forests

MDPI

Article

The Effect of Alkali Treatment on the Mechanical Strength, Thermal Stability, and Water Absorption of Bamboo Fiber/PLA Composites

Xiaoyang Fang [1,2], Xin Tao [1,2], Yuxi Xie [1,2], Wei Xu [1,2,*], Hongwu Guo [3,*] and Yi Liu [3,*]

[1] College of Furnishings and Industrial Design, Nanjing Forestry University, Nanjing 210037, China; fangxiaoyang@njfu.edu.cn (X.F.); taoxin@njfu.edu.cn (X.T.); xieyuxi@njfu.edu.cn (Y.X.)
[2] Co-Innovation Center of Efficient Processing and Utilization of Forest Resources, Nanjing Forestry University, Nanjing 210037, China
[3] Key Laboratory of Wood Material Science and Application, Beijing Forestry University, Ministry of Education, Beijing 100083, China
* Correspondence: xuwei@njfu.edu.cn (W.X.); guohongwu_305@bjfu.edu.cn (H.G.); liuyichina@bjfu.edu.cn (Y.L.)

Abstract: Alkali treatment is a prevalent method to enhance the interfacial compatibility of natural fiber-reinforced polymer composites (NFRPCs). Although the influence of alkali treatment on the properties of NFRPCs has been extensively investigated, previous studies have predominantly examined individual factors in isolation, leaving the combined effects of alkali solution concentration, treatment temperature, and time relatively unexplored. In this study, an orthogonal experiment was conducted to assess the combined impacts of alkali solution (NaOH) concentration, treatment temperature, and time on the mechanical strength, thermal stability, and water absorption of bamboo fiber (BF)/polylactic acid (PLA) composites. The findings indicated that both the NaOH concentration and temperature exhibited a statistically significant effect ($0.01 < p < 0.05$) on the mechanical strength of BF/PLA composites, while the treatment time had no significant effect. Furthermore, all three factors had an extremely significant impact ($p < 0.01$) on the thermal stability of BF/PLA composites. The water absorption of BF/PLA composites was found to be significantly influenced by treatment temperature and time ($p < 0.01$), while no significant effect of NaOH concentration was observed. The optimal combination of alkali treatment parameters (concentration—5 wt%, temperature—25 °C, time—30 min) for BF/PLA composites was determined. Additionally, it was observed that the water absorption of alkali-treated BF/PLA composites was lower than that of untreated composites for shorter dipping times, but higher for prolonged dipping times. This work offers an important reference for the efficient application of alkali treatment to NFRPCs.

Keywords: bamboo fibers; polylactic acid; alkali treatment; interface compatibility; mechanical properties

Academic Editor: Alain Cloutier

Received: 12 December 2024
Revised: 2 January 2025
Accepted: 5 January 2025
Published: 10 January 2025

Citation: Fang, X.; Tao, X.; Xie, Y.; Xu, W.; Guo, H.; Liu, Y. The Effect of Alkali Treatment on the Mechanical Strength, Thermal Stability, and Water Absorption of Bamboo Fiber/PLA Composites. *Forests* **2025**, *16*, 123. https://doi.org/10.3390/f16010123

1. Introduction

The growing societal concerns about environmental pollution and the demand for eco-friendly materials have prompted the adoption of natural plant fiber-reinforced composites (NFRPCs) as alternatives to petrochemical products. NFRPCs are increasingly gaining attention due to their environmental friendliness, biodegradability, and low density [1–3]. Some of the plant fibers, derived from the forestry, agriculture, and timber sectors, can be repurposed as bio-fillings to optimize resource utilization and generate value-added commodities [4,5]. However, the utilization of NFRPCs on a large scale presents a significant obstacle as a result of the incongruity between the hydrophilic nature of plant fibers and the hydrophobic composition of the polymer matrix [6,7]. To address this issue of interfacial incompatibility, researchers have tried several chemical treatments such as mercerization, silylation, acetylation, maleated coupling, and enzyme treatment to modify the surface properties of plant fibers [8]. Among these treatments, alkali treatment emerged as a highly effective, cost-efficient, and widely adopted approach for enhancing the interfacial compatibility between the fibers and polymers [9–11].

Numerous studies have been published regarding the characteristics of polymer composites that are strengthened with plant fibers treated with NaOH [12–16]. It was found that the concentration, temperature, and time of alkali treatment are important for the properties of NFRPCs. The existing studies mainly focused on employing a single-factor test to investigate the effects of alkali treatment on the mechanical or thermal properties of NFRPCs. The concentrations ranged from 0.5 wt% [12] to 30 wt% [13], temperatures ranged from 20 °C to 100 °C [14,15], and time ranged from 15 min [12] to several weeks [16]. Large amounts of time, effort, and money were required to carry out trials to find an optimized solution. Furthermore, the efficacy of these treatments in enhancing the performance of NFRPCs was not consistently observed and, in certain cases, was even compromised, particularly at high concentrations of alkali treatment. Despite the simplicity of the alkali treatment process, comprehensive information is scarce on the combined impact of alkali treatment concentration, temperature, and time on the characteristics of NFRPCs. Additionally, there remains a paucity of research investigating the principal factors of alkali treatment, and none of the existing studies have reached a definitive conclusion.

This study employed an orthogonal experimental design (OED) to optimize the alkali treatment parameters to reflect the comprehensive impact on NFRPC performance. OED, a cost-effective and efficient technique [17], not only minimizes the number of experimental trials [18], but also effectively highlights the significance of individual factors or their combinations, aiding in the determination of the optimal levels of each factor for achieving optimal performance [19]. Bamboo fibers (BFs) are abundantly accessible in China and possess exceptional mechanical strength and a rapid growth rate compared to other natural fibers [20–22]. Polylactic acid (PLA) has gained significant attention as a highly promising biopolymer owing to its remarkable biodegradability [23,24]. Consequently, this study selected BF/PLA composites as the subject of our research. The present study employed a three-level–three-parameter OED approach to examine the influence of variables such as concentration, treatment temperature, and treatment time on the mechanical strength, thermal stability, and water resistance of BF/PLA composites. The primary objective of this investigation is to identify the key factors and optimize the optimal process combination for alkali treatment. The outcomes of this study are expected to offer targeted suggestions for the preparation of alkali-treated plant fiber-reinforced polymer composites.

2. Materials and Methods

2.1. Materials

Anhui Sentai Wood-Plastic Composite Co., Ltd. (Guangde, China) provided the bamboo fibers with a size of 150–180 μm. PLA, Class 2003D, was purchased from Nature Works (North Ford, CT, USA). Sodium hydroxide, analytical grade, was obtained from McLean Chemical Co., Ltd. (Shanghai, China). Deionized water was prepared in our laboratory.

