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Enhancing mechanical strength of bamboo parring (Gigantochloa Atter) fiber with alkaline treatment for flame-retardant composite

Massriyady Massaguni[1] · Zulkifli Djafar[2] · Rozli Zulkifli[3] · Lukmanul Hakim Arma[2]

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Abstract

Parring bamboo fiber (*Gigantochloa Atter*), or PaBFeR, has the potential to be a composite reinforcing with mechanical properties that can be improved through alkali/ Sodium Hydroxide (NaOH) and flame retardant (FR) treatment. This study evaluated the effect of NaOH treatment with concentration variations of 3%, 5%, 7%, and 11% and immersion times of 45, 90, and 135 min on the mechanical properties of PaBFeR. Mechanical testing involves a single fiber tensile test, PaBFeR (S-PaBFeR-TST), with ASTM D3779 standard, and PaBFeR single fiber pull-out test (S-PaBFeR-POT). The test results showed that excessive NaOH treatment (NaOH 7% to 11%) reduced the mechanical strength of PaBFeR. Optimal results obtained at 5% NaOH for 45 resulted in a fiber tensile strength of 249.54 ± 0.11 MPa, tensile strain of 3.35 ± 0.01%, and shear strength of 14.47 ± 0.69 MPa without fiber pulled out from the matrix (0% pull-out). This value was validated by the significant difference in values from other samples with ANOVA (P Value < 0.05) and the Tukey HSD comparison showing differences in all sample groups during NaOH treatment. Furthermore, the fibers with this optimal treatment (NaOH5%45) were treated with flame retardant (FR) for 30, 45, and 60 min. The test results showed that excessive FR inhibitor treatment duration (45 min to 60 min) decreased the mechanical performance and adhesion of the matrix and PaBFeR. The 30-min FR treatment showed the best performance with an increase in tensile strain up to 4.59 ± 0.23%, while the tensile strength of the fiber decreased slightly to 245.81 ± 0.14 MPa, and the shear strength to 12.82 ± 0.07 MPa. These results validated the significant difference in values from other samples with ANOVA (P Value < 0.05) and the Tukey HSD comparison showing differences in all sample groups during FR inhibitor treatment. These findings contribute to developing PaBFeR-based natural fiber-based composite materials for applications in environments requiring high mechanical and thermal resistance.

Highlights

1. PaBFeR is treated with NaOH and FR inhibitors for optimal mechanical strength in PaBFeR-based refractory composite applications.
2. A 45-min 5% NaOH treatment effectively reduced lignin and hemicellulose measurably, improving the mechanical strength and adhesion of the matrix and PaBFeR.
3. The refractory FR inhibitor treatment on PaBFeR in a measured manner, which is a maximum of 30 min, offers optimal mechanical strength, that is, although the tensile strength of the fibers decreases, the tensile strain still increases and maintains the adhesion of the resin to PaBFeR with 5% NaOH treatment for 45 min

✉ Zulkifli Djafar, zulkiflidjafar3065@gmail.com; Massriyady Massaguni, massriyady.massaguni@atim.ac.id; Rozli Zulkifli, rozlizulkifli@ukm.edu.my; Lukmanul Hakim Arma, armalh@yahoo.com | [1]Politeknik ATI Makassar, Makassar 90211, Indonesia. [2]Hasanuddin University, Gowa 92171, Indonesia. [3]Universiti Kebangsaan Malaysia, 43600 Bangi, Malaysia.

4. The synergy of these two treatments is vital to know so that the PaBFeR-based composite material manufacturing process still pays attention to the material's mechanical strength and adhesion and increases the fibers' fire resistance.

Keywords Gigantochloa Atter · NaOH treatment · Flame-retardant treatment · Mechanical strength improvement · BaPFeR single fiber

1 Introduction

Bamboo natural fibers have become an attractive alternative in developing sustainable composite materials due to their abundant, lightweight, and environmentally friendly availability [1]. However, the natural characteristics of bamboo fibers, such as low adhesion to the matrix and susceptibility to heat, must be improved to be optimally applied in a wide range of heat-resistant or fire-resistant composite products [2] such as insulator walls and safety materials in the industry.

Parring Bamboo or *Gigantochloa Atter* (Fig. 1) is a bamboo that thrives in tropical areas such as Indonesia, Singapore, Malaysia, Thailand, Vietnam, the Philippines, Brunei Darussalam, Myanmar, Laos, and Cambodia. This bamboo has anatomical characteristics, such as the length of the fibers that vary by height, as well as the diameter of the fibers and lumens affected by the age of the stem, giving it special advantages in strength and durability, making it ideal for use in a variety of purposes [3].

Bamboo fiber is obtained by several treatments, such as mechanization using manual tools by hitting bamboo strips so that they become fibers or using machines such as hammermills. The other method is chemical treatment, such as NaOH treatment, which works chemically to reduce lignin and hemicellulose so that fiber can be obtained or extracted. Chemical methods are widely used because they have been tested to remove non-cellulose components, such as lignin and hemicellulose, which can improve the surface structure of fibers and bonds with polymer matrices in composite materials [4].

Alkaline treatment has been proven to significantly affect the tensile strength and modulus of bamboo fibers at optimal concentrations. The addition of concentration makes bamboo fibers b rittle and tenacious. However, an alkaline concentration that is too high can lead to the degradation of the fiber structure, which decreases its mechanical strength. In addition, the alkaline treatment also affects the surface morphology of the fibers by increasing the effective area and surface roughness, resulting in better fiber-matrix adhesion in the composite [5].

Several previous studies have shown that alkaline treatment significantly improves the mechanical properties of bamboo fibers. Zhang et al. showed that 4% NaOH treatment for 1 h improved Moso bamboo fibers'surface roughness and effective area and significantly increased tensile strength to 211 MPa from the normal treatment of 192 MPa [6]. Salih et al. report that treatment of single bamboo fiber Gigantochloa Scortechinii with 4% NaOH for 12 h resulted in the highest fiber tensile strength of 356.8 MPa compared to 234.8 MPa at 8% NaOH treatment, which decreases strength due to structural degradation [7]. Meanwhile, research by dos Santos et al. showed that the application of bamboo (Bambusa Vulgaris)-polyester hybrid composite with 5% NaOH treatment for 2 h resulted in better bending and tensile strength than untreated composites, namely up to 255.1 MPa for the tensile strength of the composite and 215.3 MPa for the bending strength [8].

On the other hand, flame retardant treatment of bamboo fibers is also important, especially for composite applications that require heat and fire-resistant materials. Although this treatment can improve the flame retardant properties and thermal stability, some studies such as the one by Tang et al. have shown a decrease in the mechanical properties of the fibers due to the use of materials such as boron compounds, melamine polyphosphate (MPP), and aluminum diethylhypophosphite (DPP) in bamboo fiber-reinforced composites [9]. Meanwhile, a study conducted by Fang et al. found that the benefits of flame retardant treatment in reducing the heat dissipation rate, extending the ignition time, and improving thermal resistance make it an important solution in developing flame retardant composite materials [10].

There is little information about the effect of flame retardant on the mechanical strength of bamboo fibers, which only focuses on the mechanical strength effects of flame retardant composites. This information is important to know to understand the effects of fire-retardant treatment on bamboo fibers before they are applied as reinforcing materials in nature-based fire-retardant composites and various types of flame-retardant polymer matrices that have been widely fabricated, such as flame-retardant polyester.



