in the cured state. In this case, the epoxy polymer does not melt and demonstrates small softening at high temperature. Researchers have conducted analysis on epoxy and illustrated that the epoxy polymer can be implemented explicitly depending on the requirement

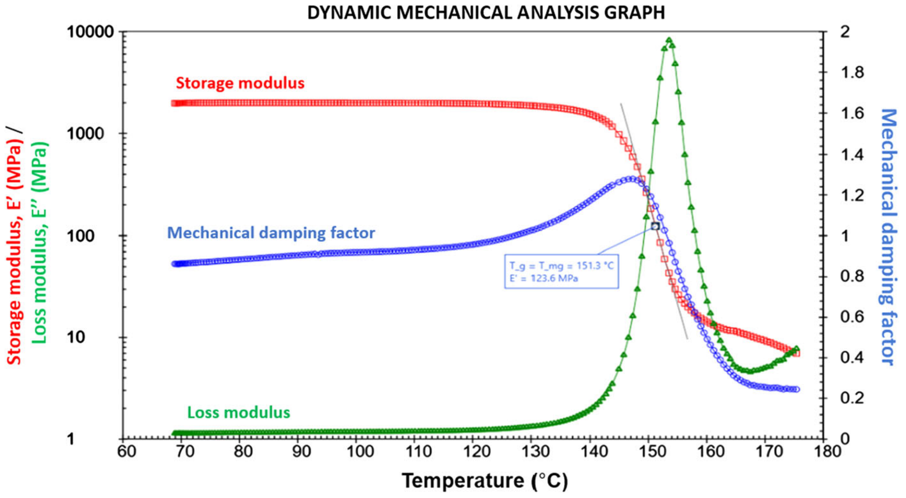


Fig. 15 Example of dynamic mechanical analysis results

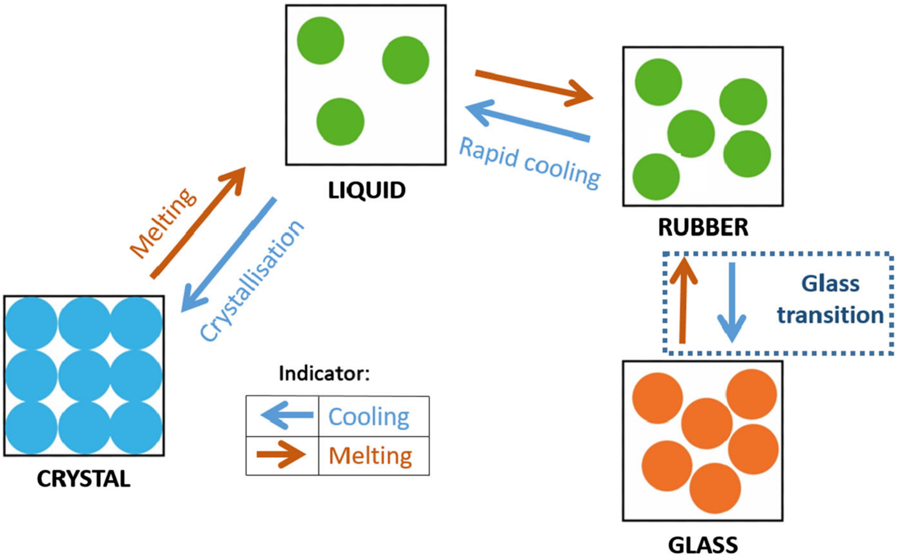


Fig. 16 Phase transition of polymeric materials

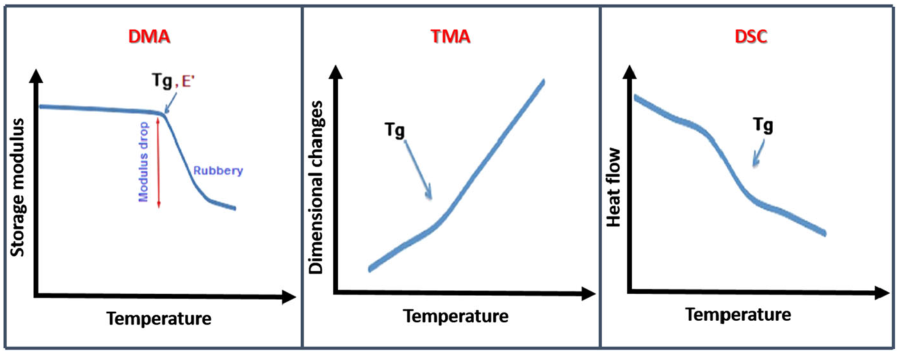


Fig. 17 Comparison of T_g obtained from DMA, TMA and DSC

applications either at below or above their T_g (Saba et al. 2016).

Previous literature on the effect of chemical treatments on the dynamic mechanical behaviour of biocomposites

Recently, several studies have reported the effect of kenaf fibre surface modifications on the dynamic mechanical properties of kenaf cellulosic fibre biocomposites. Some DMA experiments and results on modified kenaf fibre biocomposites are presented in Table 3.

Alkali treatment

Han et al. (2007) have utilised low concentrations of sodium hydroxide (2 wt. %, 5 wt. % and 10 wt. %) on kenaf fibre/PP composites and discovered that the E' values of 25 wt.% and 5 wt.% alkali-treated biocomposites have improved by 15% and 25%, respectively, compared with those of the untreated biocomposite (4.8 GPa) at − 30 °C. The optimum value of tan δ height is located at the composite treated with 5 wt. % sodium hydroxide solution.

Aziz and Ansell (2004) have treated the kenaf bast fibre with 6% sodium hydroxide for 48 hand assessed the relationship of static and dynamic mechanical performances of the hybrid kenaf/hemp fibre-reinforced PE composites via alkalisation and fibre

