



Review

An examination of recent research of water absorption behavior of natural fiber reinforced polylactic acid (PLA) composites: A review



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ABSTRACT

Researchers have begun focusing on developing biodegradable materials, such as natural fiber/polymer composites (NFPC), since the growing of environmental concerns related to waste management. One crucial aspect that must be established in the development of these composites is their water-absorption behavior. This paper examines the water absorption (WA) behavior of NFPC, with a specific emphasis on natural fiber/polylactic acid (PLA) composites. It discusses processes and numerous aspects related to this behavior, based on recent published research. This review analyzes the influence of several factors, such as the loading of natural fiber, the combination of different natural fibers, the methods used in manufacturing, and the temperature of the water, on the WA behavior of natural fiber/PLA composites. It also explores how WA affects the properties of these composites. In addition, this review also presented techniques for improving the WA resistance of the composites. This review paper provides researchers with insights into the WA behavior of the composites, aiming to facilitate the development of a versatile and eco-friendly material that may effectively address waste disposal challenges.

1. Introduction

Currently, environmental issues associated with disposal of fossil fuel-based plastics are very alarming. According to Aliotta et al. [1] 90 % of plastic wastes generated from 1950 to 2015 was not recycled, but rather they were disposed either in the land or the seas. Biodegradable materials offered solutions to the current problem. One of the important biodegradable materials is natural fiber/polymer composites (NFPC). Mahir et al. [2] stated the NFPC industry area reached 2.1 billion USD in 2010, and from 2011 to 2016, the NFPC is predicted to grow 10 % around the world. Researchers have been increasingly interested in developing biodegradable polymers due to growing concerns about material resources and the disposal of plastic [3]. Various type of biodegradable polymers, both natural and synthetic, are currently offered; among them are starch, chitin, bio epoxy resin (derived from

vegetable oil, glycerol, sorbitol, itaconic acid, terpenoid, and lignin), bio benzoxazines (derived from lignin, cardanol, urushiol, and furfurylamine), bio phthalonitrile (derived from eugenol and guaiacol), polysaccharides, polylactic acid, polylactic-co-glycolic acid, polyethylene glycol, polyhydroxy butyrate, and polycaprolactone [4–15], as illustrated by Fig. 1. These materials can be utilized as matrix for the advancement of composites [16]. Among them, polylactic acid (PLA) has been recognized as the most favorable biopolymer with the capability to substitute conventional engineering and commodity plastics in many applications, and it is considered a promising eco-friendly substitute for plastics derived from petroleum [17,18], because PLA is a versatile and extensively utilized biopolymer that comes under the category of aliphatic polyesters, and it is produced through the ring-open polymerization or condensation polymerization of lactic acid, which is obtained from the fermentation of renewable agricultural raw

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materials or biomass, such as starch, corn, soy protein, sugar beet, etc. [16,18–21]. It has attracted interest due to its renewability, bio-based nature, biodegradability, biocompatibility, excellent mechanical and thermal performance, high transparency, and compostable as an aliphatic polyester in comparison to other renewable polymers [16,19,22,23].

In the dynamic realm of sustainable materials, PLA has emerged as a prominent area of research in the materials industry and has found extensive applications, such as packaging, automotive, electronics, furniture and other sectors [24–27]. However, PLA presents numerous limitations, such as its brittle, poor thermal resistance, and lack of electrical conductivity, and these factors greatly limit its extensive application in various sectors, and PLA is also relatively expensive, but this issue can be addressed by reinforce PLA with natural fiber [28–33]. Hence, the incorporation of natural fiber with PLA to produce degradable composites with superior properties has emerged as a major area of research [24]. Natural fiber has many advantages which is availability, low price, light weight, biodegradable, good tensile strength and modulus of elasticity, and low energy consumed [24,34–41]. According to Suriani et al. [42], the price of natural fiber ranged from 400 to 4000

USD per ton, which is cheap, and the reason why it is attracting, and it is cheaper compared to synthetic fiber, which ranged from 980 to 20,000 USD per ton. Multiple sources can provide natural fiber, such as plants or cellulose including bast, seed, leaf, grass, kernel, and even roots and wood, also from animals and minerals, as shown in Fig. 2 [43–52].

Numerous works of incorporating PLA has been carried out, such as PLA reinforced with sisal [17], banana [30], flax [53], sugarcane [54], hemp [55], lemon grass [56], agave [57], pineapple [58], jute [59], kenaf [60], bamboo [61], etc. Nevertheless, the incorporation of natural fiber in composites presents a drawback regarding hydrophilicity, resulting a rise in the WA of the composites since the incompatibility between the hydrophilic fiber and the hydrophobic polymer [62,63]. The hydrophilicity of natural fiber limits the use of natural fiber in aquatic environments and water-related applications [64]. However, these issues could be addressed by employing various chemical treatments and surface modification techniques to achieve optimal performance [44,62,63,65,66].

This review paper focuses on the WA behaviors of NFPC, with specified emphasis on natural fiber/PLA, as reported in recent publications. The objective of this review is to provide a deeper understanding

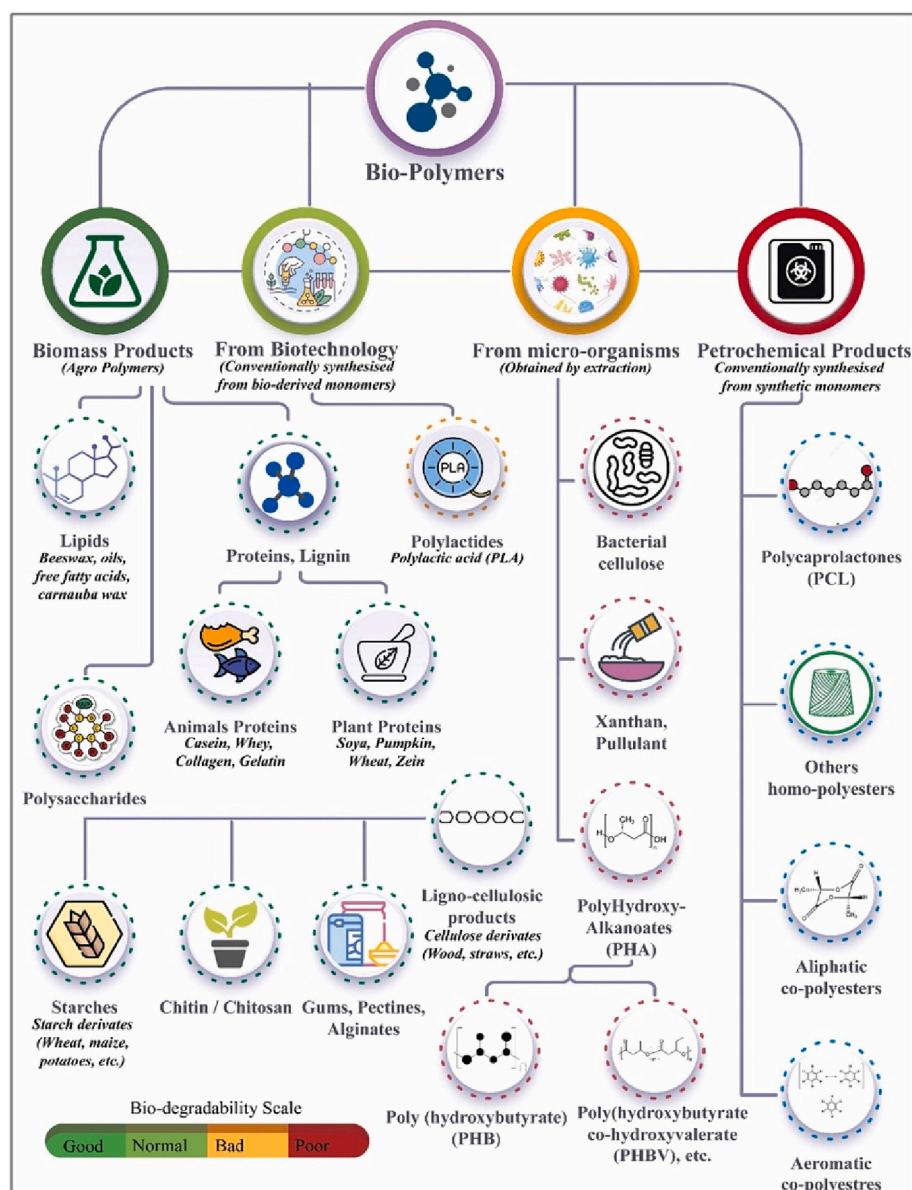


Fig. 1. Classification of different biodegradable polymers and general comparison of their biodegradability [14].

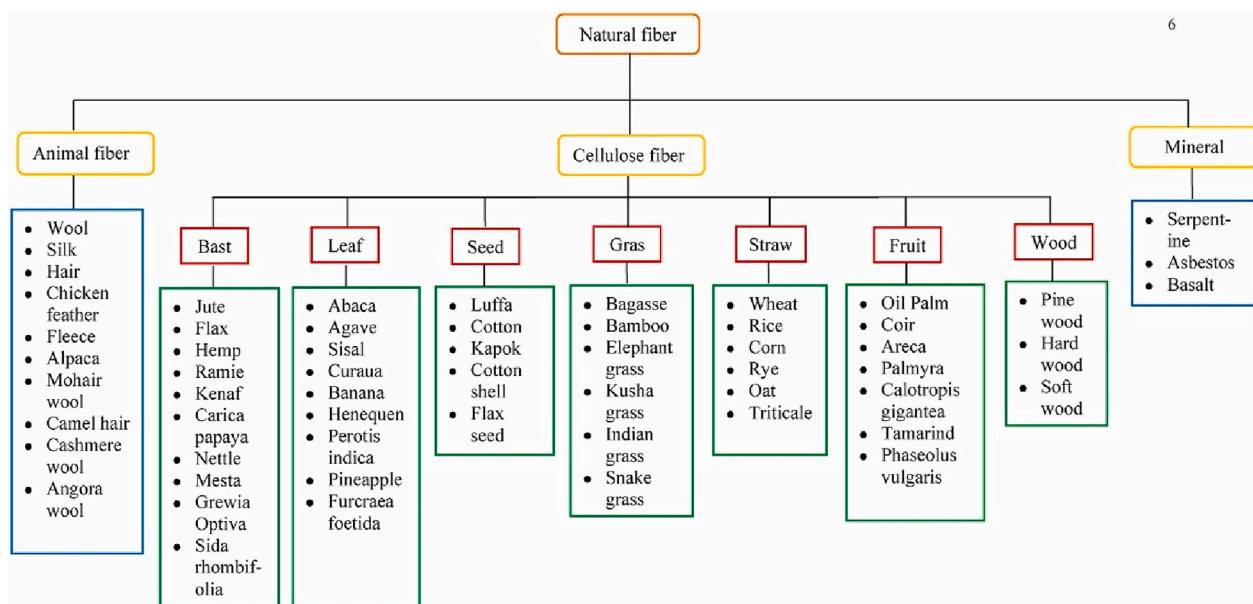


Fig. 2. Classification of natural fibers [43].

of this crucial aspect of composites. By deeply analyzing the internal factors and processes of WA in these composites, how to measure the WA properties, evaluating the influence of natural fiber loading, natural fiber hybrids, manufacturing technique, temperature, and water immersion on their properties, and exploring potential solutions through treatment to mitigate the issue. By utilizing this knowledge, researchers can optimize their usage and explore their potential for other sustainable applications.