2.2. Orthogonal Experimental Design

In the OED experiment, there are three independent variables and three levels of variation, coded for each factor. The concentration (Factor A), temperature (Factor B), and treatment time (Factor C) of alkali treatment were chosen as the three independent variables. The response variables were mechanical strength (flexural strength, flexural modulus, and impact strength), thermal stability (T_{onset} and T_{max}), and water absorption. Accordingly, an $L_9(3^4)$ orthogonal array was built to guide the subsequent alkali treatments for bamboo fibers, as listed in Table 1. Each treatment combination was conducted in triplicate to ensure the experiments' accuracy, with a total of 27 trials reported. The data were evaluated by range analysis, analysis of variance (ANOVA), and multiple comparisons to order the factors according to the degree of their impacts, thereby identifying the key influencing factors and optimizing the optimal process combination. This work did not consider the interactions among the factors.

Table 1. Orthogonal factors, levels, and test combinations of L_9 (3^4).

Number	Factor A Concentration/wt%	Factor B Temperature/°C	Factor C Time/min	Treatment Combination	Embodiments
1	1(A1)	25(B1)	30(C1)	A1B1C1	1 wt% NaOH, 25 °C, 30 min
2	1(A1)	50(B2)	60(C2)	A1B2C2	1 wt% NaOH, 50 °C, 60 min
3	1(A1)	75(B3)	90(C3)	A1B3C3	1 wt% NaOH, 75 °C, 90 min
4	5(A2)	25(B1)	60(C2)	A2B1C2	5 wt% NaOH, 25 °C, 60 min
5	5(A2)	50(B2)	90(C3)	A2B2C3	5 wt% NaOH, 50 °C, 90 min
6	5(A2)	75(B3)	30(C1)	A2B3C1	5 wt% NaOH, 75 °C, 30 min
7	9(A3)	25(B1)	90(C3)	A3B1C3	9 wt% NaOH, 25 °C, 90 min
8	9(A3)	50(B2)	30(C1)	A3B2C1	9 wt% NaOH, 50 °C, 30 min
9	9(A3)	75(B3)	60(C2)	A3B3C2	9 wt% NaOH, 75 °C, 60 min

2.3. Preparation of Composites

The BF/PLA composites were prepared with a hot-pressing process. The bamboo fibers and PLA were mixed and then melt-blended in an open mixer with a mass ratio of 3:7, with the following parameters: temperature 170 °C, speed 40 rpm, and time 5 min. The resulting mixtures were cooled down to room temperature and then were broken with a crusher and passed through a 40-mesh sieve. The pelletized material was then molded into specimens with a hot-press machine (HSXXHC-0) for mechanical flexural and impact tests. The hot-pressing process was carried out at a temperature of 170 °C and a pressure of 8 MPa for 20 min. This step was followed by cold-pressing at a pressure of 4 MPa for 4 min. Figure 1 shows the preparation process of BF/PLA composites.

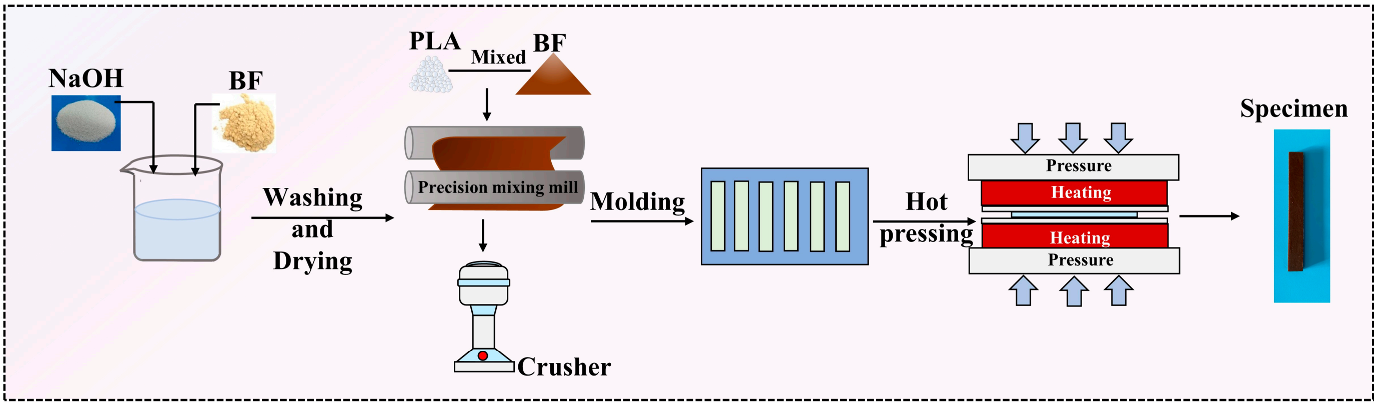

Figure 1. The preparation process of BF/PLA composites.

2.4. Characterization

2.4.1. Thermogravimetric Analysis (TGA)

The thermal stability of the samples was tested on a thermogravimetric analyzer (TGA2050, Instruments, New Castle, DE, USA). The specimens were subjected to a temperature increase from ambient to 600 °C in a nitrogen environment, with a heating velocity of 10 °C per minute.

2.4.2. Mechanical Tests

The flexural strength of specimens was tested on a universal testing machine (Instron3366, Instron, Norwood, MA, USA) following ASTM D790-2010 [25]. The impact strength of specimens was measured with a cantilever beam impact tester (KERUI, XJJ5, Dongguan, China) with the standard practices of ASTM D256-03 [26]. Five rectangular samples with a size of 80 mm × 10 mm × 4 mm were prepared for each group.

2.4.3. Water Absorption

The water absorption of the obtained composites was measured according to ASTM 5229 [27]. Firstly, the specimens were placed in the oven and dried to a constant weight, and then they were weighed, and the initial weight was determined (W_0). Subsequently, the specimens were soaked in room-temperature distilled water for the specified duration. The samples were rapidly wiped of any leftover water on the surface when taken out from the water and recorded as W_1 after being weighed. The same weighing procedure was repeated within the desired measuring interval. At least three specimens of each group were tested. The following equation was used to determine the water absorption (*WA*):

$$WA = \frac{W_1 - W_0}{W_0} \times 100 \quad (1)$$

3. Results

3.1. Analysis of Orthogonal Experiment Results

The range analysis was conducted based on the results of the orthogonal experiment test, and the corresponding results were displayed as given in Table 2. The range (R) is defined as the disparity between the maximum and minimum values of k, indicating the relative significance of each factor's impact on the assessment criterion [28,29]. The higher the R, the larger the influence of the factor on the index [30,31]. Changing the level of a factor with the highest R has a larger impact on the experimental results than changing the level of a factor with the lowest R [32].

Table 2. Range analysis of the index parameters obtained by L_9 (3^4).