Table 3 Various chemical treatments of kenaf fibre reinforced polymer composites in DMA analysis

Types of treatments	Fibre size and content	Resin	Optimum conc. of coupling agent	Dynamic mechanical properties							References
				Storage modulus (GPa)			Loss modulus (MPa)			Damping factor	
				T_g	Max	Min	T_g	Max	Min		
Alkalisation	Long fibre	PP	5% of NaOH for	4.900 (0 °C)	5.990 (– 30 °C)	3.100 (100 °C)	–	–	–	0.038	Han et al. (2007)
	Short	Polyester	6% of NaOH for 48 h	–	1.590 (40 °C)	–	195.57 (90 °C)	–	–	0.123	Aziz and Ansell (2004)
	Long UD fibre	Epoxy	6% of NaOH for 48 h	0.35 (87 °C)	10.0 (25 °C)	0.35 (87 °C)	500.0 (87 °C)	500.0 (87 °C)	0.5 (135 °C)	0.320	Fiore et al. (2015)
Silane Treatment	Non-woven fibre with 7:3 of PALF and kenaf fibre	PF	2% silane for 3 h	3.25 (51.1 °C)	3.63 (35 °C)	1.51 (140 °C)	190 (96 °C)	190 (51.1 °C)	130 (40 °C)	0.11	Asim et al. (2018)
	Long fibre	PP; and un-saturated PE	0.5 wt. % of GPS agent for 2 h	5.33 (– 7 °C) (PP) 1.92 (UPE)	5.49 (– 30 °C) (PP) 5.64 (40 °C) (PP)	3.49 (80 °C) (PP) 4.43 (80 °C) (PP)	–	–	–	0.035 (PP) 0.285 (UPE)	Cho et al. (2009)
	Long fibre (52 mm) with 1:1 fibre to resin ratio	PLA	3% of GPS agent for 2 h	4.0 (59.3 °C)	4.6 (– 20 °C)	1.7 (140 °C)	–	–	–	–	Lee et al. (2009)
	Non-woven fibre	PLA and; PBAT	2% of APTMS for 2 min	1.50 (63 °C)	3.63 (30 °C)	0.05 (80 °C)	–	–	–	1.3	Sis et al. (2013)
Acetylation	Short fibre from bast part of kenaf	PLA	400 mL of acetic anhydride for 30 min and 0.01 mol of pyridine as catalyst	–	18.0 (0.0 °C)	2.0 (82.0 °C)	–	–	–	1.1 (68.0 °C)	Chung et al. (2018)
	Non-woven	PE	10 wt.% of CH_3COOH sol. for 1 h	1046 (– 56.2 °C)	1046 (– 56.2 °C)	150 (120 °C)	175 (– 56.2 °C)	–	–	0.16	Datta and Kopczyńska (2015)