2. Water absorption behavior of natural fiber reinforced polymer composites

The research on NFPC has gained significant interest, since it have numerous advantages, including their lightweight, biodegradability, sustainability, low cost, outstanding mechanical properties, non-toxicity, and most importantly, their environmental friendliness [67–76]. Moreover, the utilization of NFPC has several promising prospects in numerous area, such as packaging, construction, industrial, sports, aerospace, and notably, the automotive industry [77–83]. However, incorporating natural fiber in composites has a prominent, which is the hydrophilic of the natural fiber [84–88]. Natural fibers consist of several chemical compositions, which are cellulose, hemicellulose, lignin, wax, ash, pectin, and moisture content [89]. Manaila et al. [90] stated that due to the hydrophilic nature of natural fiber, -OH groups of cellulose and hemicellulose play an important role in the rise of WA due to the formation of hydrogen bonds between water and -OH groups. A greater content of hemicellulose in natural fiber affects the WA ability in natural fiber [65]. According to Moudood et al. [85], the amorphous substances of cellulose and hemicellulose allow water molecules to enter the natural fiber and cause the intermolecular hydrogen bonds to break. On the other hand, the crystalline regions prevented water from diffusing. Chen et al. [91] stated surface treatment can be done in the fiber to remove amorphous substances and create new crystalline regions. The inherent qualities of natural fiber can result in significant WA in composites, leading to insufficient bonding between the fiber and polymer, weak mechanical performance, thermal stability and microbial resistance, inadequate wettability, and diminished homogeneity [92–97]. The WA of various natural fiber could be seen in Table 1.

Mohammed et al. [99] determined that the WA properties of NFPC are affected by many internal variables, as illustrated in Fig. 3. The

Table 1
Properties of various natural fiber [98].

Fiber type	Density (kg/m ³)	Water absorption (%)
Sisal	800–700	56
Roselle	800–750	40–50
Banana	950–750	60
Date palm	463	60–65
Coconut	145–380	130–180
Reed	490	100

variables include the natural characteristics of the composites, fiber loading and alignment, matrix type, fiber-matrix interaction, surface area of the composites, presence of voids, lumen size, and the humidity and the immersing medium temperature. Moreover, the surface of the composites also influences their WA properties.

The consequences of WA properties on NFPC are a major issue for many users of natural fiber. The absorption process significantly compromises the bonding between the polymer and fiber to a considerable extent. Consequently, the composites strength is diminished in such a way that is contingent upon the absorption duration or nature. Additionally, it is seen that this phenomenon contributes to inadequate stress transfer, ultimately leading to the fracture of the specimen over a period of time [100]. This is because natural fiber exhibits a notable affinity for WA attributed to their natural hydrophilicity. The presence of hydrophilic properties can result in the absorption of moisture from the surrounding and the creation of a relatively poor link between the fiber and polymer, resulting the composites degradation and the development of microcracks [99,101].

Fig. 4 illustrates the three primary mechanisms that enable NFPC to absorb water: diffusion, capillary action, and water molecule transport. Diffusion is a randomized phenomenon in which water molecules migrate from areas of high to low concentration area. Composites have diffusion behavior that can be described by both Fickian and non-Fickian diffusion models. Diffusion takes place in the micro-gaps between polymer chains, and the hydroxyl groups in the fiber facilitate the water diffusion to the composite. Nevertheless, capillary action occurs in the gaps between the polymer and fiber when the fiber is not fully incorporated with the matrix during production and has low wettability. Consequently, the more natural fiber amount in composites leads to an enhancement in WA since the increases in the interface area between the

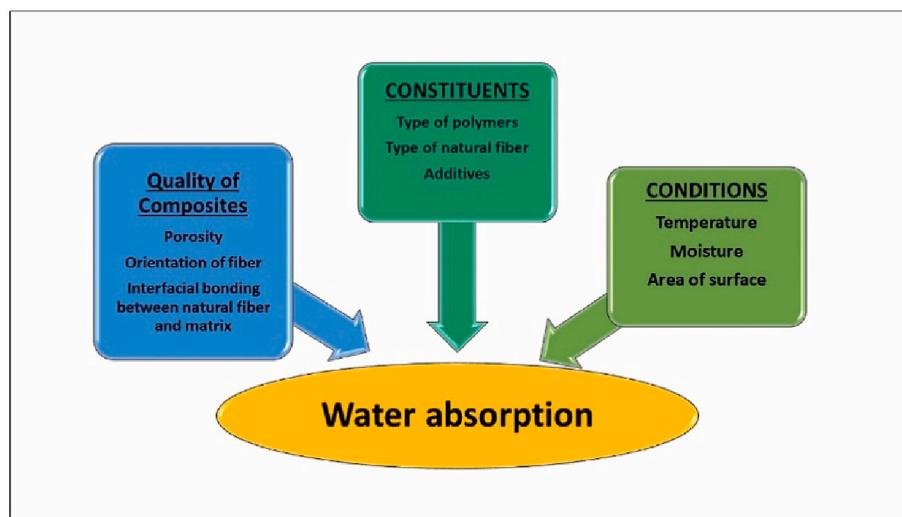


Fig. 3. Influence of water absorption on the efficiency of natural fiber composites [99].

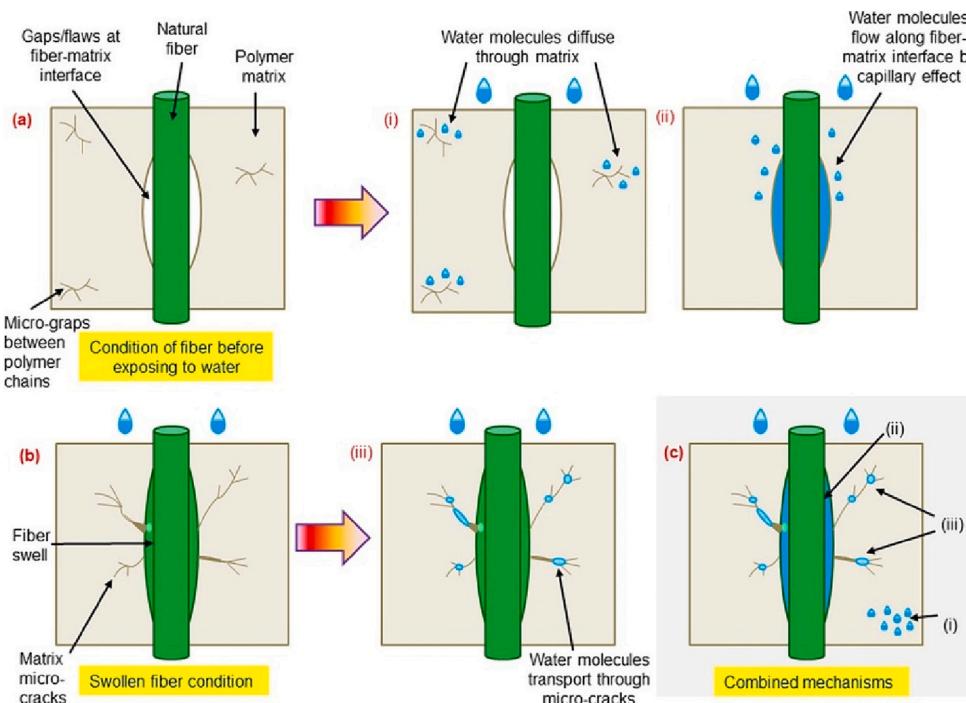


Fig. 4. Probable water absorption mechanism. (i) Diffusion of water molecules through polymer chains, (ii) capillary flow of water molecules along the fiber–matrix interface, and (iii) transport of water molecules through micro-cracks [103].

fiber and matrix, facilitating greater diffusion into the composites. Another mode of transportation happens from the microcracks that form in the matrix, since the expansion of fiber caused by WA, and the water that is absorbed begins to gradually dissolve and separate from the fiber. This results in the fiber debonding from the matrix, resulting reduction in the mechanical performance of NFPC [102–104].

3. Water absorption behavior of natural fiber reinforced PLA composites

The WA behavior of natural fiber/PLA is similar to that of NFPC. According to Avci et al. [105], the water absorbs in various ways, which include entering the available space within the polymer and by capillary action along the fiber. As seen in Fig. 5, when the flax fiber comes into contact with water, the capillary effect of the hydrophilic fiber causes



Fig. 5. Capillary effect of the biocomposites [105].

the water to be immersed in the PLA composites. This situation results in the detachment of surface fiber, the presence of fracture cavities, and the pulling out of flax fiber from the matrix. Moreover, the capillary effect negatively impacts the matrix and fiber bonding. Additionally,

microcracking within the matrix might result in decreased load transmission from the matrix to the fiber.

Chen et al. [24] explained there are two main ways of wheat straw/PLA composites immersed water. First, the hydroxyl groups existence on the fiber surfaces, resulting strong WA of the fiber. Second, they provided gaps between wheat straw fiber and PLA which allowed water to immerse in the PLA composites.

The WA in natural fiber/PLA composites can be determined using ASTM D570 [106–109] or ASTM D570-98 [104,110,111] standard test method. The WA or water uptake by natural fiber/PLA composites is obtained using Eq. (1) [112–115]:

$$M_t(\%) = \left(\frac{W_t - W_0}{W_0} \right) \times 100 \quad (1)$$

where W_t is the sample weight at different time and W_0 is the weight before immersion.

Fickian diffusion is a mechanism capture the overall WA behavior of composites [116]. The sorption of the natural fiber/PLA composites could be obtained using Eq. (2) according to the Fick's law [112,116].

$$\frac{M_t}{M_m} = k \cdot t^n \quad (2)$$

where M_t is the WA at time (t), M_m is the saturated sample weight of the sample, and k and n are the diffusion kinetic parameters. In the earlier stages of WA, diffusion was managed the WA. The WA adheres to the Fick's law of diffusion if $n=0.5$.

The mass gain correlated with the diffusion coefficient (D) and the maximum WA (M_t) could be obtained with two equation, if $M_t/M_m < 0.6$ using Eq. (3) [112], and if $M_t/M_m > 0.6$, the curve of absorption is calculated with Eq. (4) [112].

$$\frac{M_t}{M_m} = \frac{4}{h} \left(\frac{D}{\pi} \right)^{1/2} \cdot t^{1/2} \quad (3)$$

$$\frac{M_t}{M_m} = 1 - \exp \left[-7.3 \left(\frac{D \cdot t}{h^2} \right)^{0.75} \right] \quad (4)$$

where D is the diffusion coefficient and h are the composites thickness. The D could be determined using Eq. (5) [112,117].

$$D = \pi \left(\frac{kh}{4M_m} \right)^2 \quad (5)$$

where k is the slope of the initial linear portion of absorption curve.

Thickness swelling also can conducted to observe the water resistance of the natural fiber/PLA composites [118]. The thickness swelling test is obtained using the following Eq. (6):

$$\text{Thickness Swelling}(\%) = \left(\frac{T_1 - T_0}{T_0} \right) \times 100 \quad (6)$$

where T_0 is the thickness before absorption and T_1 is the thickness after absorption.

Contact angle measurements also can be conducted to determine the

hydrophilicity or hydrophobicity or surface wettability of natural fiber/PLA composites [24,119–123].