Indexes		Factors		
		A	B	C
Initial degradation temperature/T_{onset}	k1	295.74	291.18	294.16
	k2	294.11	292.08	295.05
	k3	289.52	296.12	290.17
	R	6.22	4.95	4.89
Ranking		A > B > C		
Maximum weight loss peak temperature/T_{max}	k1	345.37	341.96	342.96
	k2	342.20	336.16	340.08
	k3	334.72	344.18	339.26
	R	10.64	8.02	3.70
Ranking		A > B > C		
Flexure strength/MPa	k1	50.57	52.76	53.23
	k2	53.66	49.15	51.33
	k3	51.13	53.44	50.79
	R	3.09	4.29	2.44
Ranking		B > A > C		
Modulus of elasticity/GPa	k1	4.68	4.71	4.68
	k2	4.57	4.48	4.57
	k3	4.50	4.57	4.50
	R	0.18	0.23	0.19
Ranking		B > C > A		
Impact strength/MPa	k1	10.30	11.00	10.72
	k2	11.34	11.50	10.88
	k3	11.49	10.63	11.53
	R	1.19	0.86	0.81
Ranking		A > B > C		
WA/%	k1	5.15	5.23	4.94
	k2	5.22	4.97	5.32
	k3	5.15	5.32	5.26
	R	0.07	0.34	0.38
Ranking		C > B > A		

As evident from Table 2, when the mechanical performance was taken as the index, the degrees of the effect of the factors on the flexural strength, flexural modulus, and impact strength were as follows: B > A > C, B > C > A, and A > B > C. The treatment temperature (Factor B) had the greatest effect on flexural strength and modulus, while the treatment time (Factor C) and concentration (Factor A) had the least effect on flexural strength and modulus, respectively. The treatment concentration had the greatest effect on the impact strength, whereas the treatment time had the least effect. When thermal stability was used as the index, the influence of each factor from strong to weak was in the following order, A > B > C, with the greatest influence coming from treatment concentration, followed by treatment temperature, and then treatment time. When the water absorption was taken as the index, the three factors of alkali treatment can be ranked in order of influence degree: C > B > A. Obviously, the treatment time had the greatest effect on water absorption, while the treatment concentration had the least effect.

The ANOVA can identify whether or not the factors are significant [28]. The larger the F-value, the more significant the influence of that factor [33]. As shown in Tables 3 and 4, the concentration of NaOH treatment exerted a significant influence ($0.01 < p < 0.05$) on the impact resistance of BFRPCs and a highly significant impact ($p < 0.01$) on their thermal stability, but no significant impact on the water absorption. The treatment temperature was found to have a significant impact ($0.01 < p < 0.05$) on both the flexural strength and thermal stability, and an extremely significant impact ($p < 0.01$) on water absorption. The treatment duration had an extremely significant influence ($p < 0.01$) on thermal stability and water absorption, but no significant effect on impact strength or flexural strength. According to Tables 3 and 4, for the composites' thermal stability, the treatment concentration had the highest F-value (19.50), whereas for the composites' water absorption, the treatment time had the highest F-value (9.25). This supported the range analysis results and demonstrated that the treatment concentration had the greatest impact on thermal stability while the treatment time had the greatest influence on water absorption.

Based on the ANOVA results, Duncan's new multiple range test was performed to evaluate the significant differences among the levels of significant factors. The results of the correlation analysis are shown in Figure 2. The impact strength of A2 and A3 was significantly higher ($0.01 < p < 0.05$) than that of A1, but there was no significant difference between A2 and A3, suggesting that an appropriate increase in alkali treatment concentration would improve the impact strength of composites, while further increases in alkali treatment concentration would not result in a significant improvement. The T_{onset} and T_{max} of A1 and A2 were markedly higher ($p < 0.01$) compared to A3, with no significant differences noted between A1 and A2. This suggested that increased treatment concentrations correlate with reduced thermal stability in the composite materials. Taken together, A2 was the best level of alkali concentration treatment for achieving high impact strength and good thermal stability of composites.

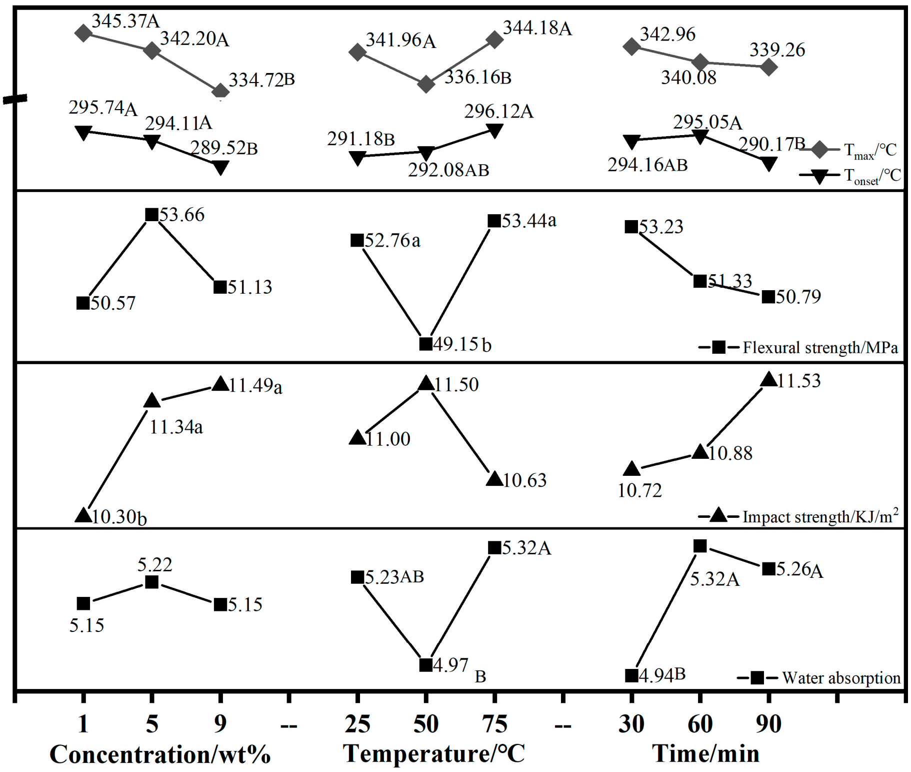

Figure 2. Comprehensive equilibrium diagram of properties of BF/PLA composites with different NaOH treatments. Notes: Different lowercase letters in the diagram indicate significant difference ($p < 0.05$). Different capital letters indicate significant difference ($p < 0.01$).

Table 3. Variance analysis results of the effect of different alkali treatments on thermal stability and water resistance of BF/PLA composites.

Source of Variation	df	T_{onset}			Significance	T_{max}			Significance	Water Absorption			Significance	F_{α}
		SS	MS	F		SS	MS	F		SS	MS	F		
Repeat	2	47.82	23.9	2.36		38.13	19.1	1.38		0.00	0.0	0.01		
Concentration	2	187.34	93.7	9.26	**	537.75	268.9	19.50	**	0.004	0.002	0.23	-	
Temperature	2	124.92	62.5	6.17	**	308.80	154.4	11.20	**	0.27	0.1	13.36	**	$F_{0.05} = 3.55$
Time	2	121.83	60.9	6.02	**	67.94	34.0	2.46	-	0.25	0.1	12.17	**	$F_{0.01} = 6.01$
Error	18	182.12	10.1			248.25	13.8			0.18	0.009			
Total	26	616.22				1162.74				0.71				

An extremely significant difference is signified with "**" ($p < 0.01$), and no significant difference is signified with "-".

Table 4. Variance analysis results of the effect of different alkali treatments on the mechanical performance of BF/PLA composites.