Fig. 1 Bamboo parring (*Gigantochloa Atter*)

The synergistic effect of alkaline and flame retardant inhibitor treatments on bamboo fiber holistically is unknown, considering that the current bamboo fiber extraction process uses more chemical treatments, such as NaOH. This combination of alkaline and flame retardant treatment allows for a synergistic approach to optimise bamboo fibres' mechanical and thermal properties simultaneously. In addition, this combination also allows minimizing mechanical weakness caused by a single flame retardant treatment, thus creating composite materials that have better performance for structural and protective applications.

In addition to the tensile strength of the fibers, the effects of alkali and flame retardant on fiber and matrix relationships are also important to be known through pull-out testing. The pull-out test aims to evaluate the strength of the interface bond between bamboo fibers and the matrix in composite materials. These tests help understand the mechanical performance and durability of fiber-reinforced composites, as some previous studies have shown that bamboo fibers are used with thermoplastic (e.g., PLA, polypropylene) and thermoset (e.g., epoxy) matrices [11, 12].

This study aims to improve the mechanical strength of bamboo fiber parring/PaBFeR as a refractory composite raw material. This study focuses not only on the effects of a single treatment but also holistically shows the synergistic relationship of NaOH treatment and flame retardant inhibitors on the mechanical strength and microstructure of bamboo fibers. The novelty offered is a new insight into the mechanical properties of PaBFeR. Bamboo parring or Gigantochloa Atter was chosen because it is abundant in the tropics and is a sustainable raw material. However, it is possible that this method can be applied to other types of bamboo using the method used in this study. Therefore, this study adds the latest information on developing PaBFeR-based flame-retardant composites and polymer matrices.

2 Materials and methodology

2.1 Alkali treatment process

PaBFeR specimens were prepared by selecting 3-year-old parring bamboo (PaB) stems. The PaB cutting area was set at 100 cm from the ground, and a 120 cm length was used as the sample area. The PaB sample was then split into strips using a bamboo splitting tool. Figure 2 shows a macro photo of the alkaline-treated bamboo fiber. The alkalization process aimed to reduce the bamboo fibers'lignin content (delignification), improve the adhesion between the fiber and the matrix, and improve the tensile strength of certain bamboo fibers within the existing reference range of 3–6%. So in this study, four NaOH concentrations were used (3%, 5%, 7%, and 11%) with soaking durations of 45, 90, and 135 min. This variation in treatment time is based on preliminary observations. Untreated specimens



Fig. 2 Macro photo of alkaline treatment of bamboo fiber

(Normal) were also prepared for comparison. Sample codes were structured as NaOHX%Y, where X represents concentration and Y represents duration. Example: NaOH5%45. After alkaline treatment, the PaB strips were processed using a hammer mill machine to extract PaBFeR. In the tensile and pull-out tests, a PaBFeR measuring 0.5 mm in diameter was prepared.

2.2 Flame retardant treatment process

Following the alkali treatment and mechanical testing (single fiber tensile test and pull-out test), fibers with the most optimal performance were selected for flame retardant (FR) treatment. This involved immersion in ABS AF 21 M flame retardant liquid for 0 min (Normal), 30 min, 45 min, and 60 min. The sample codes were labelled as NaOHX%Y + FRZ, representing alkali treatment plus flame-retardant soaking time (Z). Example: NaOH5%45 + FR30.

The stages of the research are shown in the following Fig. 3.

2.3 Single PaBFeR Tensile Test (S-PaBFeR-TST)

S-PaBFeR-TST using the universal testing machine with a load cell of 500 kN and a pulling speed of 1 mm per minute. The single fiber tensile test specimen, shown in Fig. 4, is manufactured in accordance with the ASTM D3379 standard.

The tensile strength of BaPFeR is calculated using the formula:

$$\sigma = \frac{F}{A} \tag{1}$$

σ is the tensile strength (MPa), F is the tensile force on the fiber (N), and A is the cross-sectional area of the fiber (mm^2). The percentage of tensile strain is measured by:

$$\varepsilon = \frac{\Delta L}{L_0} \times 100\% \tag{2}$$

ε is the strain percentage (%), ΔL is the change in fiber length during the test (mm), and L0 is the initial fiber length (mm). In a single fiber tensile test, each sample population was tested five times, and then the results were averaged, variance analyzed, and further comparative testing using Minitab Statistical Software 22.

2.4 Single PaBFeR pull-out test (S-PaBFeR-POT)

The S-PaBFeR-POT was carried out using a universal testing machine with a load cell of 500 kN and a pulling speed of 1 mm per minute with a distance between clamps of 5 mm [13, 14]. The pull-out test specimen is made by the following Fig. 5:

The maximum load Fmax (N) is determined in the calculation of interfacial shear strength (IFSS) with the formula:

$$\tau = \frac{F_{max}}{\pi \cdot d \cdot l} \tag{3}$$

Which "d" is the diameter of PaBFeR (mm), and "l" is the length of the fibers embedded in the FR-P (Flame-Retardant Polyester) resin (mm). In the single fiber pull-out test, it was observed whether the fiber was pulled out in the resin