3.1. Influence of natural fiber content in natural fiber reinforced PLA composites

Some works have been done to determine the influence of fiber content on the WA of natural fiber/PLA composites, as shown in Table 2. According to Kumari et al. [117] the impact of fiber contents, which is 0 %, 10 %, 20 %, 30 % in WA of sisal fiber/PLA composites is risen dramatically from 4.66 % to 23.36 %. This happened because fiber have a hollow part which make water absorbed to the fiber. It makes the increases of fiber make more water absorbed due to more surface area for water to absorbed, and the hydrophilic of natural fiber. Tengsuthiwat et al. [110] also work on sisal/PLA composites. In this study the amount of sisal fiber used are 0, 5, 10, and 20 phr. After 300 h immersion, the WA of pure PLA composite is 0.7 %, and for 5, 10, and 20 phr fiber loading in composites, representing 1.4 %, 3 %, 4 %, respectively. The study declared the increase of WA rate occurred was due to cellulosic nature of sisal fiber.

Rajesh et al. [124] examined the relation between the increase in WA with the increase in jute fiber content in jute fiber/PLA composites. The WA in composites with a 25 % jute fiber content was 11 times higher than in pure PLA composites. The jute fiber's hydrophilic characteristic is responsible for its significant WA capacity.

Gunti et al. [125] carried out a work on elephant grass/PLA composites. The composites were prepared using fiber loading of 0, 5, 10, 15, 20, and 25 % in the study. Fig. 9(a) demonstrates that the higher fiber loading in the composites leads to an elevated rate of WA. The composites with a loading of 25 % elephant grass exhibit the greatest WA, reaching 11.17 %, in comparison to the neat PLA, which absorbs just 0.88 %. The investigations indicate that the primary reason for the rise in WA capacity is attributed to the hydrophilic characteristics of elephant grass. This is because the fiber of elephant grass possesses a significant amount of hydroxyl groups.

3.2. Influence of hybridization of natural fibers in natural fiber reinforced PLA composites

Multiple investigations have been done to investigate the impact of using natural fiber hybrids in the WA of natural fiber hybrid/PLA composites, as illustrated in Table 3. Eselini et al. [127] investigated the WA behavior of basalt and flax fiber/PLA for 30 days. The studies indicate that incorporating flax fiber with PLA leads to a significant increase in WA, around 33 %, since the hydrophilic characteristic of the flax fiber. The WA of basalt fiber/PLA is lower compared to flax fiber/PLA composites, and the WA value is nearly equivalent to that of pure PLA. The authors state that adding flax fiber to the fiber-hybrid/PLA composites made them absorb more water. This is because basalt fiber does not affect WA because it is hydrophobic.

In the work reported by Siakeng et al. [128], the authors performed WA tests on PLA composites reinforced with either coir or pineapple leaf fiber (PALF) and a hybridization of coir and PALF. The coir-fiber/PLA composites have the greatest WA in comparison to the other

Table 2

Influence of natural fiber content in water absorption behavior of natural fiber reinforced PLA composites.

Fiber	Fiber loading	Water absorption testing			Water absorption result		Ref.
		Solution	Period	Standard	Water absorption (%)	Diffusion coefficient	
Sisal	0, 10, 20, 30 (%wt)	Water, RT	35 days	BS EN ISO 62:1999	4.66–23.36	–	[117]
Sisal	0, 5, 10, 20 phr	Distilled water, 30°C	300 h	ASTM D570-98	0.7–4	3.1 E – 01 – 8.9 E – 01 mm ² /h	[110]
Jute	0, 5, 10, 15, 20, 25 %	Deionized water, 23 ± 1°C	30 days	ASTM D570	0.97–10.82	1.4574–2.6420 mm ² /s	[124]
Elephant grass	0, 5, 10, 15, 20, 25 %	Water, 23°C	48 h	ASTM D570-98	0.88–11.17	–	[125]
Wood flour	0, 15, 30 (%wt)	Tap water, RT	7 days	–	~0–21	–	[126]

RT: Room Temperature.

Table 3

Influence of natural fiber hybrids on the water absorption behavior of natural fiber hybrid reinforced PLA composites.

Fiber	Fiber loading	Water absorption testing			Water absorption result		Ref.
		Solution	Period	Standard	Water absorption (%)	Diffusion coefficient	
Basalt/ Flax	30/0, 5/25, 10/20, 15/15, 20/10, 25/5, 0/30 (wt %)	Water, RT	30 days	ASTM D570	~2–33	–	[127]
Coir/ Pineapple leaf	30/0, 15/15, 9/21, 21/9, 0/30 (wt%)	Distilled water, RT	7 days	–	~2–10	–	[128]
Jute/ Coir	0, 40 % (wt%)	Distilled water, RT	180 h	ASTM D570	~1.5–20.2	–	[129]

RT: Room Temperature.

composites. On the other hand, the composites exhibit the least WA when using PALF as reinforcement. The WA rate of hybridized PLA composites increased when higher loadings of coir fiber were used as reinforcement. This occurred as a result of the high porosity and hollowness of the coir fiber.

Hossen et al. [129] observed the WA on jute and coir fiber hybrid/PLA composites for 160 h. The jute and coir fiber hybrid/PLA composites show 20.2 % water immersion compared to pure PLA, which is lower than 1.5 %. The higher water immersion in the hybrid/PLA composites is due to voids and pores that consume more water, and the existence of hydroxyl groups. The higher water absorbed in the composites contributed to reduce bonding between the fiber and the polymer.

3.3. Influence of different processing techniques in natural fiber reinforced PLA composites

Siva et al. [130] investigated the WA behavior of hemp fiber/PLA composites with different composite manufacturing processes and different types of water, such as distilled water, normal water, and sea water. After 75 h of immersion, the WA percentage in distilled water is 2.80 % for injection molding composites and 2.41 % for extrusion injection molding composites. In normal water, the WA rate for injection molding is 2.34 %, whereas for extrusion injection, it is 2.23 %. The WA rates for sea water are 2.53 % for injection molding and 2.33 % for extrusion injection molding. The study concluded that this occurred as an effect of the void content in the samples, which is created during the manufacture of the samples. According to Table 4, The void content of injection-molded samples has a higher void percentage, which is 4.76 %, than extrusion-injection-molded samples, which is 3.62 %.

A study by González-López [109] conducted coir fiber/PLA biocomposites with injection-molded manufacturing. In this study, PLA and 30 % coir fiber were first mixed in a twin-screw extruder, then cut to form pellets. The pellets were subjected to injection molding at a 30 °C mold temperature the screw temperature was 175/185/190/185 °C. The WA rate of the injection-molded biocomposites was measured. The results revealed that the WA rate of coir fiber injection-molded composites was 12 %. Compared to Siakeng et al. [128] who used the hot press manufacturing method with the same fiber fraction, which is 30 %, the WA rate of the biocomposites coir fiber/PLA composites is only 10 %, which is lower than the González-López [109] study.

Singh et al. [131] investigated the effect of different curing temperatures on the production of jute/PLA composites with compression molding. This study used three different curing temperatures, which were 160 °C, 170 °C, and 180 °C. The result showed that the WA value of the composite declined along with the increases in curing temperature.

Table 4

Void content of injection and extrusion-injection molding [130].

Composite preparation method	Void content (%)
Extrusion-injection molding	3.62 %
Injection molding	4.76 %

With 50 % fiber loading, the composites absorb around 17 % at 180 °C, which is lower than 160 °C and represents about 20 % of the WA value. The study stated that the curing temperature of the manufacturing process has an effect on the interfacial bonding of the composites.

Ecker et al. [126] examined how different ways of preparing composites affect the WA of PLA composites reinforced with wood flour (WF). The study showed that the 3D-printed PLA composites exhibited higher WA rates and faster WA compared to the composites produced using injection molding. According to the study, this occurrence was attributed to the porous structure of the 3D-printed PLA composite samples, as depicted in Fig. 6.

Omar et al. [132] conducted manufacturing of *agave tequilana* fiber/PLA using the film-stacking method. The manufacturing method had four stages. First, the fiber was spread on the vibration plate until it was well distributed. Next, the fiber mats were pre-impregnated with a PLA/dichloromethane solution and dried. Then, the processed mats were stacked with different fiber contents, which were 20, 40, and 60 %, with PLA films on the metallic frame. Finally, the stacked fiber mats underwent compression molding. With this method, the WA rate of the sample for 20 % fiber loading was about 5.9 %, and it increased along with more fiber loading. The highest WA rate was 60 % fiber loading, which was approximately 12 %.

Ayrlimis et al. [133] investigated the effect of different printing thicknesses on the WA characteristics of a 3D-printed wood/PLA composite. This study employed four distinct printing thicknesses: 0.05, 0.1, 0.2, and 0.3 mm. The result showed that the WA amount of the composite rose along with the increase in printing thickness. The study suggests that this occurred as a result of the composites' increased

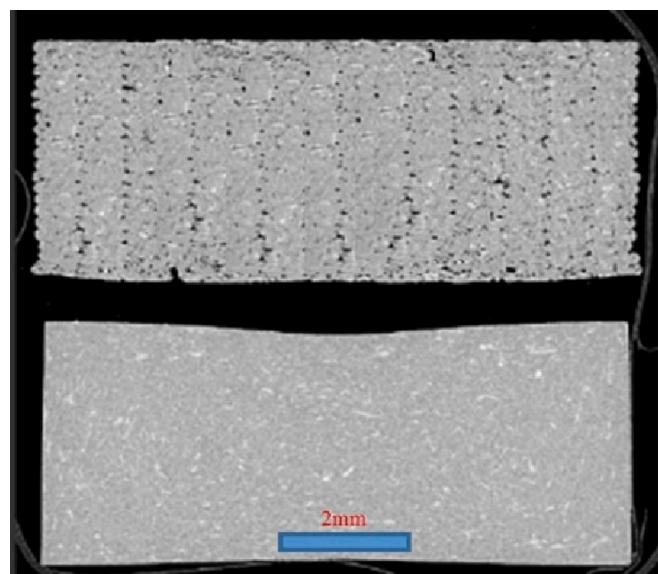


Fig. 6. CT scan of cross section of 3D printed sample (top) and injection molded sample [126].

porosity, bolstered by a decrease in density. As the printing thickness increased, the empty spaces between the filaments grew larger, absorbing more water. Additionally, as the printing layer increased, the surface area of the wood flour became more exposed, which immersed more water due to the hydrophilicity of the wood flour.

Mazur et al. [134] performed a WA test on 3D-printed PLA composites made from PLA filament filled with 30 % wood, bamboo, and cork fiber. In this study, the bamboo-filled composites absorb more water than the other fibers. This phenomenon happened because of the existence of an OH group since natural fiber is hydrophilic, as well as the higher amount of water immersed in the samples because of the micro-gaps in the composites, which allow water to be immersed in the composites. On the contrary, cork-filled composites absorb less water than the other fibers since the cellulose content in the cork fiber, which is 12–25 %, is lower than the other natural fiber-filled composites, which are 40–50 % for wood and 40–55 % for bamboo.

Wang et al. [135] investigated the WA characteristic of 3D-printed cellulose nanofiber (CNF)/PLA composites and used polyethylene glycol 600 to make the material smooth. The authors conducted the study using three different amounts of CNF: 1, 2.5, and 5 wt%. The study revealed that the use of CNF as a filler in composites increases the WA rate due to its surface containing many OH groups and its hydrophilicity. Similarly, the use of polyethylene glycol as a smoothing agent in composites affects the WA rate of the composites due to changes in PLA crystallinities, which can lead to water immersion and bonding with the hydroxyl group in the composites.