Source of Variation	df	Flexural Strength			Significance	Flexural Modulus			Significance	Impact Strength			Significance	F_{α}
		SS	MS	F		SS	MS	F		SS	MS	F		
Repeat	2	7.41	3.70	<1		0.24	0.12	<1		0.51	0.25	<1		
Concentration	2	48.77	24.39	2.15	-	0.16	0.08	<1	-	7.53	3.77	4.54	*	
Temperature	2	95.65	47.82	4.21	*	0.24	0.12	<1	-	3.38	1.69	2.04	-	$F_{0.05} = 3.55$
Time	2	29.59	14.80	1.30	-	0.16	0.08	<1	-	3.30	1.65	1.99	-	$F_{0.01} = 6.01$
Error	18	204.46	11.36			2.60	0.14			14.93	0.83			
Total	26	378.47				3.16				29.15				

A significant difference is signified with "*" ($0.01 < p < 0.05$), and no significant difference is signified with "-".

The T_{onset} of B3 was considerably higher (p < 0.01) than that of B1, while no significant differences were found between B3 and B2, or B2 and B1. The T_{max} values of B1 and B3 were significantly higher than that of B2, but no significant difference between B1 and B3 was observed. From the T_{onset} and T_{max} results, it appeared that B1 was the optimal parameter to reach better thermal stability of BF/PLA composites. The flexural strength of B1 and B3 was significantly greater (p < 0.01) compared to B2, with no significant difference observed between B1 and B3. This implied that the flexural strength of the composites can be effectively enhanced, particularly under the conditions of B1. B3 showed the highest value in water absorption, significantly different from B2 (p < 0.01). However, the water absorption showed no significant difference between B1 and B2, indicating that properly increasing the alkali treatment temperature had very little effect on the water absorption, but the water absorption increased with further increasing temperature. Generally, the water uptake behaviors of BF/PLA composites would have a substantial impact on their long-term durability. Overall, B1 was the most suitable amount of alkali temperature treatment for composites with high flexural strength and minimal water absorption.

Among the three levels of Factor C, the T_{onset} of C1 and C2 was significantly higher (p < 0.01) than that of C3, with no significant difference detected between C1 and C2. This indicates that extended treatment times could potentially expedite the initial decomposition of the composite material. The thermal stability of the composite tends to decrease. The water absorption of composites treated with C2 and C3 was significantly higher (p < 0.01) than that of C1, implying that the longer the alkali treatment time, the higher the water absorption of composites. Combined with the thermal stability and water absorption analysis, C1 was the optimal alkali treatment time for BF/PLA composites.

3.2. Property Analysis of BF/PLA Composites

3.2.1. Mechanical Property Analysis

Figure 3 depicts the effects of NaOH treatment on the flexural strength, flexural modulus, and impact strength of BF/PLA composites. It can be seen that the flexural strength and modulus of NaOH-treated BF/PLA composites were higher than those of the untreated ones (Figure 3a,b), and the NaOH treatment had a greater effect on the flexural strength than the flexural modulus, which was consistent with previous research [34]. The alkali-treated BF/PLA composites demonstrated increased impact strength except for group 1 (Figure 3c).

There is growing evidence that alkali treatment has a positive effect on the mechanical properties of NFRPCs [16,34–36], and our results reinforce this. The increase in mechanical strength of the treated fiber-reinforced composites was mainly due to the removal of the impurities from the surface of the fiber with the assistance of NaOH solution, which improved the interfacial adhesion between the fibers and the matrix, thus ensuring the transfer of load from the PLA substrate to the bamboo fibers [37]. The possible mechanism is shown in Figure 4.

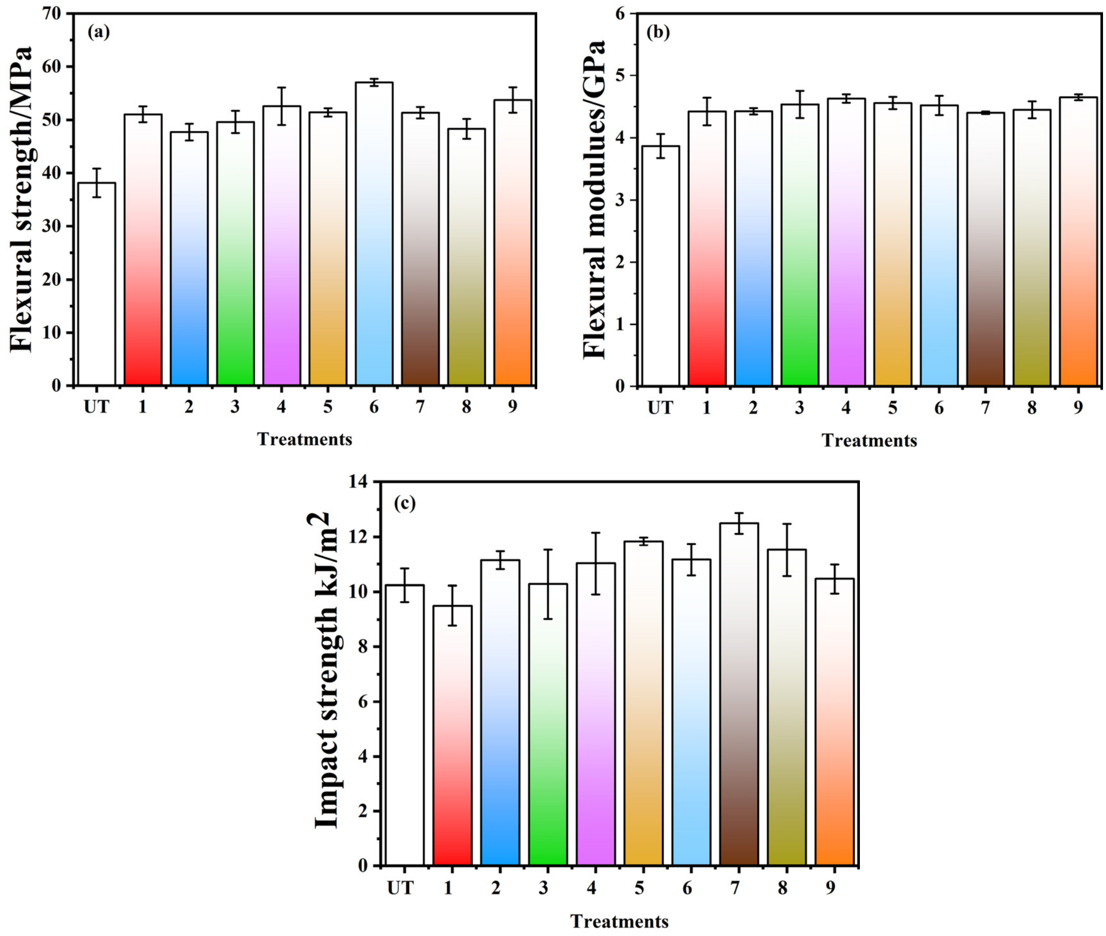

Figure 3. The mechanical performance of BF/PLA composites before and after NaOH treatments: (**a**) flexural strength; (**b**) flexural modulues; (**c**) impact strength.