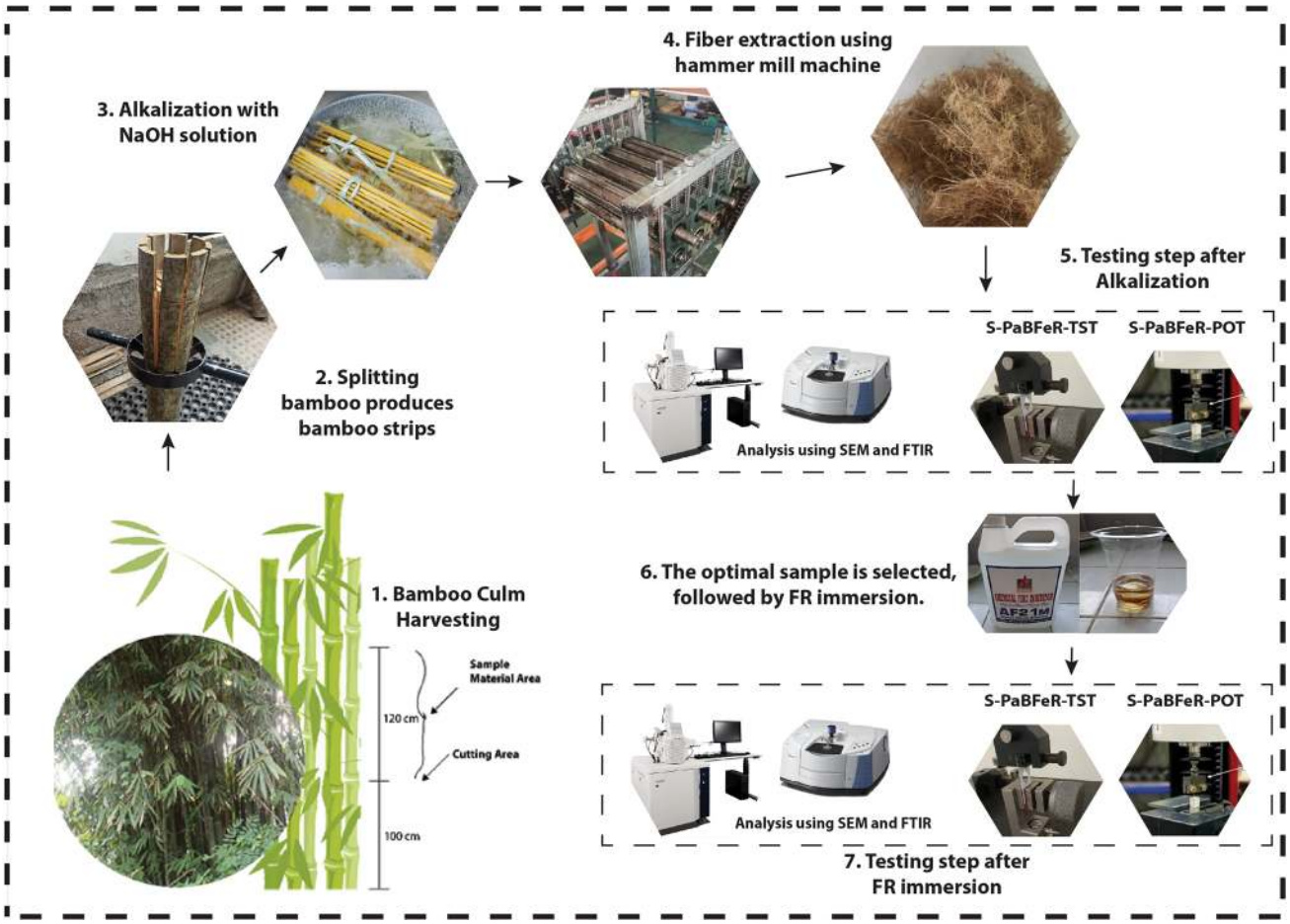

Fig. 3 Stages of research

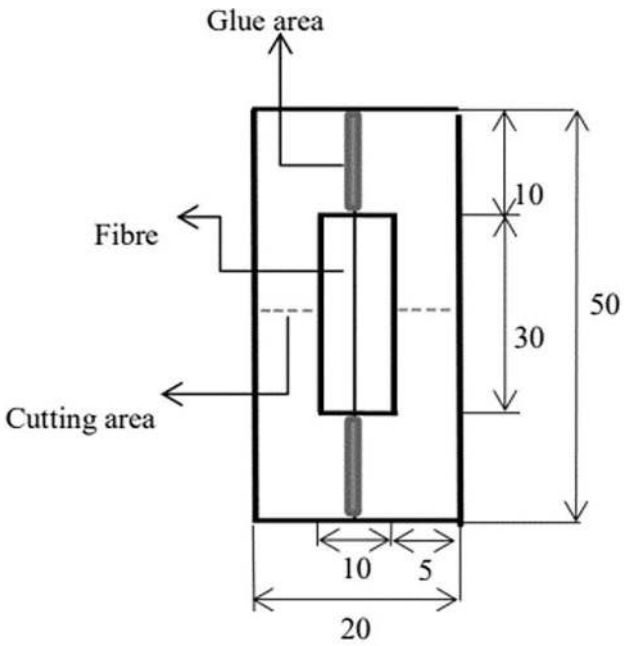

Fig. 4 ASTM D3379 single fiber test specimen

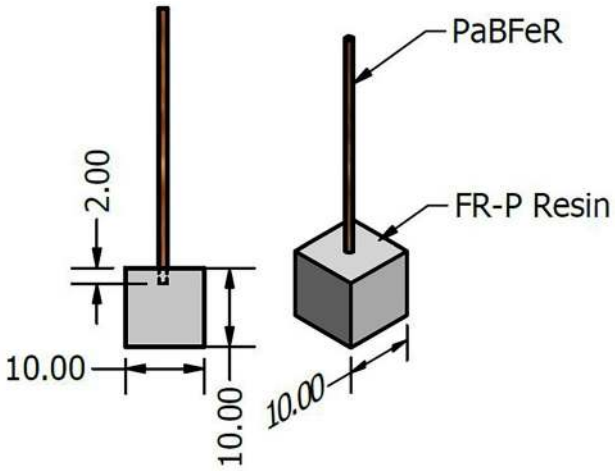

Fig. 5 PaBFeR single fiber pull-out test specimen design with FR-P matrix

or not (broken) and assessed in the form of a percentage (0%–100%) of the total number of samples. Each sample population was tested five times, and then the results were averaged and variance analyzed (ANOVA) and further comparative testing using Minitab Statistical Software 22.

2.5 Scanning electron microscope (SEM)

Specimens with optimal and minimum mechanical properties were selected for morphological analysis using SEM. SEM analysis was conducted to observe the surface morphology of the treated PaBFeR fibers, including microstructural features such as fiber surface roughness, cracks, or evidence of delignification. This characterization provided visual insight into the fibers'structural integrity and adhesion potential after alkali and flame retardant treatments.

2.6 Fourier transform infrared spectroscopy (FTIR)

FTIR was performed to identify the chemical functional groups present in the fibers. FTIR analysis was essential for detecting chemical changes resulting from NaOH and FR treatments, such as the reduction of lignin and hemicellulose content or the appearance/disappearance of specific functional bonds (e.g., O–H, C–H, C=O). These changes support the mechanical behavior observed during tensile and pull-out testing.

3 Results and discussion

3.1 NaOH treatment result

The results of S-PaBFeR-TST and S-PaBFeR-POT after NaOH treatment can be seen in Table 1.

In Table 1, 13 types of treatment conditions are shown, given by the sample code. Data obtained from five repetitions for each of these treatment conditions showed consistency of results, with a relatively small standard deviation (< 5% for tensile strength at the optimal treatment). The general pattern observed is that the results show that the NaOH5%45 treatment produces optimal performance with a tensile strength of 249.54 ± 0.11 MPa and a shear stress of 14.47 ± 0.69 MPa without pull-out (0%), an increase in mechanical strength in the concentration range of 3–5% NaOH, and a decrease of > 7% NaOH. In addition, tensile strain stability is in the range of 3.2–3.4% for all NaOH treatments, and the presence of fiber-matrix adhesions is indicated by a decrease in pull-out percentage.

Comparison of mechanical strength, validation, and data comparison of each sample with NaOH treatment is shown in the following Fig. 6.