Velarde et al. [136] conducted tests on the WA behavior of 3D-printed agave fiber/PLA composites with different fiber loading and printing angles. The study used 0, 3, 5, and 10 % fiber loading and –45°/45° and 0°/90° printing angles. The study demonstrated that the WA amount of the composites rises as the fiber amount rises, due to the existence of gaps, voids, and cracks caused by fiber swelling. Additionally, the OH group of the fiber enhances the hydrophilicity of the composites, thereby influencing the WA rate. In terms of printing angle, the difference in WA rate of the composites is noticeable in the 10 % fiber loading, which at 0°/90° printing angle absorbs more than –45°/45° printing angle.

Pereira et al. [137] reported the effects of adding different loading of rice husk fiber on the WA characteristic of PLA composites manufactured with fused deposition modeling. For the WA test, the study used three different loadings of fiber, which were 0, 5, and 10 % contained in the PLA composites. The results revealed that composites with 5 % fiber loading had the highest WA value. On the contrary, the WA rate of 10 % fiber-filled composites is lower compared to 5 % fiber-loaded composites. The study stated that this occurs due to random fiber distribution in the filament and printed composites, as well as printing defects that can generate voids in the composites.

3.4. Influence of different water temperatures in natural fiber reinforced PLA composites

Jiang et al. [112] conducted work on the different WA behaviors of jute/PLA composites at different water temperatures. The composites are immersed in water at 50 °C and 60 °C. In the study, the water temperature is affected by the water immersed in the PLA composites. According to 20 days of water immersion in the study, the 60 °C jute PLA composites have about 4 % WA than the 50 °C composites, which is approximately 2.3 %. The diffusion coefficient also increases due to higher temperatures. This happened because the glass transition temperature (T_g) of PLA (55–59 °C) lower than water temperature.

3.5. Influence of fillers and microfibers in natural fiber reinforced PLA composites

Avci et al. [105] investigated the effect of the addition of boron compounds, which are borax: boric acid (1:1), zinc borate, and ulexite,

on the WA value of flax fiber/PLA composites. In the study, 3 and 5 % ratios of boron compounds were used for each type of boron filler in the biocomposites. The boron fillers were added during the mixing stage in a twin-screw extruder. The result showed that the WA number rose with the addition of boron fillers. The rise in the WA number is due to the hydrophilicity of boron fillers, which easily absorb water. The study stated the highest amount of WA is in the ulexite-added biocomposites. This phenomenon happened because of the chemical composition of ulexite, which included Ca and Na, which tend to absorb water.

Ramesh et al. [138] performed WA properties testing on kenaf/PLA composites with different amounts of montmorillonite clay, which are 1, 2, and 3 wt%. The filler was added to the fabrication of biocomposite in the mixing through twin screw extruder phases. The study stated that the addition of montmorillonite clay in the manufacture of biocomposites could reduce the WA value of the composite. This is due to the fact that the montmorillonite clay in the biocomposites prohibits the immersion of water into the biocomposites or acts as a barrier agent in the biocomposites.

Ramesh et al. [139] conducted a study on the addition of montmorillonite clay as a filler in the hybridization of kenaf and *aloe vera* fiber/PLA. In the study, 1 and 3 wt% filler was added in the pre-mixed stages. The finding stated that the addition of montmorillonite clay as a filler in the composites decreased the WA value of the composites. This is due to the increasing water resistance of the composite since the filler restricts water immersion in the composites. The best filler amount is 3 wt% used as the filler in the composites.

Silva et al. [111] conducted a study on the WA characteristics of eucalyptus microfibers/PLA composites with 3 different types of humidity, which are 33 %, 59 %, and 85 % at room temperature. The study stated that using microfibers is good for the characteristic of the composites because of their good compatibility. The result showed that the pure PLA composites did not immerse water in every variation of humidity. However, the incorporation of eucalyptus microfibers in PLA composites increased the WA value. This phenomenon happened due to the hydrophilicity of the eucalyptus microfibers, and the higher WA occurred because of the presence of microvoids and microcracks.

3.6. Comparison of natural fiber reinforced PLA composites with other kind of biocomposites

Cervantes et al. [140] conducted a study on agave fiber reinforced with both polyhydroxy butyrate (PHB) and polyhydroxy valerate (PHBV) as the biopolymer matrix. In this study, the researcher used 20 % agave fiber loading in the fabrication of the biocomposite and conducted tests on the WA characteristic of the biocomposites. The study stated that the WA of biocomposites of both polyhydroxy butyrate (PHB) and polyhydroxy valerate (PHBV) reinforced with agave fiber was 6.6 % for both biopolymers. In contrast to Lopez et al. [106] studied on agave/PLA biocomposites, this study reveals that agave/PLA biocomposites with 20 % agave fiber loading absorb 21 % of the water. Zhuo et al. [141] studied the WA behavior of bamboo/PBHV biocomposite. In this study, the WA rate of the composites is about 2.2 %. In contrast, Li et al. [119] studied the WA behavior of bamboo fibers/PLA. The study showed that the WA value of bamboo fibers/PLA is 6.1 %. It can be concluded that reinforcing natural fiber with PLA absorbed more water than PHB and PHBV. Thiagamani et al. [142] conducted a test on the WA of hemp fiber/green epoxy. The WA value of the biocomposites was recorded at approximately 6.5 %. The water immersion rate was lower than hemp fiber/PLA, which is 23 % compared with Alao et al. [143] studies. Higher value of water immersion in fiber reinforced PLA is because of weak incorporation between fiber and PLA. [144]

4. Effect of water absorption on properties of natural fiber reinforced PLA composites

Ecker et al. [126] reported the influence of WA on the characteristics

of WF/PLA composites. The work investigated different amounts of fiber and production processes. The study discovered that storing WF/PLA composites in water for 7 days, regardless of whether they were 3D-printed or injection-molded, resulted in a decline in their tensile characteristics. According to the study, the use of WF in the composites is responsible for this phenomenon. Conversely, the study found that storing the injection-molded composites in water led to a rise in their impact properties, along with a rise in fiber loading. However, immersion in water resulted in a decline in the impact properties of the 3D-printed composites. The study concluded that the impact properties of the water-soaked injection-molded composites decreased due to the absence of wood particle agglomeration and their good homogeneity, as evidenced by the SEM graphs for these composites. Nevertheless, the absorption in water caused the crystallinity of the composites to increase since the water improved the mobility of the PLA polymer chain. Additionally, the effect of soaking the composites in water on the thermal behavior of the composites is that there is no effect on glass transition, cold crystallization, or melting temperature for both types of fabrication methods.

According to Jiang et al. [112], the impact of WA at 50 °C and 60 °C water temperatures on the jute/PLA composites is reducing tensile and flexural performance of the composite as increasing absorbing time. The decreases in its properties are due to the fiber swelling, which caused residual stress in the composites, resulting in microcracks between the matrix and fiber, as shown in Fig. 7. In this work, the mechanical characteristic of the composites was reduced faster in 60 °C water temperature, since the PLA degradation by high temperature water immersion, resulting in the diffusivity of water to the PLA. But as the immersion time goes up, the crystallinity of jute/PLA composites increases because the jute fiber acts as a nucleating agent and stops hydrolysis of the crystal area. Additionally, the thermal behavior, which were the T_g of the jute/PLA composites, rose until 14 days of immersion, but after that, the T_g suddenly decreased until 28 days immersion time.

Jiang et al. [113] examined the microstructure of the jute/PLA composites after thorough hydrothermal by X-ray tomography. The study concluded that the effect of hydrothermal water immersion in the jute PLA composites is divided into three parts. First, in the microstructure there is no change detected, however, the strength and tensile elastic modulus exhibit a drop, and the ductility increases due to the plastination from water immersion in the composites. Second, differential swelling and weakening of the composite affect the debonding between matrix and fiber, which lowers the mechanical strength of the composites. Last, the ductility fall since the absorbed water in the composites causes microcracking in the composites. In addition, Jiang et al. [145] reported a decline in the mechanical strength of jute/PLA after conducting hydrothermal water immersion in the composites. The decreases occurred due to swelling in the composites, a result of the jute fiber's high number of OH groups, which accelerated the debonding between the fiber and the matrix. Also, PLA hydrolysis makes the

molecular weight of PLA decrease, resulting in a void in the composites. Additionally, the hydrolysis of the fiber's chemical composition weakened the fiber's strength. Moreover, the high temperature allows water to absorb into the matrix, which makes the degradation of PLA faster and cracks on the PLA matrix. The study found that the high temperature of the water had a big effect on the thermal characteristic of the composites. This was because the high temperature made the crystallinity of the PLA chains higher, which caused the cold crystals to loosen and the melting endotherm to rise. Moreover, the T_g of the composite has almost gone along with the increases in time since the PLA chain action was restricted due to the increases in crystallinity. However, at lower temperatures, the T_g decreases in the first phases because of the plasticizing effect of water. In the next phases, the T_g rose along with the increase in crystallinity, but it decreased again due to the reduction in molecular weight. Yu et al. [146] carried out a work in the effect of hydrothermal ageing on the mechanical characteristic of ramie fiber/PLA. The composite was immersed in water at 60 °C. In this study, as the WA time increased, the T_g gradually rose and then suddenly fell. The rises occurred as a result of the enhancements in crystallinity since the limited PLA molecular chain activity. However, the reductions happened because of the decreases in molecular weight and the plasticizing impact on the degradation of PLA, resulting a rise in crystallinity. The mechanical strengths of the ramie fiber/PLA composites diminished as a result of PLA degradation during the ageing process. Additionally, according to the microstructure evolution in the study, cracks in the composites showed after two weeks, which was also the reason for the reduced mechanical characteristics.

Pantalone et al. [147] conducted a work on the consequences of hygroscopic ageing on the characteristic of a flax/PLA composite. The study examines several hygroscopic settings, including relative humidity levels of 50 %, 75 %, and 98 %, and also immersion ageing. The schematic process is illustrated in Fig. 8.

Table 5 shows the tensile strength of the composites under different ageing. The study stated that the three conditions, which were 50_RH_re, 50_RH, and 75_RH, did not have an influence on the tensile performance of the composites. However, the 98_RH and immersion-conditioned composites got a decline in tensile strength and tangent modulus. The author declared that the reduced mechanical strength in both 98_RH and immersion composites are due to the damage caused by the water immersion. Additionally, the microcrack present at the PLA matrix is due to immersion ageing since the fiber swelled water and the stress applied to the PLA matrix, resulting in a decline in density and a rise in porosity of the composites, which is also the reason for the declined mechanical behavior of the composites.

5. Different methods to increase water absorption resistance in natural fiber reinforced PLA composites

Several researchers are exploring methods to enhance the water resistance of PLA composites reinforced with natural fiber. The WA in PLA composites is an important issue that must be solved. The WA by the fiber in the composite could lead to abnormal swelling and dimensional instability, resulting a fall in strength owing to the fiber-matrix interface degradation [124].