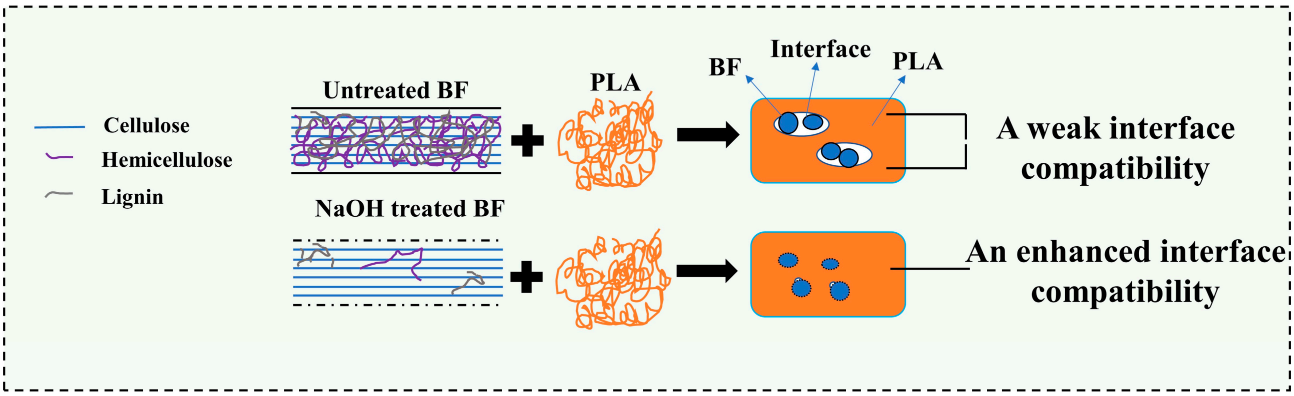

Figure 4. The mechanism of NaOH treated BF/PLA composites.

3.2.2. Thermal Stability Analysis

The T_{onset} (corresponding to a 5% weight loss [38]) and maximum decomposition temperature (T_{max}) were used to assess the thermal stability of the composites, with corresponding results as shown in Figure 5. Obviously, the treated composites had higher T_{onset} and T_{max} values than the untreated ones (Figure 5a,b), indicating that alkali treatment improved the interfacial adhesion between the fibers and PLA matrix, and the energy required for the movement of the PLA molecular chain was increased [39], thus improving the thermal stability of the composites. The corresponding TGA curves are shown in Figure 5c,d, and can be divided into three stages. The composites showed a slight mass loss around 50–150 °C, presumably due to the loss of adsorbed water molecules. The second stage was observed at 150–400 °C, with the biggest increase in mass loss seen in this stage, which is mainly related to the decomposition of cellulose, hemicellulose, and PLA. In the third stage, performed at a higher temperature range (400–600 °C), the decomposition of carbonaceous materials in the composites occurred, leading to minor weight loss.

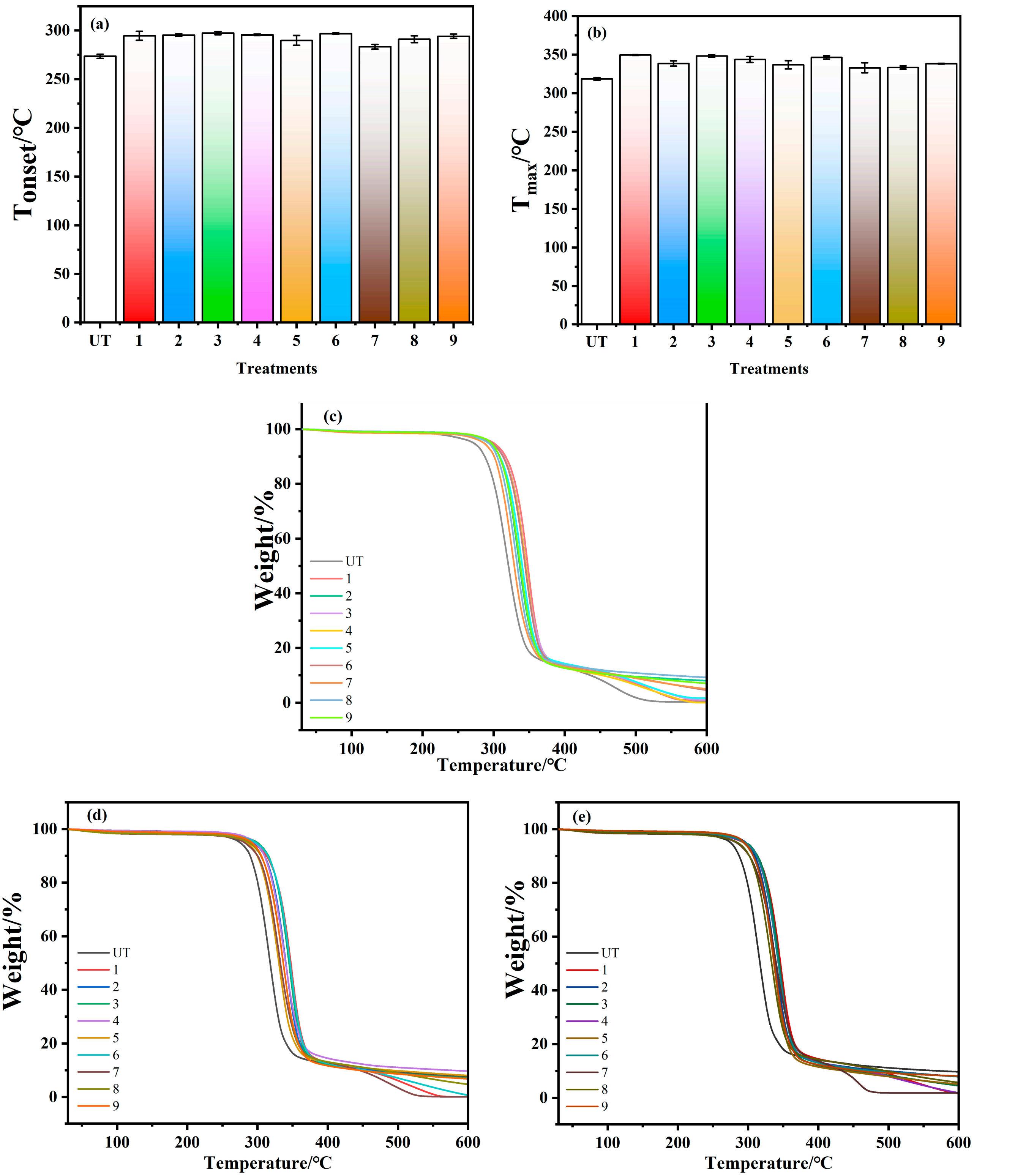

Figure 5. The thermal stability of BFRPCs before and after alkali treatment: (**a**) T_{onset}; (**b**) T_{max}; (**c**) TG patterns of run 1; (**d**) TG patterns of run 2; (**e**) TG patterns of run 3.

