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Research Article

Adhesion Strength of Parring Bamboo (*Gigantochloa atter*) Fiber With Flame Retardant Polyester Resin: Wettability

Massriyady Massaguni,[1] **Zulkifli Djafar**,[2] **Rozli Zulkifli**,[3] **and Lukmanul Hakim Arma**[2]

[1]*Agro Industry Manufacturing Engineering Department, ATI Makassar Polytechnic, Makassar, Indonesia*
[2]*Mechanical Engineering Department, Engineering Faculty, Hasanuddin University, Gowa, Indonesia*
[3]*Mechanical and Materials Engineering Department, Engineering and Built Environment Faculty, The National University of Malaysia, Bangi, Selangor, Malaysia*

Correspondence should be addressed to Zulkifli Djafar; zulkiflidjafar3065@gmail.com

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This study explores the potential of parring bamboo fiber (PaBFeR) as a composite reinforcement material with flame retardant polyester (FR-P). This study addresses the wettability of PaBFeR by measuring the contact angle of FR-P resin on the surface of PaBFeR uses the sessile drop method in accordance with ASTM D7334-08 (2022), as many as seven drops per sample. The alkaline treatment at 85°C for the delignification of PaBFeR varied by 3%, 5%, and 7% and the washing varied for 45, 90, and 135 min to see the effect on the value of the formed contact angle. The results obtained are that the optimal wettability for application in FR-P matrix PaBFeR-reinforced composites is NaOH11%90 with a contact angle range value of $42^\circ \leq \theta \leq 48^\circ$ and average contact angle $44.86^\circ \pm 1.88^\circ$. In contrast, the NaOH3%45 sample was declared the least optimal configuration because it had an average contact angle value of $33.57^\circ \pm 7.44^\circ$ and a very wide range of contact angles ($21^\circ \leq \theta \leq 46^\circ$), indicating the instability of the results.

Keywords: adhesion strength; ASTM D7334-08; bamboo fiber; fire-retardant polymer; *Gigantochloa atter*; wettability

1. Introduction

Indonesia is rich in bamboo, with more than 100 species found in the country, making it the second largest producer of bamboo after China [1]. One of them is *Gigantochloa atter* or bamboo parring, which has been studied for its density, morphology, tensile strength, and modulus of elasticity [2]. In addition, this bamboo has anatomical features that are of commercial value, so it provides insight to further research on the characteristics of this species of bamboo [3].

Nowadays, bamboo fiber is widely used for composite materials such as in the raw materials of wall and floor panels which have improved mechanical resistance and currently have developed fire resistance properties for special applications in environments that range fire [4]. Some studies include the addition of glass fibers that significantly improve the flame retardant properties and postcombustible mechanical strength as well as water resistance when extinguished [5]. In addition, the discovery of the addition of euphorbia biocoagulant to bamboo fiber composites adds a reference to the addition of flame retardant strength to bamboo fiber composites [6]. Bamboo fiber composites are also widely applied in the fields of construction, automotive, and packaging because of their excellent physical properties [6].

One of the things that affects the strength of bamboo fiber–reinforced composites is the interface relationship between the matrix and its reinforcement [7]. Good interface relationships produce good interface adhesion properties as well, increasing mechanical strength, for example, bamboo fiber–reinforced polymer composites (BFRP) [8]. In addition to the chemical factors in fibers, hydrophilic/hydrophobic properties, and sensitivity to moisture, adhesion

properties are also influenced by the elements present in the binder or matrix [9–11], as is the case with coupling agents such as maleic anhydride polypropylene (MAPP) in a polypropylene (PP) matrix [8].

The adhesion properties between bamboo fibers and matrices are influenced by various factors, including the chemical composition and physical characteristics of the fibers. Key chemical properties include cellulose, lignin, and hemicellulose content, which play a role in determining the compatibility of fibers with the matrix. Physical characteristics, such as microfibril angle and density, also affect the bonding efficiency at the interface. Additionally, the fibers' hydrophilic or hydrophobic tendencies and their moisture sensitivity significantly influence the adhesion quality. These factors collectively determine the strength and durability of the interface in bamboo fiber–reinforced composites.