Figure 6 shows a comparison of mechanical strength, ANOVA results, and Tukey's follow-up test, which shows that NaOH treatment has a significant effect on the tensile strength, tensile strain, and shear strength of PaBFeR fibers (p-value = 0.000). On tensile strength, a statistical F-value of 10.38 indicates that the treatment makes a real difference between groups. The Tukey test showed that 5% NaOH for 45 min produced the highest tensile strength (249.54 MPa, group A),

Table 1 Results of S-PaBFeR-TST and S-PaBFeR-POT treatment NaOH

Sampel code	S-PaBFeR-TST		S-PaBFeR-POT	
	Tensile strength (MPa)	Tensile strain (%)	Shear strength (MPa)	Pull out (%)
N	224.99 ± 7.88	4.53 ± 0.27	5.57 ± 1.74	100
NaOH3%45	246.16 ± 4.28	3.34 ± 0.004	13.60 ± 0.47	0
NaOH3%90	240.32 ± 8.53	3.40 ± 0.06	11.67 ± 0.27	0
NaOH3%135	237.33 ± 5.62	3.23 ± 0.14	9.62 ± 0.25	0
NaOH5%45	249.54 ± 0.11	3.35 ± 0.01	14.47 ± 0.69	0
NaOH5%90	246.33 ± 2.42	3.37 ± 0.06	13.53 ± 0.21	0
NaOH5%135	245.12 ± 4.97	3.33 ± 0.01	12.16 ± 0.12	0
NaOH7%45	240.36 ± 14.13	3.42 ± 0.16	12.69 ± 0.35	80
NaOH7%90	236.48 ± 14.63	3.31 ± 0.01	11.92 ± 0.82	60
NaOH7%135	222.49 ± 11.43	3.31 ± 0.01	9.63 ± 0.46	60
NaOH11%45	230.43 ± 7.01	3.22 ± 0.12	10.72 ± 1.09	80
NaOH11%90	221.29 ± 2.60	3.28 ± 0.07	7.95 ± 0.88	100
NaOH11%135	212.53 ± 4.52	3.22 ± 0.13	5.32 ± 0.51	80

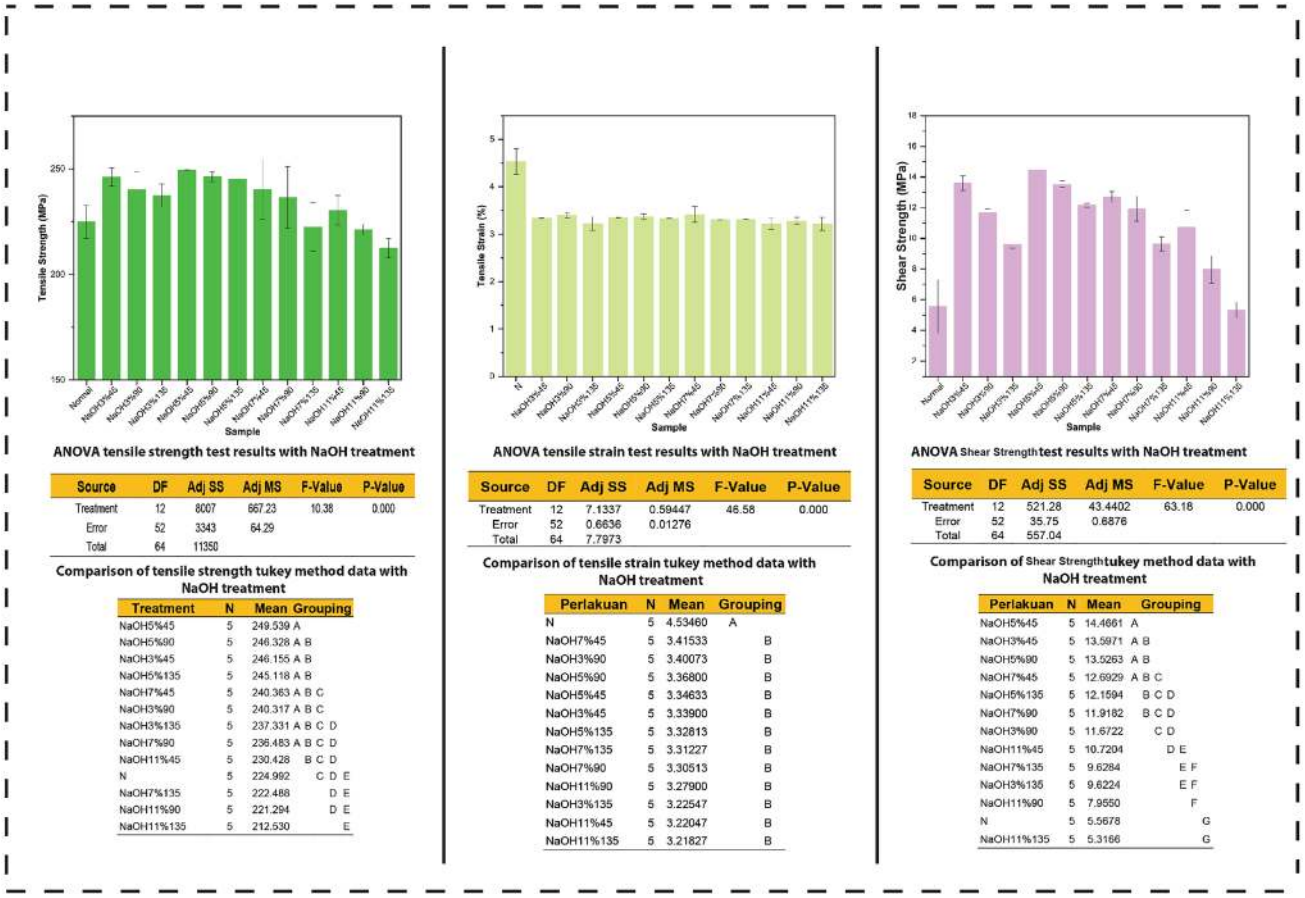

Fig. 6 Comparison of mechanical strength, validation, and data compression of BaPFeR samples of NaOH treatment

while 11% NaOH for 135 min produced the lowest tensile strength (212.53 MPa, group E), suggesting that high concentrations and long durations can lead to fiber degradation.

For tensile strain, the F-statistical value of 46.58 showed a significant difference between treatment groups. Untreated fibers had the highest strain (4.53%, group A), while all NaOH treatments were included in group B with lower values (3.22%–3.42%), indicating that NaOH reduced fiber elasticity with no significant differences between treatment variations.

At shear strength, the F-statistical value of 63.18 indicates a very significant treatment effect. 5% NaOH for 45 min produced the highest shear strength (14.47 MPa, group A), while 11% NaOH for 135 min and untreated fibers had the lowest values (5.57 and 5.32 MPa, group G). This suggests that NaOH in high concentrations and durations can weaken the adhesion properties of fibers. Overall, the optimal treatment to improve the mechanical properties of PaBFeR fibers is 5% NaOH for 45 min, while overtreatment tends to degrade fiber quality due to degradation.

The following SEM results from PaBFeR NaOH5%45 are shown in Fig. 7.

Figure 7 shows a comparison of the microstructure of PaBFeR bamboo fibers that have undergone chemical treatment with 5% NaOH solution for 45 min and 11% NaOH for 135 min, with 500× magnification. The microstructure of bamboo fibers treated with 5% NaOH for 45 min appeared cleaner and more structured, showing optimal removal of non-cellulose components such as lignin and hemicellulose without causing excessive damage. The small cracks (crack areas) that appear are insignificant and do not interfere with the overall integrity of the fibers, which contributes to an increase in tensile strength up to 249.54 ± 0.11 MPa. Meanwhile, the structure of bamboo fibers treated with 11% NaOH for 135 min showed more significant degradation in the presence of wider cracks and more irregular fiber structures. This led to a decrease in tensile strength to 212.53 ± 4.52 MPa, which indicates that overly intensive alkali treatment can weaken the structural integrity of the fibers.