3.2.3. Water Absorption Analysis

Figure 6 shows the water absorption of BF/PLA composites. It was found that for the first five days after immersion, the water absorption of most alkali-treated samples was lower than that of untreated ones, which was similar to Ramamoorthysk's study [40]. However, on day 8, it appeared that some alkali-treated composites were absorbing more water than untreated ones. On day 18, the alkali-treated composites showed significantly higher water absorption than the untreated ones, a trend that continued until day 40. This work suggests a mechanism for this, as outlined in Figure 7. It is possible that the alkali treatment enhanced the interfacial connection between the fibers and the PLA matrix, reducing the internal porosity of the composites. As a result, the alkali-treated composite absorbed less water in a short time. However, the alkali-treated fibers had a larger specific surface area because of the decreased diameter, which caused the fibers to swell throughout the process of water absorption over time. Consequently, the micro gap between the alkali-treated bamboo fiber and PLA matrix grew larger, making it possible for the treated composites to absorb more water than the untreated ones.

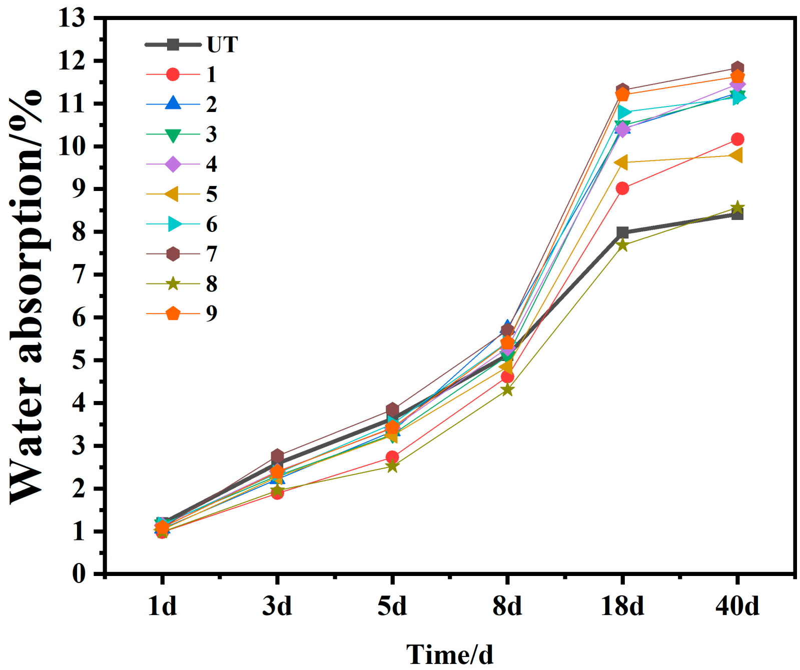

Figure 6. The water absorption characteristics of BF/PLA composites.

The water absorption of alkali-treated composites was higher than that of untreated ones after prolonged water immersion, which was in contrast to the findings of most studies [41,42]. However, given the majority of water absorption studies reported in the literature [12,43–45] have short durations, it was easy to conclude that alkali treatment reduces composite water absorption. Regarding the concerns about NFRPCs' long-term exposure to the environment, it would be more sensible for this work to investigate composite water absorption over an extended time. NFRPCs' low resistance to moisture absorption, which can be a challenging issue that accelerates the aging of composites and shortens their service life, makes their environmental stability unsatisfactory even though an appropriate alkali treatment process can improve their mechanical properties and thermal stability.

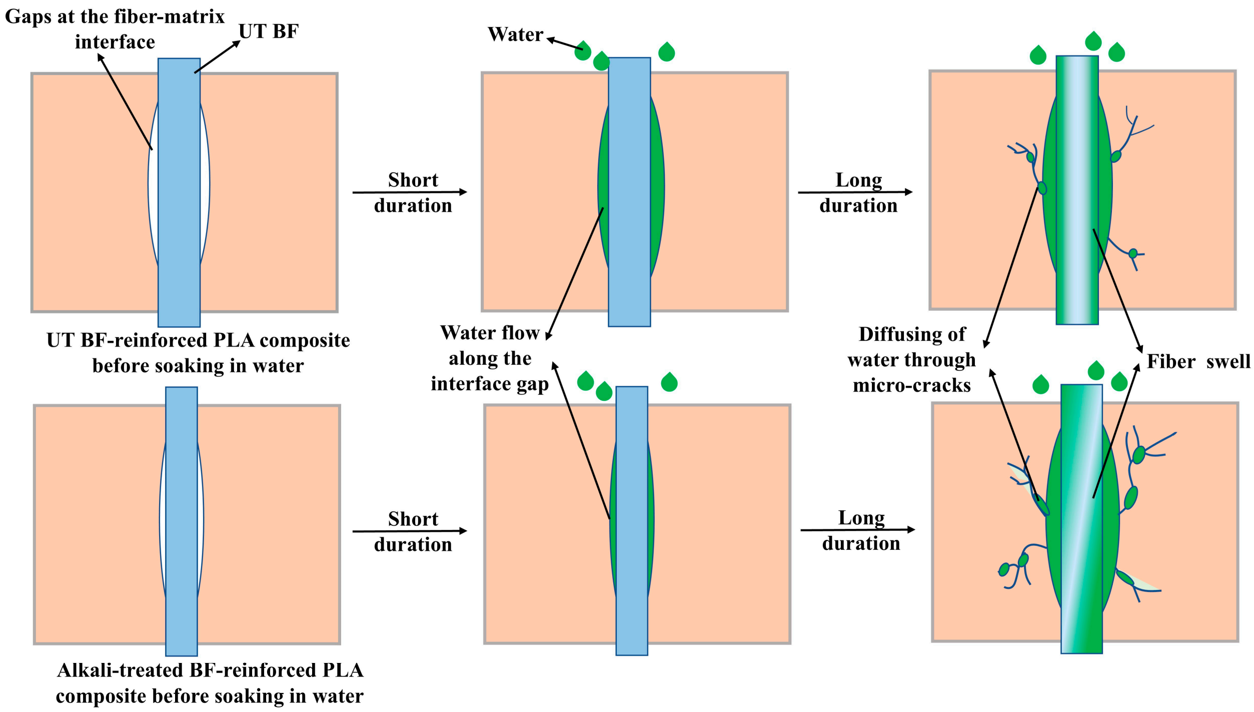

Figure 7. The possible water absorption mechanism of NaOH-treated BF/PLA composites.

4. Conclusions and Discussion

The orthogonal test results of the alkali-treated BF/PLA composites were analyzed by range analysis, variance analysis, and multiple comparisons analysis. When mechanical strength was taken as the criterion, both the alkali treatment concentration and the temperature significantly influenced ($0.01 < p < 0.05$) the impact and flexural strength of the BF/PLA composites, while the alkali treatment time failed to produce any significant effect on the mechanical strength. When the thermal stability was taken as the index, all three factors of alkali treatment showed an extremely significant effect ($p < 0.01$). When considering the water absorption as the index, it was significantly affected ($p < 0.01$) by the treatment temperature and time, but was not significantly affected by the treatment concentration. Through comprehensive evaluation, the superior proportions for alkali treatment were determined to be 5 wt%, 25 °C, and 30 min. Optimizing the BF/PLA composites' overall performance can be achieved by combining the factors at their optimal levels.