5.1. Alkali treatment in natural fiber reinforced PLA composites

To reduce the hydrophilicity of natural fiber, one of the treatment which commonly used is Alkali treatment [148]. Alkali treatment makes the fiber surface rougher, since hemicellulose, lignin, and impurities are eliminated, which enhances the fiber and matrix adhesion [149]. Omar et al. [132] conducted a work to modify the fiber surface by using a NaOH solution. The fiber undergoes the treatment by rinsing it with water, dried, and then soaked for 3 h in 8 % NaOH solution. Afterwards, the fiber is rinsed and dried in an oven. Then, the treated fiber is ready for production as a PLA composite. The PLA composites with 60 wt% of

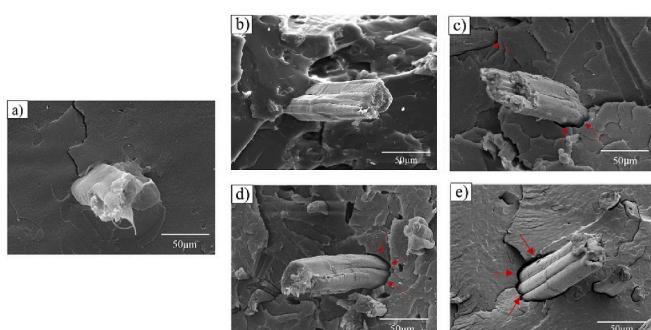


Fig. 7. Interface of jute/PLA, observed by SEM: a) no ageing, b) aged for 7 days at 50 °C, c) aged for 14 days at 50 °C, d) aged for 7 days at 60 °C, e) aged for 14 days at 60 °C [112].

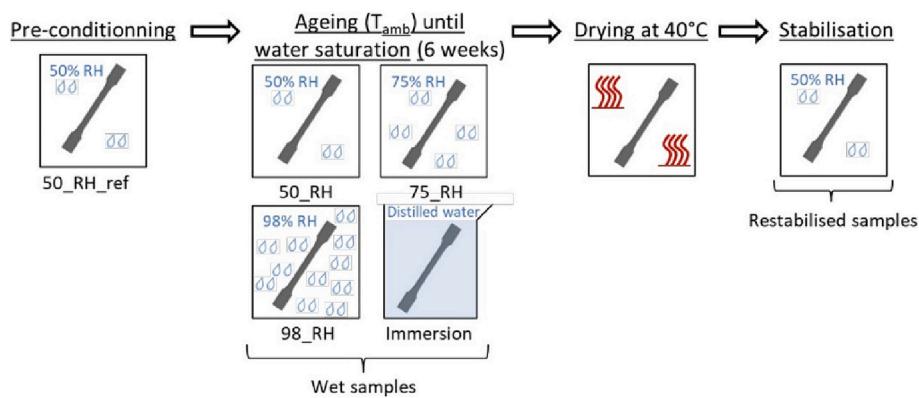


Fig. 8. Schematic representation of the ageing protocol applied to a flax/PLA non-woven composite with a fiber volume fraction of 36 % [147].

Table 5

Tensile properties of flax/PLA composite ($W_f = 40\%$) after ageing under several conditions, drying and reconditioning at 50 % RH (restabilised state) [147].

	50_RH_re	50_RH	75_RH	98_RH	Immersion	Pure PLA (unaged)
Tangent modulus [GPa]	7.4 ± 0.3	7.4 ± 0.6	7.9 ± 0.7	5.9 ± 0.2	4.9 ± 0.4	3.4 ± 0.1
Ultimate strength [MPa]	51.6 ± 3.3	54.3 ± 1.6	51.9 ± 3.0	37.8 ± 1.8	32.3 ± 1.3	37.6 ± 0.8
Strain at failure [%]	1.5 ± 0.2	1.5 ± 0.1	1.4 ± 0.1	1.6 ± 0.1	1.8 ± 0.3	2.6 ± 0.4

treated fiber exhibit a significant decrease in comparison to PLA composites with untreated fiber, amounting to approximately 15 %. This attributed to fiber hydrophilicity decreases, resulting enhanced incorporation between the matrix and the fiber.

Other researchers also conducted research on alkali treatment in natural fiber hybrid/PLA composites. Siakeng et al. [118] reported the impact of alkali treatment on natural fiber hybrid/PLA composites. Both fibers were subjected to an alkali treatment by immersing them in a 6 % NaOH solution for 3 h. Then, the fibers were washed until the pH level reached neutral and then dried for 2 days. The WA test was observed on both untreated and treated coir fiber and PALF hybrid/PLA composites. The untreated fiber-hybrid/PLA with 30 % coir fiber and 70 % PALF composites exhibited the highest WA, measured at 7.36 %. After the fiber treated by alkali treatment, the WA rate of the composites declined since the treatment remove the fiber hydroxyl groups. The lowest WA rate in this work was PLA composite reinforced with 70 % coir fiber and 30 % PALF, measured at 5.24 %.

5.2. Benzoylation treatment in natural fiber reinforced PLA composites

Zulyadain et al. [150] investigated the effect of benzoylation treatment on sugarcane bagasse fiber/PLA on the WA properties. In the study, the fiber underwent alkali treatment first, then was soaked in a alkali solution, and then benzoyl chloride was rapidly added to undergo the benzoyl treatment. The results showed a decrease in the biocomposite's WA following the benzoyl treatment. The reduced WA value of the treated biocomposite is due to the change in OH group amount with the benzoyl group, as showed in the Fourier Transform Infrared spectrum in the study.

Sherwani et al. [151] performed benzoylation treatment on sugar palm fiber/PLA composites. The benzoyl chloride treatment on the fiber was done at different immersion times, which are 10, 15, and 20 min. In this study, the fiber was first pre-treated by soaking it in an 18 % alkali solution. It was then subjected to benzoyl treatment by soaking it in 10 % alkali solution and stirring it with 50 ml of benzoyl chloride. The finding stated the WA value of the biocomposite decreased after underwent benzoyl treatment, with a 15 min immersion time representing 12.33 %. The study revealed that the benzoylation treatment reduced the hydrophilicity of the fiber, leading to a rise in compatibility between the fiber and PLA. The result was also supported by the decrease in void

content and moisture content of the sample after undergone benzoyl treatment.

Mobarak et al. [152] conducted a research on the WA characteristic of sponge gourd fiber/PLA with different percentages of chemical and fiber loading. The result revealed that the WA rate of the benzoyl-treated fiber composites was reduced by 10 wt%, which is the best benzoyl loading in both fiber loading variations. The study indicated that the reduced number of WA is because of the better bonding of fiber and PLA since the hydrophilicity of the fiber decreased.

5.3. Successive alkali treatment in natural fiber reinforced PLA composites

Successive alkali treatment is a two-stage treatment that could lower the hydrophilicity of the fiber. According to Rajesh et al. [124], the jute fiber was treated with an alkali treatment in the first stage. Then in the second stage, the alkali treated fiber was then subjected to a treatment with H_2O_2 (10 ml/l). This treatment involved by soaking the fiber at room temperature for 45 min. Afterwards, the fiber is washed and dried in an oven until a consistent mass was achieved. This treatment method minimizes oxidative damage to the fiber and effectively eliminates fatty compounds, wax, and lignin while preserving the quality of the fiber, and it exhibits a longer shelf life. The outcomes of the treatment of jute fiber in PLA composites are detailed in Table 6. The authors conducted a comparison between the ratio of NaOH in fiber alkali treatment and the impact of varying jute fiber additions to the PLA matrix. The addition of jute fiber is resulting in an enhancement of the diffusion coefficient, as natural fiber possesses natural hydrophilic properties. Nevertheless, the diffusion rate was decreased by the hydrophobicity of the treated natural fiber. Furthermore, the WA of the PLA composites was reduced by a greater concentration of NaOH when 25 % of the jute fiber was used. However, a deviation in the diffusion rate is observed in PLA composites containing 15 % NaOH, as elevated alkali concentrations in the treatment damage the fiber [124].

Gunti et al. [125] also conducted successive alkali treatments in their study, but the differences are that they use elephant grass fiber for the reinforced PLA composites and only use a 10 % NaOH solution for the alkali treatment. Based on the higher fiber loading, which is 25 % elephant fiber/PLA composites, as shown in Fig. 9(a)(b), the WA decreased from 11.17 % to 6.05 % after being treated with successive

Table 6

Equilibrium water uptake M_∞ , initial linear slope K and diffusion coefficient values for PLA and its composites at various fiber weight fractions [124].

Type of composite	% of fiber	M_∞ (%)	K (%/hr ^{1/2})	$D \cdot 10^5$ (mm ² /s)
Plain PLA	0	0.89	0.0906	1.6824
Composite with untreated fiber	5	1.52	0.1325	2.0448
	10	3.42	0.3138	2.0448
	15	5.56	0.2719	2.0228
	20	7.85	0.3905	2.0292
	25	9.93	0.4672	2.0283
Composite with successive alkali treated (5 % NaOH)	5	1.91	0.5927	2.0234
	10	3.16	0.3556	2.0448
	15	4.12	0.3556	2.0448
	20	5.29	0.3835	2.0448
	25	6.51	0.4044	2.0283
Composite with successive alkali treated (10 % NaOH)	5	1.79	0.2092	1.8661
	10	2.83	0.3277	1.9704
	15	3.25	0.2301	2.0448
	20	4.48	0.2370	2.0356
	25	5.57	0.2859	2.0374
Composite with successive alkali treated (15 % NaOH)	5	2.06	0.1952	2.0448
	10	2.80	0.3277	1.9688
	15	2.77	0.2789	1.4842
	20	3.16	0.2650	2.6420
	25	4.44	0.4811	1.4574

alkali methods. The study indicates this happened due to the reaction of treatment, which reduced the hydroxyl group.

5.4. Silane treatment in natural fiber reinforced PLA composites

Multiple works have performed silane treatments on natural fiber to augment the water resistance of PLA composites that are reinforced with natural fiber. Silane (SiH_4) functions as a coupling agent to reduce the quantity of hydroxyl groups present at the interface of the fiber matrix [148], and Applying a silane treatment to the natural fiber enhance the bond between the fiber and polymer at the interface [153]. Chen et al. [24] investigated the hydrophobicity of wheat straw fiber/PLA composites treated with four distinct silane coupling agents: APS, KH-560, KH-570, and KH-590. According to the Fig. 10, the contact angle of four distinct silane-treated fiber composites exhibited an increase, and the WA value exhibited a decrease. The study determined that the use of silane treatment leads to a decrease in WA, since the reduction in hydrophilic hydroxyl groups on the fiber after silane treatment, resulting in a weakened WA capacity of the composites. Moreover, the PLA composites treated with silane exhibit enhanced bonding compatibility between the PLA and fiber. The study found that the fiber composites treated with KH-570 exhibited the highest level of water resistance. This is because the KH-570-treated PLA composites are better at sticking to other materials, as shown by the fact that they have the largest contact angle and the smallest amount of water that can get into them.

Abdallah et al. [154] performed the silane treatment on date palm

wood fiber with different solution mixtures, which used acetone-water and ethanol water as solvents. Based on the room-temperature WA, the effect of both different solvents in the silane treatment reduces the WA of the composites. The silane-treated fiber composites with the ethanol-water solvent absorbs less water than the acetone-water solvent. The study states that this reduction in WA is proved by the lower peak of the hydroxyl group in FTIR analysis, which means the treatment can reduce the hydrophilicity of the fiber.

Alao et al. [143] conducted silane modification on hemp fiber to reinforce PLA composites. The silane modification in this study was divided into two different steps, which were washed-silane and alkali-silane fiber treatment. After the fiber were silane treated with both different processes, the WA for both treatments decreased. Based on the data of the study, the alkali-silane-treated fiber shows greater water resistance. The study stated that the silane treatment makes the fiber and matrix bonding better and has higher water resistance due to silane coupling on the fiber surface. Wang et al. [155] also conducted studies on two different processes for silane treatment, which were silane and alkali-silane. The study declared that after the treatment, the WA of ramie/PLA composites decreased since the hydroxyl group and silicon hydroxyl reacted, resulting in a decline of ramie fiber hydrophilic groups and enhance the bonding between ramie and PLA.