Table 3 continued

Types of treatments	Fibre size and content	Resin	Optimum conc. of coupling agent	Dynamic mechanical properties							References
				Storage modulus (GPa)			Loss modulus (MPa)			Damping factor	
				T_g	Max	Min	T_g	Max	Min		
Combined Alkali-Silane treatment	40% wt. of kenaf fibre	PLA	5% NaOH sol. for 2 h; and 5 wt. % APS in 1 h	7.1 (60 °C)	8.1 (25 °C)	3.0 (83 °C)	400.0 (60 °C)	625 (67 °C)	180 (20 °C)	0.20	Huda et al. (2008)
	Chopped fibre (5 mm)	Co-polyester	4% of NaOH sol. for 1 h; and 3, 6, & 9% of silane sol. for 5 min	1.5 (− 30 °C)	7.0 (− 60 °C)	0.03 (100 °C)	650 (− 30 °C)	650 (− 30 °C)	9 (100 °C)	α 0.22 β 0.19	Mokhothu et al. (2011)
Grafting	Chopped and flecked fibre (100 and 150 μ m)	PVC/ Ethylene vinyl acetate	Graphted using H_2O_2; ammonium ferrous sulfate	–	7.3 (− 100 °C)	0.2 (140 °C)	–	400.0 (− 30 °C)	25.0 (90 °C)	α 0.76 β 0.12	Bakar et al. (2015)
Zein treatment	Non-woven fibre	Polypropylene	2% of zein sol. mixed for 2 h	0.7 (1.4 °C)	1.5 (− 20 °C)	0.05 (140 °C)	–	–	–	0.125	John et al. (2010)
Amino acid treatment	Non-woven fibre	Epoxy	10% of both glutamic acid (GA) and lysine (L) sol for 24 h	0.15 (62 °C) (GA) 0.15 (62 °C) (L)	2.98 (25 °C) (GA) 2.85 (25 °C) (L)	0.06 (100 °C) (GA) 0.06 (100 °C) (L)	325.0 (62 °C) (GA) 340.0 (62 °C) (L)	325.0 (62 °C) (GA) 340.0 (62 °C) (L)	6.3 (100 °C) (GA) 6.3 (100 °C) (L)	0.75 (GA) 0.72 (L)	Krishna and Kanny (2016)

alignment. Results show that the alkalised and long fibre composites have high E' and low tan δ values, which correspond to high flexural performance. A chemically modified and long fibre in the composite permits improved strength because the fibre orientation is well organised and has good fibre–matrix adhesion bonding. Subsequently, these factors aid in localised stress in a bending condition.

Fiore et al. (2015) have treated the kenaf fibre with 6% sodium hydroxide for 48 and 144 h. Best E' and tan δ of kenaf/epoxy composites are achieved when the unidirectional kenaf fibre is treated at 48 h. Additionally, the trends in the dynamic mechanical behaviours of the composites are not influenced by the fibre stacking arrangement.

Silane treatment

Asim et al. (2018) have studied the effect of the silane treatment on kenaf and PALF fibres to improve the performances of hybrid kenaf/PALF in phenolic-reinforced composites. Both fibres are dipped into 2% silane solution for 3 h. A notable improvement is observed in the dynamic mechanical behaviours of hybrid phenolic composite at treated fibres at 7:3 of PALF and kenaf fibre, showing the highest E' and E''. This finding is clearly due to the PALF that provides increased stiffness and enhanced interfacial adhesion of fibre–matrix after treating both fibres with silane.