In addition to SEM analysis, the FTIR spectrum of PaBFeR fibers that had been treated with 5% and 11% NaOH solutions showed significant changes in functional group characteristics, as shown in Fig. 7. The main peak in the wavenumber range of 3600–3200 cm^{-1}, which indicates the presence of hydroxyl groups (–OH) of cellulose, hemicellulose, and lignin, showed a slight decrease in intensity after treatment with 11% NaOH, indicating partial removal of hemicellulose and lignin. In addition, the peak at 3288.18 cm^{-1} associated with hydrogen bonds in the cellulose structure is weakened, indicating an increase in regularity in the cellulose structure due to the degradation of amorphous components. Further changes were observed in the range of 1592.45 cm^{-1} associated with aromatic C=C vibrations in lignin, where the intensity was reduced after treatment with 11% NaOH, indicating a greater degradation of lignin compared to 5% NaOH.

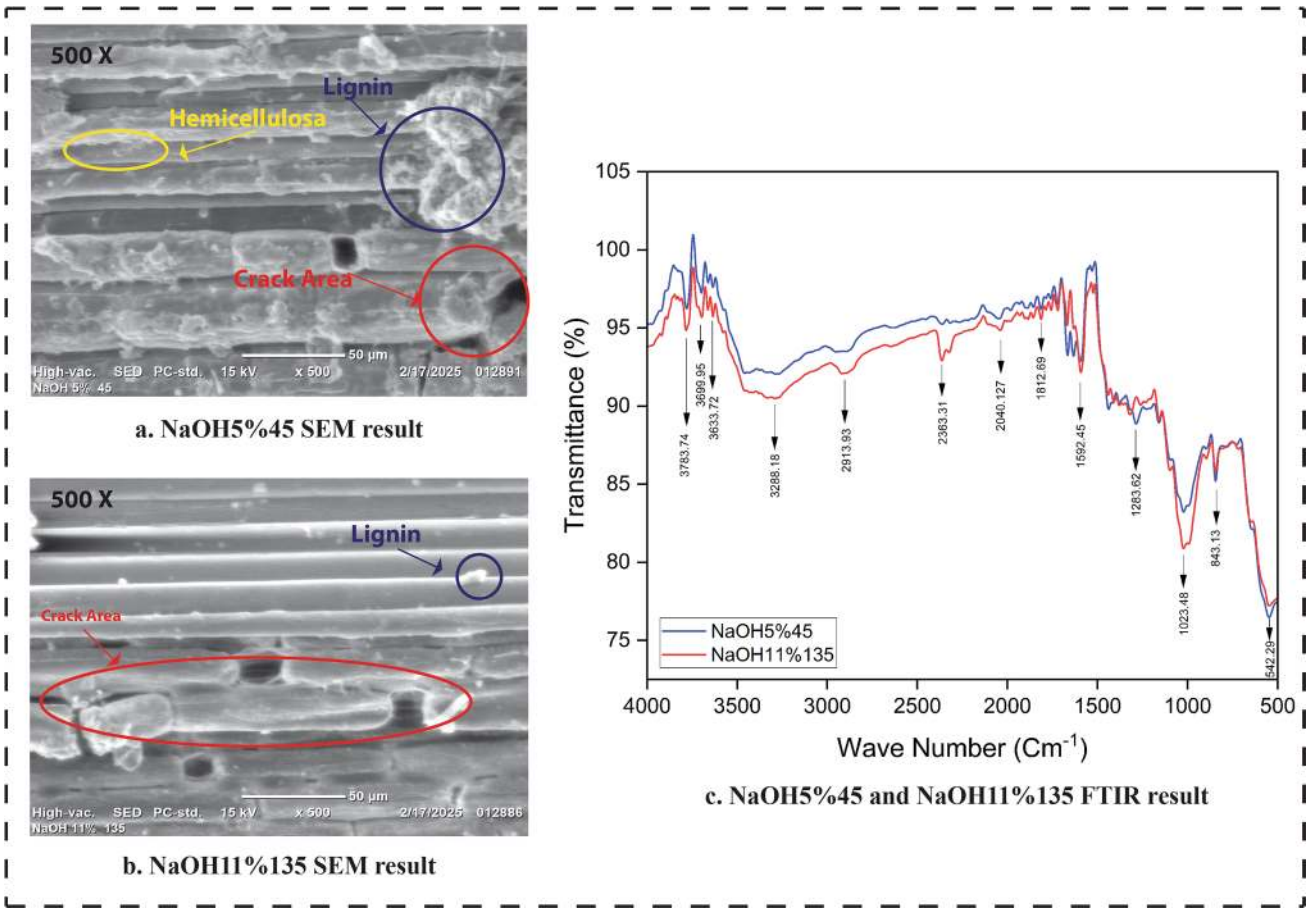

Fig. 7 SEM and FTIR Results of PaBFeR NaOH5%45 and NaOH11%135 sample

However, the absorption band at 1023.48 cm^{-1} associated with C–O vibrations in cellulose remained stable, suggesting that the main structure of cellulose was preserved despite the degradation of lignin and hemicellulose.

Based on the results of SEM and FTIR analysis, treatment with NaOH had a significant effect on the structure and chemical composition of PaBFeR bamboo fibers. Treatment with 5% NaOH for 45 min showed optimal results in increasing the tensile strength of the fibers without causing excessive damage, while 11% NaOH for 135 min led to greater degradation of the fiber structure, resulting in a decrease in tensile strength. Thus, the concentration and immersion time of NaOH need to be optimally controlled to achieve a balance between the removal of non-cellulose components and maintaining the structural integrity of bamboo fibers.

3.2 FR treatment result

The results of S-PaBFeR-TST and S-PaBFeR-POT after FR treatment in the optimal sample of NaOH5%45 can be seen in the following Table 2.

Previous results of PaBFeR fiber samples that were alkalized with 5% NaOH for 45 min (NaOH5%45) showed a tensile strength of 249.54 ± 0.11 MPa, with a strain of 3.35 ± 0.01%; this value will be a comparison for FR treatment samples for 30 min, 45 min, and 60 min. After receiving additional treatment with FR substances for 30 min (NaOH5%45 + FR30), there was a slight decrease in tensile strength to 245.81 ± 0.14 MPa, but the strain increased to 4.59 ± 0.23%, which indicates an increase in deformation ability before breaking. The 45-min FR treatment (NaOH5%45 + FR45) resulted in a further decrease in tensile strength to 241.87 ± 1.18 MPa, with the strain again approaching the initial value of 3.34 ± 0.003%. At the 60 min FR treatment (NaOH5%45 + FR60), there was a more significant decrease in tensile strength to 237.26 ± 1.78 MPa, while the strain increased slightly to 3.40 ± 0.06%. In addition, the resulting shear strength reached 14.47 ± 0.69 MPa, while the pull-out value was at 0%, indicating the absence of loose fibers in the test. The shear strength decreased to 12.82 ± 0.07 MPa, while the pull-out value remained at 0%, indicating that this treatment had not yet led to the release of the fibers from the matrix. The shear strength drops to 11.87 ± 0.05 MPa, which indicates that the increase in the immersion time of the Flame Retardant reduces the binding power between the components. The pull-out value remains at 0%, which indicates that the fibers are still well embedded in the matrix. The shear strength continues to decrease to 11.17 ± 0.12 MPa, indicating a diminishing cohesion between materials. The pull-out value also increased to 14.8%, which suggests that immersion in FR over a longer duration began to lead to the release of fibers from the matrix, which likely affected the overall mechanical integrity of the composite.