5.5. Acetylation treatment in natural fiber reinforced PLA composites

Chung et al. [144] performed work on the impact of acetylation treatment on PLA composites reinforced with kenaf fiber, using varying

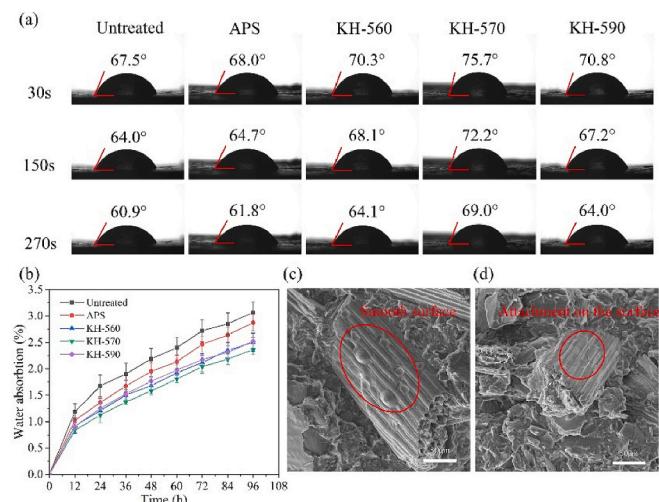


Fig. 10. Contact angle and water absorption rate of silane-modified composite (a and b, respectively), micromorphology of tensile section of unmodified and silane modified composites (c and d, respectively) [24].

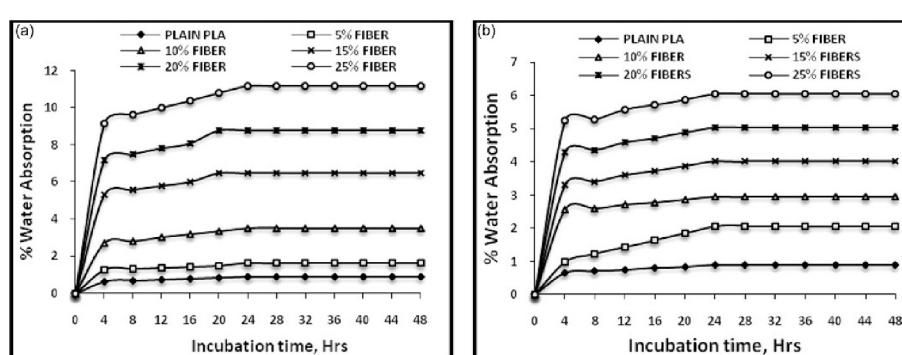


Fig. 9. Effect of fiber loading on water absorption of PLA composite with (a) untreated elephant grass fiber (b) treated elephant grass fibers [125].

treatment durations. The composites exhibit reduced water immersion in the PLA composites after undergoing acetylation treatment compared to the untreated PLA composites. The WA of the composites decline since the conversion of the hydroxyl group to the acetyl group in the fiber, leading to enhanced adhesion between the fiber and PLA and improved water resistance.

5.6. Enzymatic treatment in natural fiber reinforced PLA composites

Werchefani et al. [156] carried out a work on how enzymatic treatment affects the moisture absorption of natural fiber/PLA composites. The xylanase and pectinase enzymes were used to modify the alpha fiber in their study. As shown in Fig. 11, the untreated fiber composites show the greatest amount of moisture absorption. Nevertheless, the moisture absorption in PLA composites treated with enzymes is diminished since the elimination of lignin and hemicellulose components, which impact the WA of the composites. The study revealed that pectinase enzyme-treated fiber PLA composites exhibit a WA reduction of approximately 26 % compared to untreated fiber, therefore establishing it as the most effective enzyme treatment for reducing moisture absorption.

5.7. Silane + melamine-formaldehyde-treated in natural fiber reinforced PLA composites

Research of Tengsuthiwat et al. [110] aimed to reduce water uptake by doing silane treatment on the fiber and coating the fiber with melamine formaldehyde (MF) coating. Fig. 12 shows the method to prepare the silane and melamine formaldehyde coating on the sisal fiber and the fabrication of PLA/sisal MF-coated biocomposites.

Fig. 13 illustrates the WA characteristics of MF-sisal/PLA composites. It is noteworthy that the WA of MF-sisal/PLA composites, with a weight ratio of 1:1, exhibited a drop from 3 % to 1.4 % when compared to an uncoated counterpart following a 300-h immersion in water. In addition, it was observed that the WA of MF-sisal/PLA composites exhibited a modest decrease when the sisal/MF ratio increased. The observed phenomenon can be attributed to the significant decrease in the hydrophilic characteristics of sisal resulting from the application of the MF coating. Table 7 presents a similar pattern observed in the relationship between WA and diffusion coefficients, as determined through the application of Fickian law. The diffusion value of sisal/PLA composites exhibited a decrease upon the introduction of MF-coating. In the case of PLA composites featuring a weight ratio of 1:1 with MF-sisal coating, the D value shows a six-fold reduction in comparison to an uncoated counterpart [110].

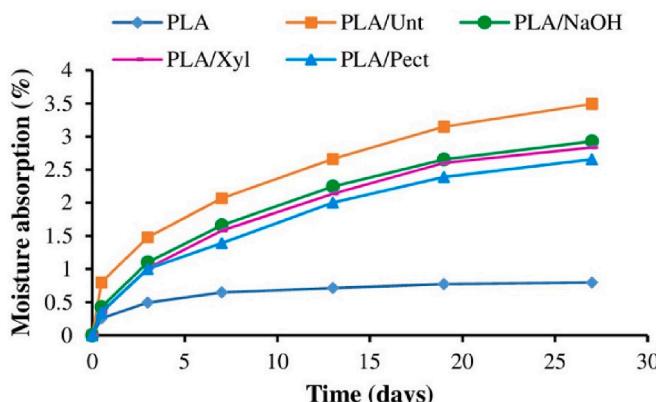


Fig. 11. Evolution of the moisture uptake of virgin polylactic acid (PLA) and its composites at 23 °C temperature and 95 % relative humidity [156].

5.8. Deep eutectic solvent modified in natural fiber reinforced PLA composites

Fang et al. [157] developed a simple and eco-friendly approach to modify the fiber using a deep eutectic solvent (DES), which is layered the surface of fiber with lignin. This method improved the interfacial performance of bamboo fiber (BF)/PLA composites, which is important because it is one of the variables that affects the WA of the composites. The DES was produced by heating choline chloride and oxalic acid in a 1:1 M ratio until a uniform and colorless mixture was obtained. Fig. 14 shows the process of fabricating of the DES treated BF/PLA composites.

Fig. 15 shows the water contact angle and WA of DES treated BF/PLA composites. In Fig. 15(a), the work indicates that the implication of the DES method on the BF surface makes the BF hydrophobic because the lignin layers on the surface of the fiber have hydroxyl groups and non-polar groups, which can reduce the interaction between fiber and water, and act as a barrier to prevent water immersion. However, when it dried at 150 °C (D-BF-150), the contact angle decreased due to more cellulose exposed on the fiber. After the treated fiber is incorporated with the PLA, as shown in Fig. 15(b)(d), the water contact angle increases since the lignin reduce the hydrophilicity of the composites. In Fig. 15(e) shows the treated and dried at 150 °C fiber/PLA composites had the lowest WA due to fewer voids in the composites [157].

5.9. Super-heated steam in natural fiber reinforced PLA composites

Alaa et al. [60] modified kenaf fiber with the super-heated steam (SHS) method to eliminate hemicellulose. The reduction of hemicellulose particles in fiber is eventually contributing to reducing its WA. The modification of kenaf fiber is started by drying kenaf fiber in the oven. Then, the SHS treatment is carried out in a SHS oven under normal pressure, 230 °C for 1 h, and water is used to modify the kenaf fiber. The SHS oven is switched on and adjusted to a stable state corresponding to the required condition. Afterwards, the kenaf fiber is evenly distributed over an aluminum foil tray and then put inside the SHS oven, adhering to the determined experimental conditions. Upon completion of the treatment process, the fiber is promptly taken out of the heating chamber and subsequently chilled in a desiccator. Fig. 16 presents kenaf fiber before treated and after treated with SHS treatment.

According to the data presented in Table 8, the application of the SHS treatment to kenaf fiber led to a decrease in WA by approximately 5.87 %. The phenomenon of decreased WA is commonly described as a decline in the amount of hydrophilic groups present in the fiber [60].

Similar work in modified natural fiber with SHS treatment was carried out by Ahmed et al. [158], in this work the authors use PALF and used 220 °C for 1 h to treat the fiber in a SHS oven. The authors used scanning electron microscopy (SEM) to observe both PALF that had not been treated and that had been treated with SHS, as shown in Fig. 17. The PALF that had not been treated with any treatment showed a surface that showed the presence of impurities and a few waxy compounds. Nevertheless, the PALF treated with SHS treatment did not exhibit any contaminants, lead to an enhance the fiber surface roughness, and this improve the bonding between matrix and fiber, and affected the WA as seen in Fig. 18. The WA rate of the SHS-PALF/PLA biocomposites exhibited a reduction of 42 % in contrast with the untreated PALF/PLA biocomposites. The observed outcome might be ascribed to the elimination of hemicellulose content inside the fiber.

5.10. Nano-SiO₂ in natural fiber reinforced PLA composites

Zuo et al. [74] utilized a method to increase WA resistance with incorporating nano-SiO₂ into PLA-g-BF/PLA. The preparation process consists of three distinct steps: the initial production of PLA-g-BF, the dispersion of nano-SiO₂, and the molding stage, as depicted in Fig. 19.

Fig. 20 shows the rate of WA and contact angle that were seen in PLA-g-BF/PLA composites with various nano-SiO₂ ratios. The occurrence of

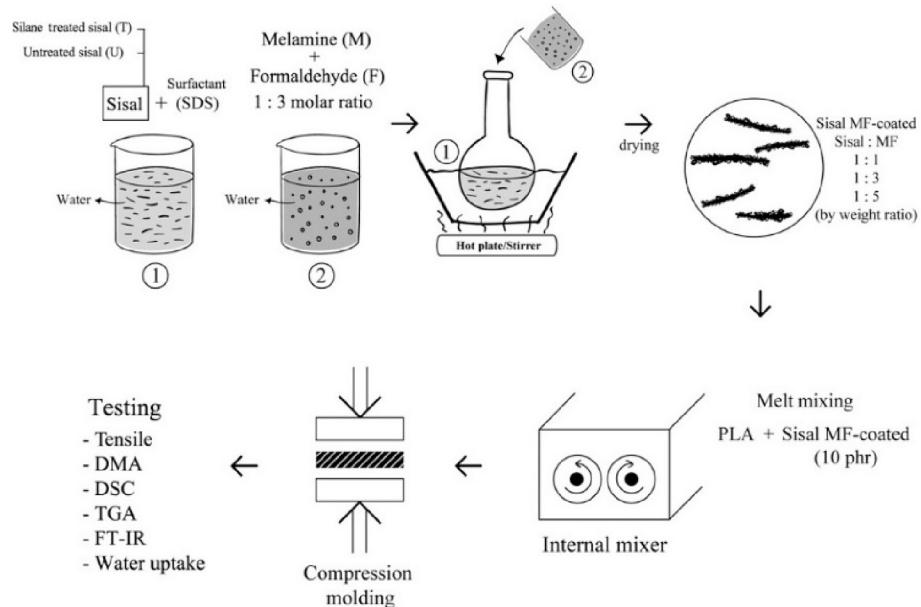


Fig. 12. Preparation steps of the PLA/sisal composites [110].