Cho et al. (2009) have stressed that the chopped kenaf fibre is treated with three distinct silane treatments, i.e. 3-glycidoxypropyltrimethoxy silane (GPS), 3-aminopropyltriethoxy silane (APS) and 3-methacryloxypropyltrimethoxy silane at 0.5 wt. %, 1.0 wt. % and 3.0 wt. %, respectively. The composite laminate is made up of thermosetting (PP) and thermoplastic (unsaturated PE [UPE]). Cho et al. (2009) have exposed that the silane treatment by using 0.5 wt. % GPS for 2 h of immersion increases E' value of the kenaf/PP and kenaf/UPE composites.

Lee et al. (2009) have studied the influence of GPS (1, 3 and 5 pph) on the dynamic mechanical performance of the long bast kenaf fibre–PLA composite by using the carding process. Lee et al. (2009) have indicated that 3 pph GPS-treated kenaf biocomposites with 1:1 kenaf to PLA ratio formulation are observed with optimal E' results. The silane treatment by using GPS as a coupling agent can enhance the viscoelastic properties in the kenaf/PLA green composite.

According to Sis et al. (2013), the E' of kenaf/PLA/PBAT/composites tremendously increases when the kenaf fibre is modified using 2 wt. % (3-aminopropyl)trimethoxysilane (APTMS). Thus, 2 wt. % APTMS is used to treat the fibre by washing out the hydroxyl components and improve the compatibility of hydrophilic fibre with hydrophobic resins. Subsequently, the tan δ values of treated kenaf/PLA/PBAT biocomposites decrease. This result is attributed to the limited molecular flexibility of the polymer chain because of the vicinity of the fibre surface caused by chemical bridging at their interfacial interaction (Ibrahim et al. 2010; Kumar et al. 2010).

Acetylation

Datta and Kopczyńska (2015) have implied that the greatest of E' values kenaf fibre-based polyurethane composites are exhibited by samples with short kenaf fibre subjected to acetylation (1385 MPa) followed by those subjected to permanganate treatment (1046 MPa). Moreover, the T_g values of both treated kenaf composites are lower than those of pure kenaf composites because the treated composite has improved capability for irreversible viscoelastic dissipation of mechanical energy (Prisacariu 2011).

Chung et al. (2018) have studied the effect of time of immersion (0.5, 1, 2 and 3 h) into 28.5 vol. % acetic acid on the behaviours of kenaf/PLA biocomposites. Results display a detrimental improvement in the storage modulus (E') at 2 and 3 h of immersion into acetic acid, which strengthens the kenaf/PLA composites. This observation is due to the improved bonding strength because the hydrophilic characteristics of the kenaf fibre are altered. The tan δ of biocomposite containing kenaf significantly improves for 2 h and longer because the low mobility of the polymer chain improves the interfacial adhesion.

Combined alkaline–silane treatment

Huda et al. (2008) have reported that alkali- and silane-treated kenaf fibre composites with 50 vol. % fibre offer a 161% increase in E' compared with other kenaf/PLA composites due to the 3-APS pretreatment, which hydrolyses the OH group of the kenaf fibre by their ethoxy group to form silanol, thereby allowing the fibre to form stable covalent bonds to the cell wall (Dupraz et al. 1996). Subsequently, this treatment enhances the degree of crosslinking in the interfacial region of fibre–matrix and offers a suitable bonding result along with the creation of high-fibre surface area (Herrera-Franco et al. 1997; Singh et al. 1998). The application of sodium hydroxide solution also aids in the fibre compatibility with the matrix by removing the lignin, which contributes to the change in hydrogen bonding profiles to lower the tan δ of the individual composite.

Mokhothu et al. (2011) have investigated the behaviours of a biodegradable aliphatic–aromatic copolyester (CP) mixed with kenaf fibre. This study focuses on the influence of silane concentration (3%, 6% and 9%) in combined alkali–silane on the thermal and the dynamic mechanical behaviour of CP–kenaf composites. Results show all silane-treated composites show an almost similar E' with their modulus value between the untreated kenaf and the single treatment of alkali. Outcomes depict that the silane–alkali-treated composites show two relaxation peaks, whereas the neat CP polymer and the kenaf composite subjected to single-alkali treatment have only one relaxation peak because the combination of alkali and silane treatments results in a semicrystalline composite.