Comparison of mechanical strength, validation, and data comparison of each sample with FR treatment is shown in the following Fig. 8.

ANOVA results showed that FR treatment had a significant effect on the tensile strength, tensile strain, and shear strength of PaBFeR NaOH5%45 fibers (p-value = 0.000). On tensile strength, the F-statistical value of 40.31 confirms that the variation due to FR treatment is much greater than the variation in the group. The Tukey test showed that the 30-min FR treatment did not make a significant difference compared to the sample without FR. Still, there was a significant decrease from 45 min onwards, which intensified at 60 min. For tensile strains, the F-statistical value of 149.52 showed a strong FR treatment effect, with the highest strain at 30 min, while longer treatments (45 and 60 min) lowered the strain and did not differ significantly from samples without FR. At shear strength, a statistical F-value of 102.81 confirms that the variation between treatments is dominant. The Tukey test showed that samples without FR had the highest shear strength, which decreased as the duration of the FR treatment increased. Overall, FR treatments tend to decrease tensile strength and shear strength after 45 min, while tensile increases at 30 min but decreases afterwards.

In Fig. 9, SEM and FTIR spectra for FR treatment at PaBFeR NaOH5%45 with 500× magnification, it can be seen that the fiber structure undergoes significant changes due to FR treatment at various durations. At 500× magnification, the

Table 2 Results of S-PaBFeR-TST and S-PaBFeR-POT after FR treatment in optimal samples, namely NaOH5%45

Sample Code	S-PaBFeR-TST		S-PaBFeR-POT	
	Tensile strength (Mpa)	Tensile strain (%)	Shear strength (N/mm2)	Pull-out (%)
NaOH5%45	249.54 ± 0.11	3.35 ± 0.01	14.47 ± 0.69	0
NaOH5%45 + FR30	245.81 ± 0.14	4.59 ± 0.23	12.82 ± 0.07	0
NaOH5%45 + FR45	241.87 ± 1.18	3.34 ± 0.003	11.87 ± 0.05	0
NaOH5%45 + FR60	237.26 ± 1.78	3.40 ± 0.06	11.17 ± 0.12	14.8

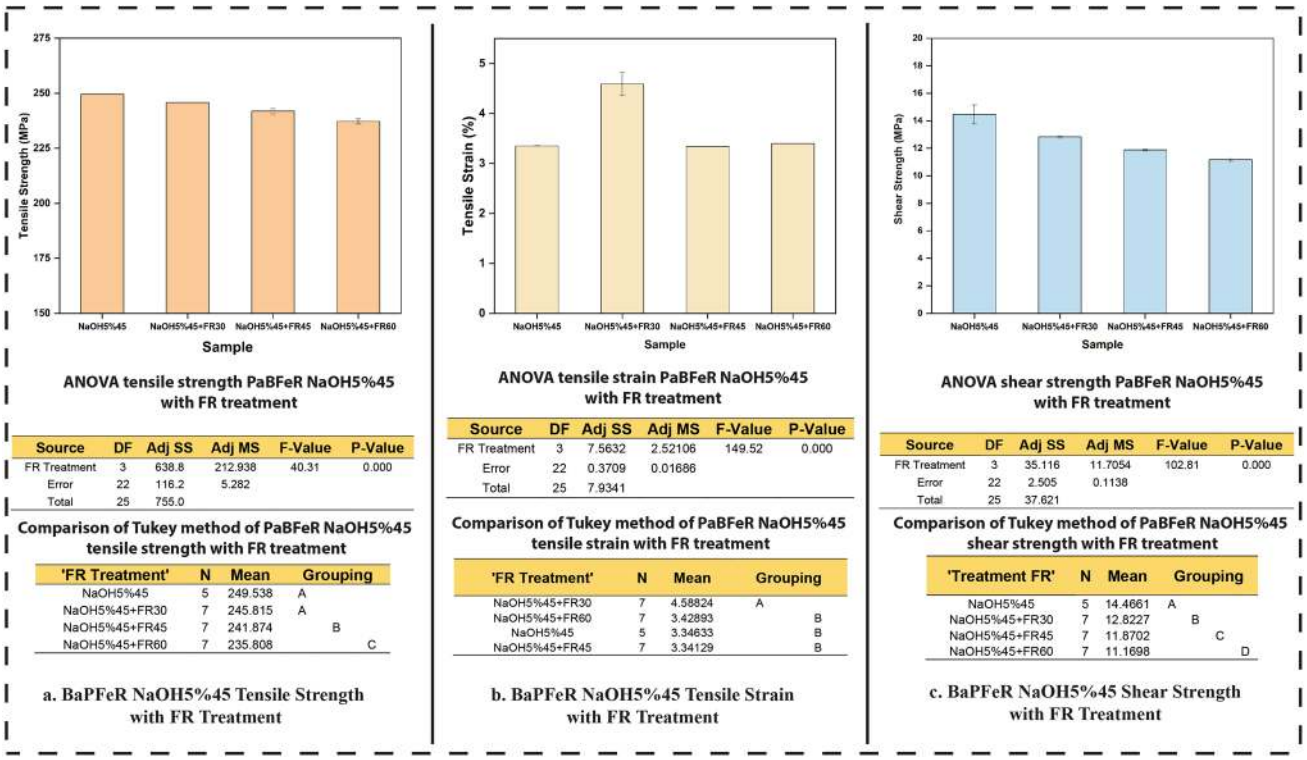

Fig. 8 Comparison of mechanical strength, validation, and data compression of BaPFeR samples of NaOH5%45 with FR treatment