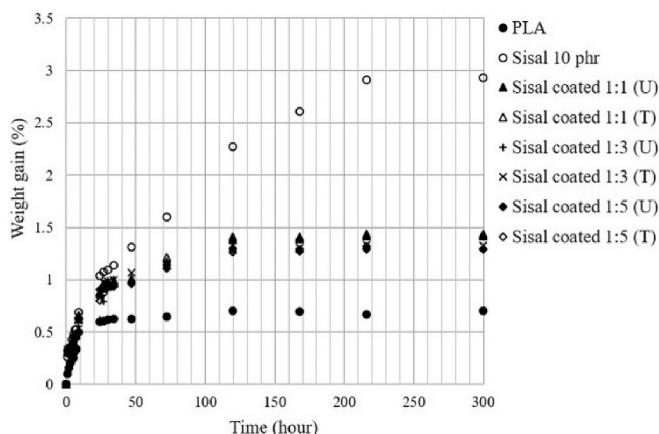


Fig. 13. Water absorption curves of PLA composites as a function of MF-sisal coating [110].

Table 7

Calculated diffusion coefficients (D) along with the related regression coefficients (R^2) [110].

Samples	M_m (%)	D (mm^2/h)	R^2
PLA	0.7	5.4 E-03	0.907
Sisal 5 phr	1.4	4.1 E-02	0.988
Sisal 10 phr	3	3.1 E-01	0.955
Sisal 20 phr	4.1	8.9 E-01	0.954
Sisal Coated 1:1 (U)	1.4	4.6 E-02	0.925
Sisal Coated 1:1 (T)	1.4	4.7 E-02	0.933
Sisal Coated 1:3 (U)	1.4	3.2 E-02	0.989
Sisal Coated 1:3 (T)	1.3	3.5 E-02	0.916
Sisal Coated 1:5 (U)	1.2	2.9 E-02	0.933
Sisal Coated 1:5 (T)	1.2	3.2 E-02	0.92

this can be attributed to the significant elevation in surface energy, resulting in an increased energy requirement for water diffusion onto the samples. Additionally, the enhanced interfacial compatibility of the samples plays an important part in reinforcing the bonding between PLA-g-BF and PLA, and as a result, it prevents the movement of water molecules between hydroxyl groups. Nevertheless, when the 2.0 % ratio

is used, the WA rises and the contact angle decreases. This phenomenon happened because the presence of active silicon groups on the surfaces of nano-SiO₂. The extensive surface areas offered several locations exhibiting the same polarity for the absorption of water. Nevertheless, in cases where the concentration of nano-SiO₂ was overly increased, it underwent agglomeration, resulting in the loss of its initial extensive surface area and nanoscale effects, resulting in a reduction in interface bonding and an increase in hydrophilicity.

5.11. In-situ solid phase grafting in natural fiber reinforced PLA composites

Li et al. [119] carried out an in-situ solid phase grafting method in bamboo fiber/PLA composites. The study carried out the grafting method by mixing the fiber with lactic acid, placing it in a hydrothermal reaction vessel for 7 h at 80 °C, and then drying and fabricating the grafted fibers into composites. The study revealed that the application of this method to the fibers resulted in a change in the WA value, which was 6.1 % for the untreated fiber composites and 4.3 % for the graft-treated fiber composites after soaking in water for 24 h. The study stated that the reduction in WA value is because of the fact that the number of OH groups on the treated fiber surface is lower than that of the untreated fiber, which can reduce WA value. The study also conducted a contact angle test to determine the hydrophobicity of the composites. It showed that after being treated by the in-situ solid phase grafting method, the hydrophobicity of the composites reduced, representing 34.5° for the untreated fiber composites and 41.5° for the treated fiber composites.

5.12. Heat treatment in natural fiber reinforced PLA composites

The heat treatment method is a cheap and environmentally friendly method that can be used to decrease WA value instead of using chemical treatment, which is not effective in time and is not environmentally friendly. [159] Chien et al. [159] conducted heat treatment methods on sapwood fiber with different heat treatment times, which were 2, 4, and 6 h, then reinforced it with PLA with a 3D-printed method to fabricate the composites. The treatment was conducted in an oven at a temperature of 180 °C. The WA properties in the study were determined by immersing the composite samples in water for 24 h. The result revealed that the WA value of the composites reduced along with the increases in

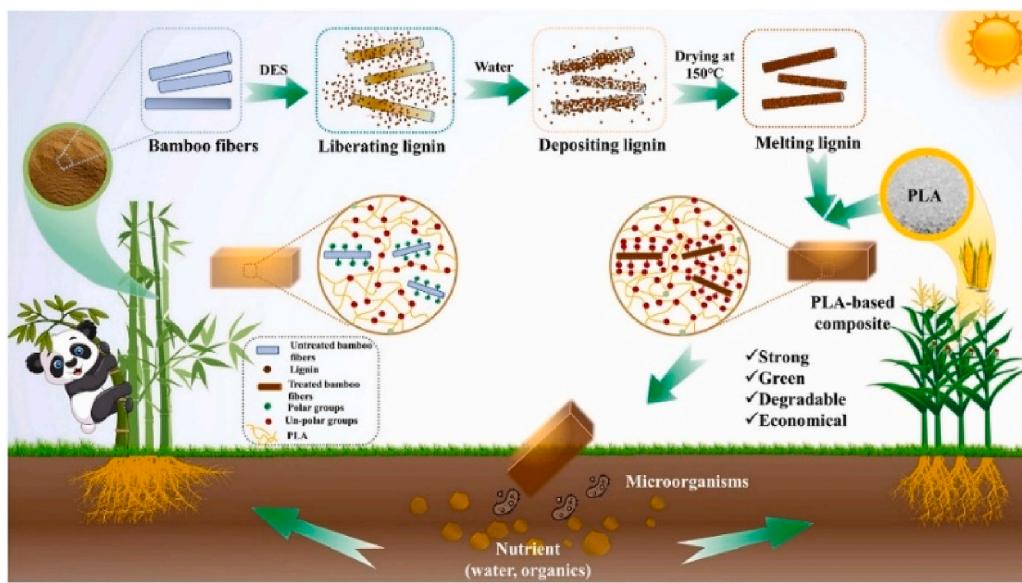


Fig. 14. The schematic illustration of the PLA-based composites reinforced with the modified bamboo fibers [157].

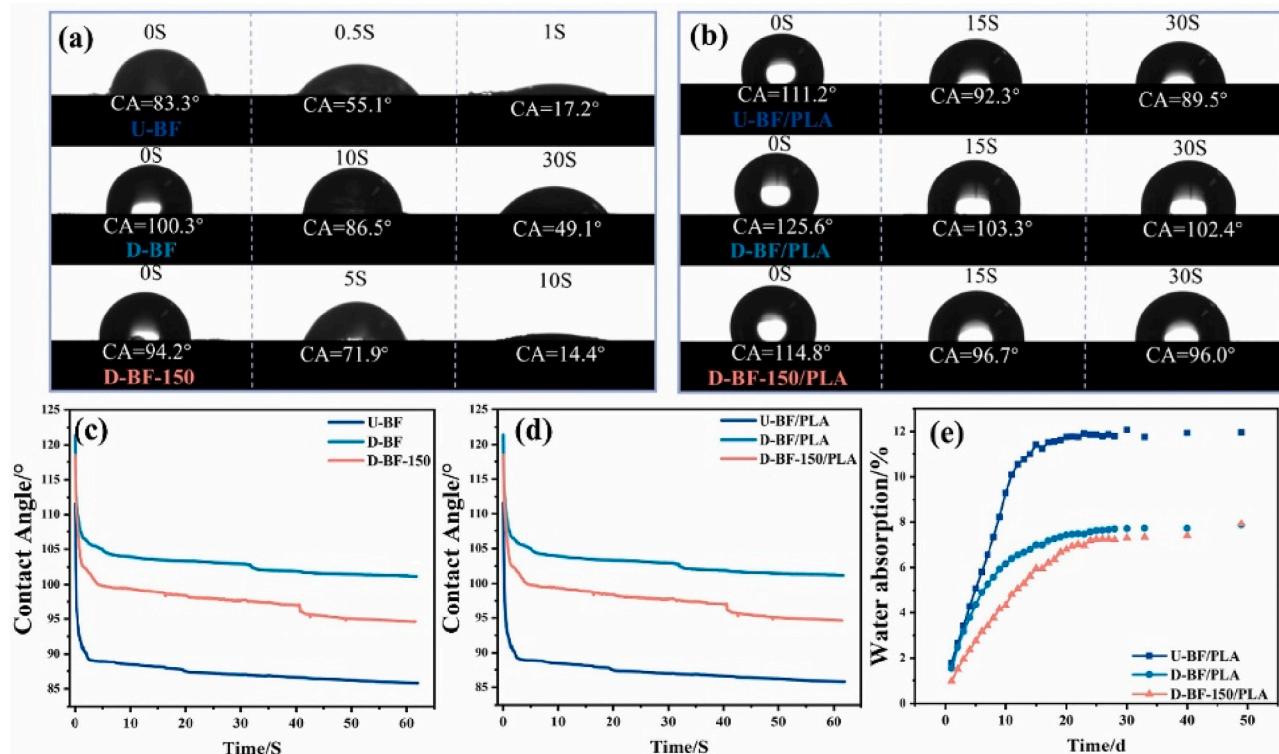


Fig. 15. The wettability and water absorption of the samples: (a) Contact Angle of the bamboo fibers. (b) Contact Angle of the PLA-based composites. (c) Contact angle-time curves of the bamboo fibers. (d) Contact angle-time curves of the PLA-based composites. (e) Water absorption-time curves of the PLA-based composites [157].

heat treatment time, representing 3.9 % for untreated composites and 3.2 % of the composites with the longest treatment time. This study stated that this phenomenon occurred due to the reduction of hemicellulose, which increased the hydrophobicity of the composites.