In summary, this work investigated the main dominant parameters and mechanisms of alkali treatment on the mechanical, thermal stability, and water resistance properties of BF/PLA composites, and the optimal combination for alkali treatment was determined. This not only improved efficiency but also reduced costs and achieved the best performance. Furthermore, the alkali-treated composites' water absorption was found to be correlated with the length of submersion time, and the water absorption increased with longer immersion time, which may exert a negative effect on the NFRPC performance under long-term use conditions. This research offers a significant guide for the design and fabrication of alkali-treated plant fiber-reinforced polymer composites, as well as for the environmental considerations necessary for the practical use of natural fiber-reinforced polymer composites (NFRPCs).

Author Contributions: X.F.: conceptualization, methodology, formal analysis, resources, writing—review and editing; X.T.: data curation, software; Y.X.: investigation, formal analysis; W.X.: conceptualization, project administration; H.G.: resources, supervision; Y.L.: conceptualization, supervision. All authors have read and agreed to the published version of the manuscript.

Funding: This study has been funded by the International Cooperation Joint Laboratory for Production, Education, Research and Application of Ecological Health Care on Home Furnishing (202101148006).

Data Availability Statement: Data will be made available on request.

Conflicts of 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.

References

1. Nascimento, H.M.; Granzotto, D.C.T.; Radovanovic, E.; Fávaro, S.L. Obtention and characterization of polypropylene composites reinforced with new natural fibers from *Yucca aloifolia* L. *Compos. Part B Eng.* **2021**, *227*, 109414. [CrossRef]
2. Yang, K.; Guan, J.; Numata, K.; Wu, C.; Wu, S.; Shao, Z.; Ritchie, R.O. Integrating tough Antheraea pernyi silk and strong carbon fibres for impact-critical structural composites. *Nat. Commun.* **2019**, *10*, 3786. [CrossRef] [PubMed]
3. Mazaherifar, M.H.; Hosseinabadi, H.Z.; Cosereanu, C.; Cerbu, C.; Timar, M.C.; Georgescu, S.V. Investigation on *Phoenix dactylifera*/*Calotropis procera* Fibre-Reinforced Epoxy Hybrid Composites. *Forests* **2022**, *13*, 2098. [CrossRef]
4. Mohanty, A.K.; Vivekanandhan, S.; Pin, J.M.; Misra, M. Composites from renewable and sustainable resources: Challenges and innovations. *Science* **2018**, *362*, 536–542. [CrossRef]
5. Zheng, Z.Y.; Yan, N.A.; Lou, Z.C.; Jiang, X.Z.; Zhang, X.M.; Chen, S.; Xu, R.; Liu, C.; Xu, L. Modification and Application of Bamboo-Based Materials: A Review-Part I: Modification Methods and Mechanisms. *Forests* **2023**, *14*, 2219. [CrossRef]
6. Hong, H.; Xiao, R.; Guo, Q.; Liu, H.; Zhang, H. Quantitively Characterizing the Chemical Composition of Tailored Bagasse Fiber and Its Effect on the Thermal and Mechanical Properties of Polylactic Acid-Based Composites. *Polymers* **2019**, *11*, 1567. [CrossRef]
7. Rashid, B.; Leman, Z.; Jawaid, M.; Ghazali, M.J.; Ishak, M.R. Physicochemical and thermal properties of lignocellulosic fiber from sugar palm fibers: Effect of treatment. *Cellulose* **2016**, *23*, 2905–2916. [CrossRef]
8. Kabir, M.M.; Wang, H.; Lau, K.T.; Cardona, F. Chemical treatments on plant-based natural fibre reinforced polymer composites: An overview. *Compos. Part B Eng.* **2012**, *43*, 2883–2892. [CrossRef]
9. Belouadah, Z.; Ati, A.; Rokbi, M. Characterization of new natural cellulosic fiber from *Lygeum spartum* L. *Carbohydr. Polym.* **2015**, *134*, 429–437. [CrossRef]
10. Bartos, A.; Anggono, J.; Farkas, A.E.; Kun, D.; Soetaredjo, F.E.; Moczo, J.; Antoni; Purwaningsih, H.; Pukanszky, B. Alkali treatment of lignocellulosic fibers extracted from sugarcane bagasse: Composition, structure, properties. *Polym. Test.* **2020**, *88*, 106549. [CrossRef]
11. Sunny, T.; Pickering, K.L.; Lim, S.H. Alkali treatment of hemp fibres for the production of aligned hemp fibre mats for composite reinforcement. *Cellulose* **2020**, *27*, 2569–2582. [CrossRef]
12. Wang, H.; Sheng, K.C.; Lan, T.; Adl, M.; Qian, X.Q.; Zhu, S.M. Role of surface treatment on water absorption of poly(vinyl chloride) composites reinforced by Phyllostachys pubescens particles. *Compos. Sci. Technol.* **2010**, *70*, 847–853. [CrossRef]
13. Aydin, M.; Tozlu, H.; Kemaloglu, S.; Aytac, A.; Ozkoc, G. Effects of Alkali Treatment on the Properties of Short Flax Fiber-Poly(Lactic Acid) Eco-Composites. *J. Polym. Environ.* **2011**, *19*, 11–17. [CrossRef]
14. Orue, A.; Jauregi, A.; Unsuain, U.; Labidi, J.; Eceiza, A.; Arbelaiz, A. The effect of alkaline and silane treatments on mechanical properties and breakage of sisal fibers and poly(lactic acid)/sisal fiber composites. *Compos. Part A Appl. Sci. Manuf.* **2016**, *84*, 186–195. [CrossRef]
15. Serra-Parareda, F.; Xavier Espinach, F.; Angels Pelach, M.; Alberto Mendez, J.; Vilaseca, F.; Tarres, Q. Effect of NaOH Treatment on the Flexural Modulus of Hemp Core Reinforced Composites and on the Intrinsic Flexural Moduli of the Fibers. *Polymers* **2020**, *12*, 1248. [CrossRef]
16. Gu, H. Tensile behaviours of the coir fibre and related composites after NaOH treatment. *Mater. Des.* **2009**, *30*, 3931–3934. [CrossRef]
17. Fang, L.; Liao, X.; Zhang, Q.; Shi, L.; Zhou, L.; Zhao, H.; Kong, W. An orthogonal experimental design and QuEChERS based UFLC-MS/MS for multi-pesticides and human exposure risk assessment in Honeysuckle. *Ind. Crops Prod.* **2021**, *164*, 113384. [CrossRef]
18. Cui, B.; Li, J.; Lai, Z.; Gao, F.; Zeng, Z.; Zhao, X.; Liu, G.; Cui, H. Emamectin benzoate-loaded zein nanoparticles produced by antisolvent precipitation method. *Polym. Test.* **2021**, *94*, 107020. [CrossRef]
19. Ding, X.; Xu, G.; Wang, D.; Luo, Z.; Wang, T. Effect of Synergistic Aging on Bauxite Residue Dust Reduction Performance via the Application of Colloids, an Orthogonal Design-Based Study. *Polymers* **2021**, *13*, 1986. [CrossRef]
20. Youssefian, S.; Jakes, J.E.; Rahbar, N. Variation of Nanostructures, Molecular Interactions, and Anisotropic Elastic Moduli of Lignocellulosic Cell Walls with Moisture. *Sci. Rep.* **2017**, *7*, 2054. [CrossRef]
21. Jhu, Y.-S.; Yang, T.-C.; Hung, K.-C.; Xu, J.-W.; Wu, T.-L.; Wu, J.-H. Nonisothermal Crystallization Kinetics of Acetylated Bamboo Fiber-Reinforced Polypropylene Composites. *Polymers* **2019**, *11*, 1078. [CrossRef] [PubMed]