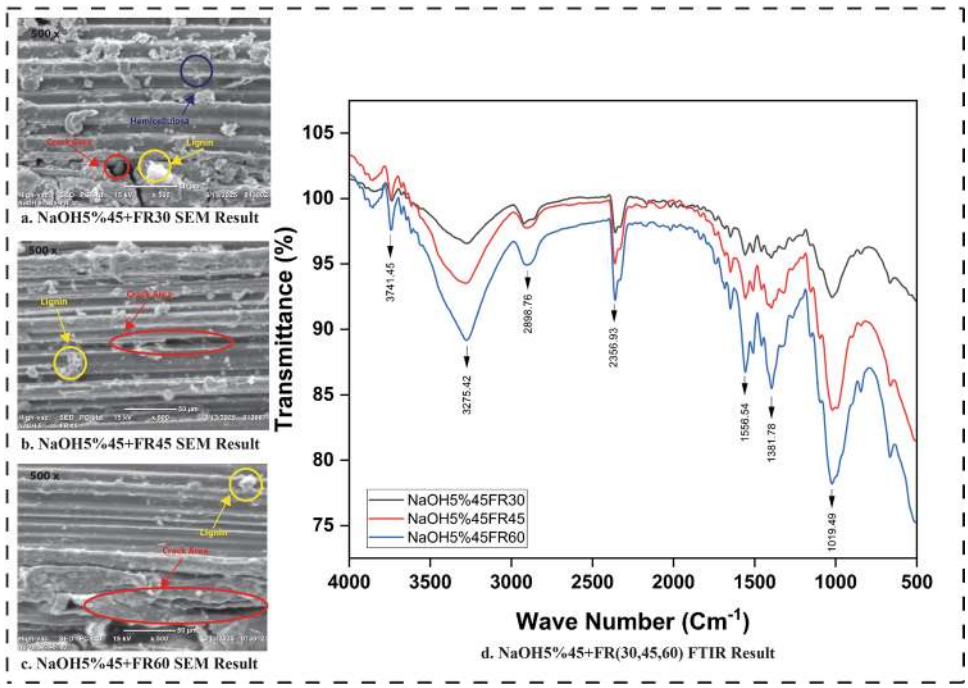

Fig. 9 SEM and FTIR Results of PaBFeR NaOH5%45 sample with FR Treatment

sample with 30 min FR treatment still retained a relatively intact fiber structure, even though small cracks had begun to appear. The components of hemicellulose and lignin can still be identified, although they appear to be slightly fragmented due to interaction with FR. In the FTIR spectrum, the peak absorption at 3741.45 cm^{-1} and 3275.42 cm^{-1} indicates that the hydroxyl group is still quite dominant, indicating that chemical modifications have not been too intense. The absorption band at 2898.76 cm^{-1} related to aliphatic C–H vibration remained detected. In comparison,

the peak at 2356.93 cm^{-1} indicated the possibility of a volatile compound that began to form as a result of reaction with the FR compound.

In samples with 45 min FR treatment, the fiber structure appeared more fragmented compared to the 30 min treatment, with more and deeper cracks. Lignin and hemicellulose appear to be more degraded, which indicates that the 45-min FR treatment begins to disrupt the integrity of the fibers more significantly. This is in line with the results of the FTIR spectrum, where the peak at 3741.45 cm^{-1} experienced a slight shift in intensity, suggesting further interaction between the FR and the hydroxyl group. The 3275.42 cm^{-1} band still shows the presence of the –OH group but with a slight decrease in intensity due to increasingly strong chemical modifications. The peak of 2898.76 cm^{-1} still indicates aliphatic C-H vibrations, while the peak of 2356.93 cm^{-1} is still present, indicating the possibility of trapped gases or more volatile compounds forming. In addition, the 1556.54 cm^{-1} band associated with lignin showed a slight shift, indicating further lignin degradation due to FR treatment.

In samples with FR treatment for 60 min, the fiber structure experienced more severe degradation compared to the 30 and 45-min treatments. At 500× magnification, cracks appear deeper and wider, suggesting that longer FR treatment causes significant damage to the integrity of the fibers. Hemicellulose and lignin are almost no longer identified, which indicates that the degradation process has been quite intense. The results of the FTIR spectrum showed a more significant change, where the peaks of 3741.45 cm^{-1} and 3275.42 cm^{-1} experienced a sharp decrease in intensity, indicating a further reduction of hydroxyl groups due to stronger interactions with FR. The absorption band at 2898.76 cm^{-1} remained to show C–H vibrations, while the peak of 2356.93 cm^{-1} was slightly increased, indicating the possibility of the formation of more volatile compounds. The 1556.54 cm^{-1} band underwent further shifts, indicating a greater change in lignin structure due to the longer FR treatment. The peak of 1381.78 cm^{-1} experienced a slight decrease in intensity. In comparison, the peak of 1019.49 cm^{-1} associated with C–O vibrations in the cellulose structure was also further weakened, indicating that the 60-min FR treatment had led to deeper modifications to the structure of the bamboo fibers.

Treatment of 5% NaOH for 45 min on PaBFeR bamboo fiber proved to be an optimal condition in improving its mechanical properties. In the S-PaBFeR-TST test, the tensile strength value of this treatment (NaOH5%45) was higher, up to 249.54 MPa, than other treatments such as 3%, 7%, and 11%. In this condition, it can be seen that the addition of concentrations of (normal 0%), 3%, and 5% increases the tensile strength of the fibers and gradually decreases with the addition of 7% and 11%. These results are in line with previous research by Salih et al. and Zhang et al. that in the range of 3%–4%, it is able to increase the tensile strength of bamboo fibers [6, 7]. Although the study conducted found a tensile strength value of 5% in BaPFeR, the Tukey HSD comparison results of the sample tensile strength values of 3%, 5%, and 7% were in the same group, only the standard deviation of 5% sample was smaller than in other samples, so the NaOH5%45 sample was taken as the most optimal sample at tensile strength. In the context of the duration of immersion, it can be seen that the longer the immersion the tensile strength value decreases. In the initial study, the variation in soaking duration of 45 min, 90 min, and 135 min showed that the standard deviation of the tensile strength of the fiber was also higher, which indicates a decrease in the performance of the tensile strength of the fiber. This is in line with the results of Bhagat et al. research that soaking bamboo fibers for a long time (24 jam, 30 menit, dan 20 menit) reduces the tensile strength of the fibers [15]. Although it was found that some samples such as NaOH5%90 had a significant tensile strength with NaOH5%45 (Group A in Tukey HSD), the standard deviation value of NaOH5%90 was quite high and was in the same group as several other samples (Group B) so that the characteristics of this treatment material were not statistically optimal. In the context of tensile strain, NaOH treatment was significantly reduced from normal conditions (4.53 ± 0.27%) according to ANOVA validation. The tensile strain value of the NaOH treatment was significantly the same based on the Tukey comparative test, all in group B. This is due to lignin and hemicellulose acting as a natural matrix that fills the space between cellulose microfibrils, which are more amorphous and elastic in normal samples, and when treated NaOH will have implications for the removal of lignin and hemicellulose, which affects the strain value of the fibers. This is in line with what Yang et al. and Lu et al. stated that the effect of removing lignin from bamboo fibers can lead to a reduction in tensile and strain strength, as lignin and hemicellulose act to maintain the fiber's structural [16, 17].

In addition, the adhesion of the matrix was also analysed using the S-PaBFeR-POT test by assessing the shear strength and the percentage of samples removed from the matrix. The optimal value was obtained in the NaOH5% sample, which was an average of 14.47 ± 0.69 MPa with 0% of the BaPFeR sample plucked from the resin. This reinforces the optimal value of the mechanical strength of this sample. Normal samples had an average shear strength of 5.57 ± 1.74 MPa, with 100% of the BaPFeR sample plucked from the matrix, meaning that with optimal treatment (NaOH5%45) the results increased almost 3 times the shear strength and showed the best adhesion value. Meanwhile, treatment with high concentrations such as (NaOH11%45) showed a decrease with an average shear strength value of 10.72 ± 1.09 MPa or almost double under normal conditions, but showed that 80% of the samples were removed

from the matrix. This result is in line with the research of Wong et al. by comparing untreated bamboo fiber and 10% NaOH treated fiber, the result obtained was that the value of the interface relationship between polyester resin and bamboo fiber was three times better obtained in samples with NaOH treatment than without treatment [18].