Other chemical treatments

Grafting

Bakar et al. (2015) has concluded the highest E'' values of PVA/EVA polymer with the occurrence of grafted kenaf fibre at 30% loading. The T_g of PVC for the composite comprising grafted kenaf–PMMA composite has shifted to a higher temperature compared with that for the composite comprising ungrafted kenaf fibre. Moreover, the damping peak of grafted kenaf composites shows a slightly declined tan δ compared with that of the PVC/EVA blend and the ungrafted kenaf composites due to improved interfacial locking between kenaf and PVA/EVA upon grafting.

Amino acid treatment

Krishna and Kanny (2016) have modified the surface of kenaf/epoxy composites by using amino acid treatments, such as glutamic acid and lysine as reagent, for 24 h at room temperature. From their results, the E' value in lysine treatment (2937 MPa) is found higher followed by that in glutamic acid treatment (2791 MPa) in comparison with neat resin at the temperature in the plastic region. Additionally, the presence of amino acid-treated kenaf fibres has dramatically reduced the tan δ value. These observations are noticeable due to the good effect or performance of the amino acid especially lysine on the fibre–matrix compatibility, enhancing the stress transfer and improving the fibre–matrix interfacial adhesion.

Zein treatment

John et al. (2010) have researched nonwoven kenaf/PP composites with fibre immersed in 2% zein solution for 2 h. In their study, the zein solution is obtained from corn gluten meal via the wet milling process. Results show that the dynamic mechanical behaviours especially E' have increased in treated 30% kenaf composite. Moreover, the magnitude of the damping parameter in this experiment seems to have declined when the composite is chemically modified.

Conclusion

The kenaf fibre is one of the suitable or potential alternatives to conventional-based fibres in the development of composite material. The kenaf fibre has numerous advantages, e.g. energy saving and low cost of raw material and production in comparison with the current man-made fibre especially in their manufacturing. Kenaf is also a low-density fibre, which contributes in low-weight composite laminate. The surface modification of fibre is introduced in the fabrication of composite laminate to improve their material performance especially their dynamic mechanical properties. Common chemical modifications include *alkalisation*, acetylation and silane treatments. Several other treatments, such as grafting, immersion in zein solution, immersion in amino acid solution and combined treatments, have also been conducted on the kenaf fibre. Amongst the various chemical treatments, alkalisation appears to be the most efficient and cost-effective technique to improve the dynamic mechanical performance and the stiffness of the material.

The alkalisation (alkali treatment) on kenaf fibre aids in increasing the surface area of the fibre via the fibrillation effect as the process splits the single-fibre bundle into small ones. Hence, this treatment escalates the effective area for the mechanical interlocking between the two phases of composites, which are fibre and polymer, and subsequently leads to increased interfacial loading, which contributes to improve dynamic mechanical properties. Acetylation reacts with the cell wall hydroxyl groups in lignocellulosic materials via an acetic compound. In this case, the amorphous and the crystalline components react with acetic acid to form a covalent bonding by using hydrogen linkage to decrease the hydrophilicity of the kenaf fibre. Hence, acetylation improves the bonding of kenaf fibre with the polymer in the composite laminate, improving the storage of the composite. Acetylation reacts with the cell wall hydroxyl groups in lignocellulosic materials via an acetic compound. In this case, the amorphous and the crystalline components have reacted with the acetic acid reagent to form a covalent bonding by using hydrogen linkage to decrease the hydrophilicity of the kenaf fibre, thereby improving the kenaf fibre bonding with the polymer in the composite laminate and the storage modulus of the composite. Other treatments, such as grafting, zein and amino acid treatments, also seem to increase the fibre surface and the mechanical interlocking between the fibre and the matrix and the dynamic mechanical behaviours of kenaf fibre-reinforced polymer composite. For the combined treatment of alkali and silane, the reaction does not provide a significant improvement in terms of storage and loss moduli and damping parameter. Further studies are necessary to compare the combinations of treatment sequences as a function of the formed kenaf fibre and the effects of treatments on the physical, mechanical, dynamic mechanical, thermal and chemical properties of the kenaf composite.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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