22. Sujaritjun, W.; Uawongsuwan, P.; Pivsa-Art, W.; Hamada, H. Mechanical Property of Surface Modified Natural Fiber Reinforced PLA Biocomposites. *Energy Procedia* **2013**, *34*, 664–672. [CrossRef]
23. Hu, W.; Zhang, Y.; Qi, Y.; Wang, H.; Liu, B.; Zhao, Q.; Zhang, J.; Duan, J.; Zhang, L.; Sun, Z.; et al. Improved Mechanical Properties and Flame Retardancy of Wood/PLA All-Degradable Biocomposites with Novel Lignin-Based Flame Retardant and TGIC. *Macromol. Mater. Eng.* **2020**, *305*, 1900840. [CrossRef]
24. Yu, W.W.; Yuan, T.C.; Yao, Y.; Deng, Y.H.; Wang, X.Z. PLA/Coffee Grounds Composite for 3D Printing and Its Properties. *Forests* **2023**, *14*, 367. [CrossRef]
25. *ASTM D790-10*; Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. ASTM International: West Conshohocken, PA, USA, 2016.
26. *ASTM D256-03*; Standard Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics. ASTM International: West Conshohocken, PA, USA, 2017.
27. *ASTM D5229/D5229M-20*; Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials. ASTM International: West Conshohocken, PA, USA, 2020.
28. Fu, Q.; Xu, W.; Bu, M.; Guo, B.; Niu, D. Orthogonal experimental study on hybrid-fiber high-durability concrete for marine environment. *J. Mater. Res. Technol.* **2021**, *13*, 1790–1804. [CrossRef]
29. Liu, H.; Zheng, G.; Cheng, X.; Yang, X.; Zhao, G. Simulation Analysis of the Influence of Nozzle Structure Parameters on Material Controllability. *Micromachines* **2020**, *11*, 826. [CrossRef]
30. Li, X.; Hao, J. Orthogonal test design for optimization of synthesis of super early strength anchoring material. *Constr. Build. Mater.* **2018**, *181*, 42–48. [CrossRef]
31. Han, Z.Y.; Ma, D.; Chen, Q.Y.; Wang, Y.H. Orthogonal test design for optimization of synthesis of Bi_2WO_6 superstructure with high photocatalytic activity by hydrothermal method. *J. Chem. Technol. Biotechnol.* **2021**, *97*, 943–949. [CrossRef]
32. Zhang, F.; Wang, B.; Jie, P.; Zhu, J.; Cheng, F. Preparation of chitosan/lignosulfonate for effectively removing Pb(II) in water. *Polymer* **2021**, *228*, 123878. [CrossRef]
33. Chavan, S.; Gaikwad, A. Optimization of enzymatic hydrolysis of bamboo biomass for enhanced saccharification of cellulose through Taguchi orthogonal design. *J. Environ. Chem. Eng.* **2021**, *9*, 104807. [CrossRef]
34. Fiore, V.; Di Bella, G.; Valenza, A. The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites. *Compos. Part B Eng.* **2015**, *68*, 14–21. [CrossRef]
35. Arrakhiz, F.Z.; Elachaby, M.; Bouhfid, R.; Vaudreuil, S.; Essassi, M.; Qaiss, A. Mechanical and thermal properties of polypropylene reinforced with Alfa fiber under different chemical treatment. *Mater. Des.* **2012**, *35*, 318–322. [CrossRef]
36. Yan, L.; Chouw, N.; Huang, L.; Kasal, B. Effect of alkali treatment on microstructure and mechanical properties of coir fibres, coir fibre reinforced-polymer composites and reinforced-cementitious composites. *Constr. Build. Mater.* **2016**, *112*, 168–182. [CrossRef]
37. Yousif, B.F.; Shalwan, A.; Chin, C.W.; Ming, K.C. Flexural properties of treated and untreated kenaf/epoxy composites. *Mater. Des.* **2012**, *40*, 378–385. [CrossRef]
38. Feng, M.; Pan, Y.; Zhang, M.; Gao, Q.; Liu, C.; Shen, C.; Liu, X. Largely improved thermal conductivity of HDPE composites by building a 3D hybrid fillers network. *Compos. Sci. Technol.* **2021**, *206*, 108666. [CrossRef]
39. Zhang, K.; Chen, Z.; Smith, L.M.; Hong, G.; Song, W.; Zhang, S. Polypyrrole-modified bamboo fiber/polylactic acid with enhanced mechanical, the antistatic properties and thermal stability. *Ind. Crops Prod.* **2021**, *162*, 113227. [CrossRef]
40. Ramamoorthy, S.K.; Bakare, F.; Herrmann, R.; Skrifvars, M. Performance of biocomposites from surface modified regenerated cellulose fibers and lactic acid thermoset bioresin. *Cellulose* **2015**, *22*, 2507–2528. [CrossRef]
41. Chandrasekar, M.; Ishak, M.R.; Sapuan, S.M.; Leman, Z.; Jawaid, M. A review on the characterisation of natural fibres and their composites after alkali treatment and water absorption. *Plast. Rubber Compos.* **2017**, *46*, 119–136. [CrossRef]
42. Nor Arman, N.S.; Chen, R.S.; Ahmad, S. Review of state-of-the-art studies on the water absorption capacity of agricultural fiber-reinforced polymer composites for sustainable construction. *Constr. Build. Mater.* **2021**, *302*, 124174. [CrossRef]
43. Haameem, J.A.M.; Majid, M.S.A.; Afendi, M.; Marzuki, H.F.A.; Hilmi, E.A.; Fahmi, I.; Gibson, A.G. Effects of water absorption on Napier grass fibre/polyester composites. *Compos. Struct.* **2016**, *144*, 138–146. [CrossRef]
44. Nabinejad, O.; Debnath, S.; Ying, T.J.; Liew, W.Y.H.; Davies, I.J. Influence of Alkali Treatment and Nanoclay Content on the Properties of Rice Husk Filled Polyester Composites. *Mater. Sci. Forum* **2017**, *882*, 89–100. [CrossRef]
45. Hassan, M.M.; Wagner, M.H.; Zaman, H.U.; Khan, M.A. Study on the Performance of Hybrid Jute/Betel Nut Fiber Reinforced Polypropylene Composites. *J. Adhes. Sci. Technol.* **2012**, *25*, 615–626. [CrossRef]