The results of mechanical strength are verified by assessing the functional group and visualising the microstructure of the sample with optimal and minimum values. This was done to significantly compare the effect of NaOH on the decrease in mechanical strength. The FTIR results showed that the peaks of lignin and hemicellulose (e.g. at wavenumber 1592.45 cm^{-1} for C=C aromatic lignin and 3288.18 cm^{-1} for the –OH group) in fibers treated with 11% NaOH were directly correlated with structural degradation and decreased tensile strength in NaOH11%135 samples (212.53 ± 4.52 MPa). The reduced intensity at 1592.45 cm^{-1} confirmed the excessive delignification that damaged the natural composite structure of bamboo fibers, in which the protective lignin matrix that binds the cellulose microfibrils was degraded. Simultaneously, the weakening of the -OH peak (3288.18 cm^{-1}) indicates the occurrence of cellulose chain breaks due to alkaline hydrolysis at prolonged exposure to high concentrations. This double degradation of the lignin skeleton and cellulose backbone resulted in damage to the integrity of the fibers, triggering the formation of micro-cracks visible in SEM and a 15% decrease in tensile strength compared to fibers with 5% NaOH treatment (249.54 ± 0.11). In SEM testing, it was visually apparent that the NaOH treatment made it appear cleaner and more structured (rough-regular) with cellulose density without damage which means this contributes to the increased tensile strength and shear strength of the fibers. This happens because the microfibrils are still intact, NaOH 5% for 45 min has not broken the chain of crystalline cellulose, and the density of cellulose caused by small amounts of lignin and hemicellulose protects the microfibrils. This is in line with the research of Xu et al. who observed the extraction process of Pleioblatus Amarus bamboo with an alkaline boiling process using 4% NaOH, which suggests that NaOH in the range still leaves lignin and intact bamboo parenchyma tissue [19]. Similar results were also reported by Luna et al. that the SEM results of Guadua angustifolia bamboo fibers with NaOH treatment above 5% to 10% showed lignin and hemicellulose separated compared to treatment below 5% [20].

The FR treatment of NaOH5%45 fibers produces an interesting synergistic pattern between the requirements of mechanical and flame retardant properties. Flame retardant treatment of NaOH5%45 fibers shows results that vary depending on the duration of application. The 30-min immersion resulted in the best performance with a tensile strength of 245.81 ± 0.14 MPa and an increase in strain of up to 4.59 ± 0.23%, suggesting that the penetration of FR compounds in a short period is able to fill the cavities between the fibers without damaging the base structure. The SEM results showed that the microcracks formed during the alkaline treatment did not experience significant expansion after the FR30 treatment, supported by the FTIR results, which showed the stability of the cellulose functional group. However, a longer treatment of 60 min leads to a significant decrease in mechanical strength due to the thermo-chemical degradation process. Further analysis revealed that under the FR60 condition, the ammonium compounds in the acidic FR solution reacted with the cellulose hydroxyl group at a temperature of about 40–45 °C, triggering hydrolysis that weakened the fiber structure. This mechanism is confirmed by the decrease in the peak intensity of C–O cellulose by 28% in the FTIR analysis, as well as the appearance of fragmented interface layers in the SEM image.

These findings provide important guidance for future optimisation of flame retardant treatments, particularly in determining the right application parameters. Based on the observed strength degradation curve, the ideal immersion duration should not exceed 45 min to minimise the degradation effect on the mechanical strength of the fibers. Other alternatives worth considering include modifying the FR system to be nitrogen-phosphorus-based with a more neutral pH, or applying a coating method in place of immersion to limit the penetration of FR compounds into the core structure of the fiber. For practical applications, FR30 treatments have met the requirements of a wide range of industrial needs, from transport panels to construction materials, where the balance between mechanical properties and fire resistance is a crucial factor. The findings of Mebratie et al. on polymer composites reinforced by Ethiopian Highland Bamboo that the tensile strength of composite materials for panel applications reached 193.3 MPa [21] while the findings in this study are significantly stronger (245.81 ± 0.14 MPa). Similarly, the results of Hu et al.'s research found that bamboo fiber-based composite materials with the dissociation pretreatment method with a composite tensile strength reached 891 MPa [22] This allows synchronisation of the research that has been carried out to obtain the same or exceed the strength of panels in industries that require mechanical and thermal resistance. However, several aspects still need to be studied more deeply, including the material's resistance to long-term environmental exposure, both temperature and water degradation, its compatibility with various types of polymer matrices, and its potential to replace synthetic fibers such as glass and carbon. Further research is directed to explore the formulation of FR in BaPFeR, which is more stable while developing a more precise treatment protocol.

4 Conclusion

It was concluded that PaBFeR samples with treatment of variations in NaOH concentration (0%, 3%, 5%, 7%, and 11%) at variations in time duration (0 min, 45 min, 90 min and 135 min) were found to have optimal mechanical strength and adhesion values, namely in the 5% NaOH treatment sample for 45 min (NaOH5%45) showing the most optimal mechanical performance, with a tensile strength of 249.54 ± 0.11 MPa, a tensile strain of 3.35 ± 0.01%, and a shear stress of 14.47 ± 0.69 MPa without a fiber pulled out (0% pull-out) of its matrix. The value was validated by the significant difference in values from other samples with ANOVA values (P Value < 0.05) and the Tukey HSD comparison which showed differences in all sample groups during NaOH treatment. The FTIR results in this sample confirm that the presence of a functional group (–OH) in the range of 3600–3200 cm^{-1} which is indicated from lignin and hemicellulose and is evidenced by SEM results which show that the presence of lignin, hemicellulose, and a small crack area significantly affects the values of PaBFeR mechanical strength. In addition, it was also concluded that after being treated with FR inhibitors (30, 45, and 60 min) on the sample (NaOH5%45), it was found that the mechanical performance and adhesion of the matrix to PaBFeR decreased. The 30-min FR treatment showed the best performance with an increase in tensile strain up to 4.59 ± 0.23%, while the tensile strength of the fiber decreased slightly to 245.81 ± 0.14 MPa, and the shear strength to 12.82 ± 0.07 MPa. These results validated the significant difference in values from other samples with ANOVA (p value < 0.05) and the Tukey HSD comparison showing differences in all sample groups during FR inhibitor treatment. The FTIR results confirmed that the presence of the –OH function group at the wavenumber 3741.45 cm^{-1} indicated by lignin and hemicellulose and the presence of volatile compounds indicated by FR compounds and evidenced by SEM results which showed that the presence of lignin, hemicellulose, and crack areas increased from previous affected the values of mechanical strength and adhesion of PaBFeR. Thus, for applications that require a balance between optimal mechanical properties and fire resistance, NaOH5%45 + FR30 can be considered the best treatment condition, as it still retains good mechanical properties with minimal degradation compared to longer FR treatments.

Author contributions M.M. conducted the experiments, performed data analysis, and wrote the main manuscript text. Z.D. supervised the research and contributed to the manuscript structure and revisions. R.Z. provided methodological guidance and critical review of the manuscript. L.H.A. assisted with microstructure analysis and contributed to data interpretation. All authors reviewed and approved the final manuscript.

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Data availability All data generated or analysed during this study are included in this published article.

Plant Material: The plant material used in this study was mature culms of *Gigantochloa atter* (Hassk.) Kurz, originally described by Justus Carl Hasskarl in 1868. The bamboo, locally known as "Bambu Parring," was collected from Tanralili District, Maros Regency, South Sulawesi, Indonesia. Zulkifli Djafar and Massriyady Massaguni, experts in composite materials and natural fibers, identified the species. Although no formal voucher specimen was deposited, species identification was performed based on morphological characteristics and regional taxonomic references.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare no competing interests.

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