5.13. Ultrasound in natural fiber reinforced PLA composites

As a surface treatment for fibers, high-intensity ultrasound can effectively remove waxy substances and amorphous areas from the fiber

surface. The high-intensity ultrasonic sound produced bubbles, which could provide high pressure, temperature, and energy at the violent collapse, which is the same as hydrogen bond energy. Hence, it is possible to separate the complex multilayer structure of cellulose fiber from the non-crystalline substances. [160]

Krishnaiah et al. [160] performed a research on the effect of high-intensity ultrasound on the WA characteristic of the sisal/PLA composites. The treatment was carried out using an ultrasonic processor for 90 min with a 20 kHz frequency and 1000 W power, and the temperature

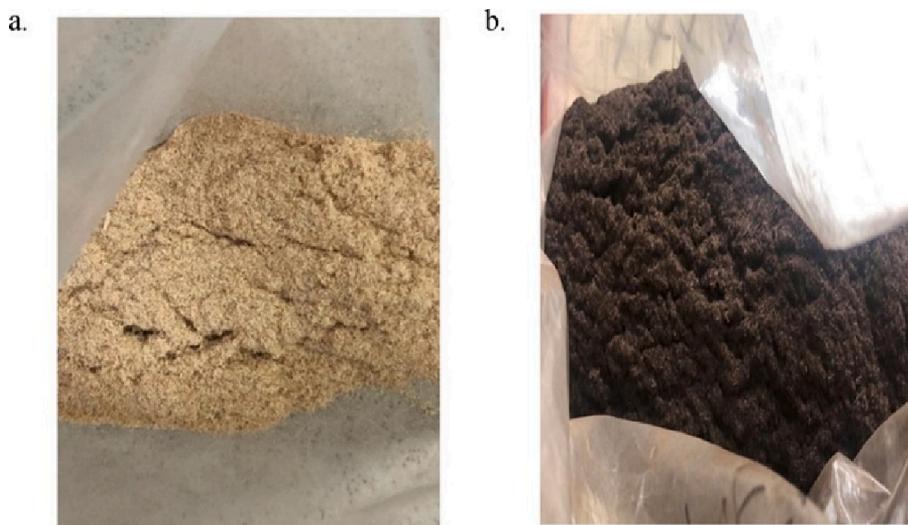


Fig. 16. 60 Mesh Kenaf Fiber (a) Untreated; (b) SHS Treated [60].

Table 8
Average water absorption [60].

Code	Average water absorption (%)
PLA-KF10	20.68
PLA-SKF10	14.81

was maintained between 25 and 30 °C. Then, the fibers were rinsed with water and dried in an oven and manufactured into composites. The results indicated that the high-intensity ultrasound treatment reduced the WA rate of the composites than the untreated composites, with the treated composites showing about a 4 % WA rate and the untreated composites showing a 5 % WA rate. The study confirmed that this was because of the removal of amorphous substances from the fiber surface, which led to better bonding between PLA and sisal.

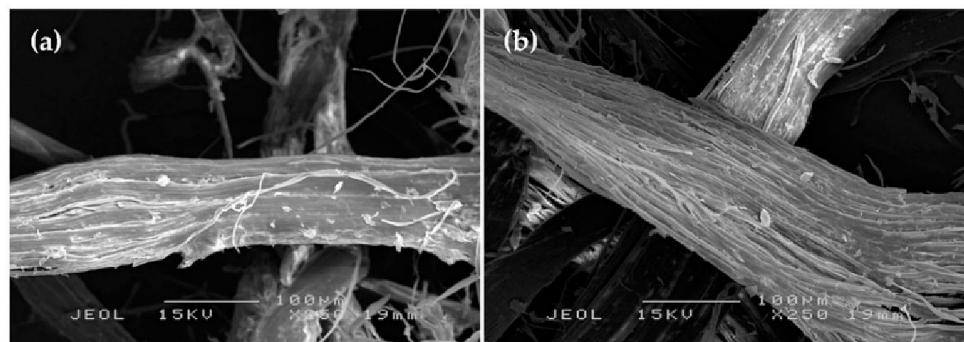


Fig. 17. SEM micrographs of (a) 250× PALF and (b) 250× SHS-PALF [158].

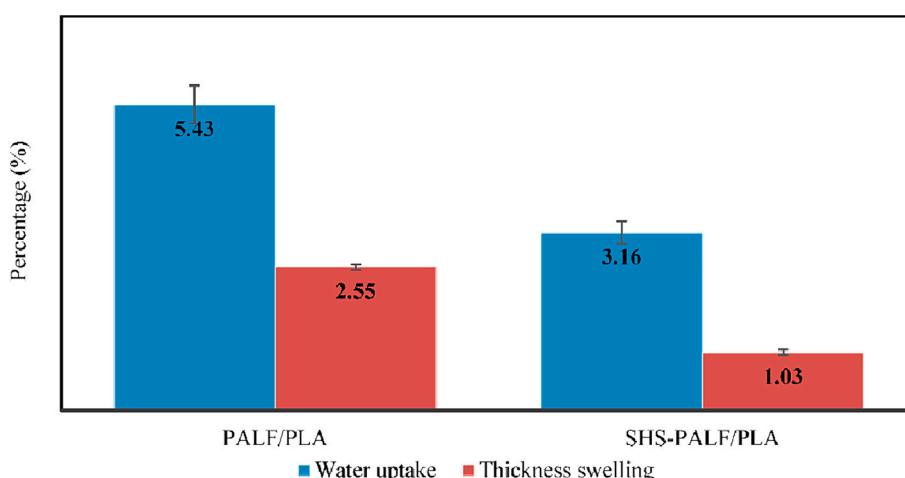


Fig. 18. The water uptake and thickness swelling of PALF/PLA and SHS-PALF/PLA biocomposites [158].

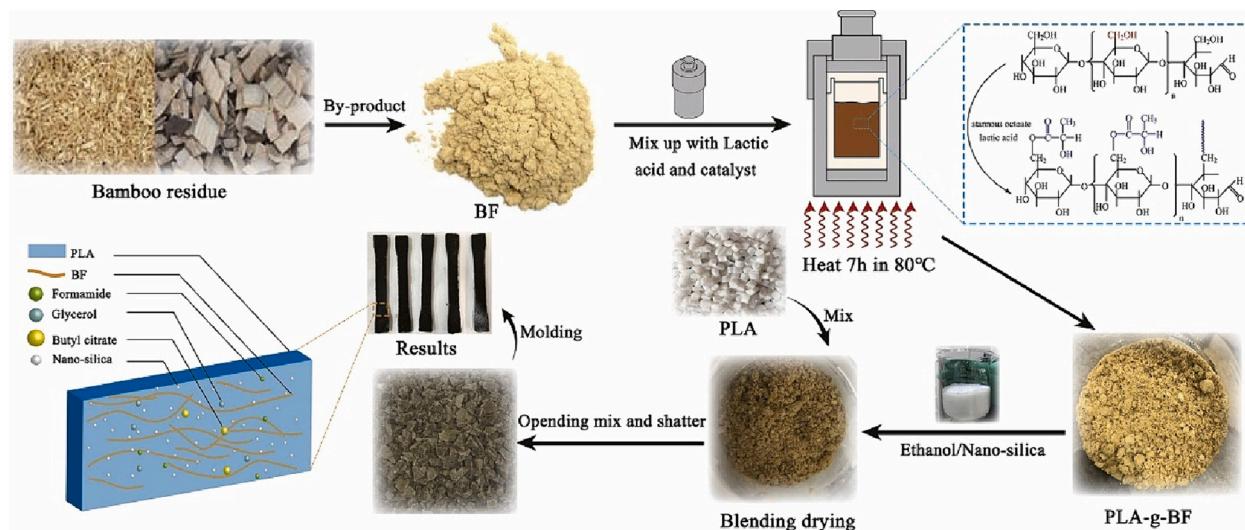


Fig. 19. Preparation process and principle of PLA-g-BF/PLA composites [74].

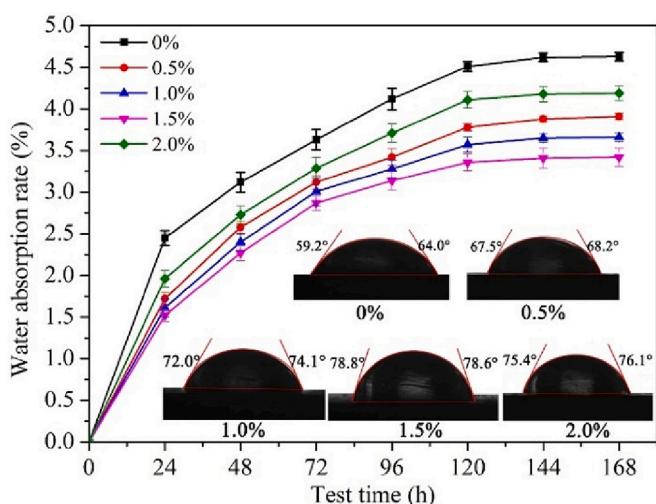


Fig. 20. Contact angle and water absorption rate of composites with different mass ratio of nano-SiO₂ [74].

Yu et al. [161] conducted a WA test on the rice straw powder treated by ultrasonic treatment and reinforced it with PLA to manufacture composites with the 3D-printing method. The treatment was carried out for 30 min at 20–30 °C with 600 W output power, then dried in an oven and fabricated into 3D-printed PLA composites. Compared to the untreated fiber composites, the ultrasound-treated fiber composite WA value decreased from about 10 % to 9 %. The study stated that the treatment of the fiber led to reduced porosity and gaps in the composites, which can lead to lower water immersion into the composites.

6. Conclusion and future directions

This review paper covers several variables that impact the water absorption (WA) behavior and the process of WA in natural fiber/polymer composites (NFC), with an especially on natural fiber/PLA composites. This review focused on looking into the impact of natural fiber loading, natural fiber hybrids, manufacturing techniques, and temperature on the WA of these composites. Additionally, it aimed to examine how WA affects the performance of natural fiber/PLA and explore various treatment techniques for natural fiber to mitigate this issue.

Recent research explains that the incorporation of natural fiber with PLA could result in a higher WA value, since the hydrophilic characteristic of natural fiber possesses many hydroxyl groups and hemicellulose content. The amorphous substance in the hemicellulose allows water to absorb easily into the natural fiber. Additionally, some works utilize natural fiber hybrids to reinforce PLA composites. The incorporation of a higher ratio of a more hydrophilic natural fiber than other fiber utilized in PLA composites leads to greater WA in the composites. Different fabrication processes could also affect the WA of natural fiber/PLA composites, resulting in varying amounts of voids in the composites for different fabrication techniques. The addition of fillers and the use of microfibers can also affect the WA characteristics of the natural fiber PLA composites. Furthermore, different water temperature could affect the absorption of water, since higher temperatures could lead to the PLA matrix degradation. Additionally, compared to another biopolymer, the use of PLA as the biopolymer absorbs more water than other biopolymers due to the bad incorporation between fiber and PLA. Moreover, the WA in the natural fiber/PLA composites could negatively impact the performance of the composites as a result of the damaging impacts generated by the WA, since WA in the composites leads to microcracking and the detachment of fiber and matrix. To sort out the issue of high WA in natural fiber/PLA composites, recent works have proposed various treatment methods to improve the WA resistance of the natural fiber/PLA composites. These methods include alkali, benzoylation, successive alkali, silane, acetylation, enzymatic, silane/melamine-formaldehyde, deep eutectic solvent, superheated steam, nanoSiO₂, grafting, heat treatment, and ultrasound. Recent published work on natural fiber-treated/PLA composites has effectively improved the WA resistance of the composites.

Further investigation into natural fiber/PLA composites should prioritize the exploration of novel techniques to enhance the composites WA resistance. The research needs to produce cost-effective, environmentally friendly technologies that do not involve the use of chemical components. Chemical treatment is cost-effective, but it is not environmentally friendly and has the potential to be hazardous to the environment. Therefore, the requirement of an ecologically sustainable and cost-effective approach is very important, so the composites could be implemented in a various type of applications and address the issue of waste management.

CRediT authorship contribution statement

Muhammad Adlan Azka: Writing - Original Draft,

Conceptualization, Methodology, Investigation. S.M. Sapuan: Writing - Review & Editing, Conceptualization, Supervision. **Hairul Abral:** Writing - Review & Editing, Supervision. **E.S. Zainudin:** Writing - Review & Editing, Supervision. **Faieza Abdul Aziz:** Conceptualization.

Declaration of competing interest

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

Data availability

No data was used for the research described in the article.

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