Several studies have found an increase in the mechanical strength of bamboo fiber composites, potentially suitable for use as reinforcements in refractory composites [12, 13]. The polymer matrix was tested to obtain mechanical strength, and a flame retardant was added to test the fire resistance of the composite material [14]. However, various flame retardant polymers have been discovered that consist of chemical compounds to slow the spread of fire, usually used in wire and cable components, construction, equipment, and others. Due to the diversity of flame retardant substances in flame retardant polyester (FR-P) products and each manufacturer limits the information on chemical compounds used for confidential reasons, so to understand the compatibility of the matrix and fiber, one of the properties that need to be known is the adhesion properties of the fiber and the matrix; one of the parameters is wettability or known wettability.

Fiber wettability is defined as the surface ability of a fiber to interact with fluids that meet properties such as bond strength and mechanical characterization of composites [15–18]. In general, several researchers have previously researched the wettability of bamboo fiber with the research concept toward the temperature effect, drying process, wetting properties, and moisture content shown in Figure 1.

Figure 1 illustrates the concepts of previous research that explain how wettability parameters in bamboo, such as temperature effect and drying process, can change the structure of bamboo fibers, thereby affecting the fiber–matrix interaction. Wetting behavior is studied using wettability tests, and contact angle measurements are often associated with bond strength testing and mechanical characterization to understand the extent to which fibers are able to integrate with the composite matrix. In addition, the moisture content can affect the fiber's ability to absorb the matrix liquid, contributing to the adhesion strength.

Factors affecting fiber wettability include fiber surface free energy (γSV), liquid–air interface stress (γLV), fiber–liquid interface stress (γSL), surface lignin, surface charge, structure, density, and treatment of fibers such as sodium hydroxide (NaOH) immersion [15–17] and other chemical substances. The wettability evaluation method used is usually the measurement of the contact angle. Modifying the wettability of the fiber surface can improve the performance of the composite such as the mechanical strength of the fiber with the matrix of transverse properties, the bending properties, the shear strength of the interlaminar, and *the pull-out properties* of the fiber with the matrix [19, 20].

The properties of natural fibers are generally divided into three types, namely, hydrophilic, hydrophobic, and superhydrophobic shown in Figure 2.

Natural fibers such as bamboo, ramie, or kenaf are known to have hydrophilic properties, which means they tend to absorb water and moisture from the environment [21–23]. This property poses a significant challenge in the composite industry, especially when these natural fibers are reinforced in hydrophobic resin matrices such as epoxy or polyester. The tendency of natural fibers to absorb water creates a chemical mismatch between the fibers and resins, which often results in a deterioration in the quality of the fiber–resin interface. As a result, manufacturing problems such as uneven fiber dispersion, weak bonding, and decreased mechanical strength of the composite can arise. To overcome this challenge, it is necessary to modify the surface of the fiber or use *coupling agents* or alkaline treatment using NaOH, which can improve the compatibility and interaction between the fiber and the resin, resulting in composites with better mechanical properties and performance.

Flame retardant polymers are materials designed to provide fire protection to consumer goods and reduce fire growth in various scenarios [24]. These materials are used in various applications, including thermoplastics, thermosets, textiles, coatings, and polyurethanes, and are organic and inorganic [25, 26]. The working mechanism of refractory materials includes improving the formation of charcoal, forming resistive materials, and improving the thermal resistance of materials. Fire-retardant polymers must also comply with fire safety standards related to parameters such as fire spread, heat release rate, smoke turbidity, and toxicity of degradation products [25].

The wettability properties of parring bamboo fiber (PaBFeR) to FR-P resin are currently unknown. However, previous similar studies examined the wettability of bamboo fiber against epoxy resin with polyacrylate-based wetting agent (BYK-358N) treatment, which was able to improve the interface relationship between the fiber and the matrix [27]. In addition, several other studies used chitosan-coated treatment on bamboo fibers by analyzing the wettability of polyvinylidene fluoride (PVDF), PP, and grafted MAPP [28]. In addition, the specific wettability of PaBFeR with NaOH concentration and temperature treatment to FR-P is also unknown. Other studies, such as the alkaline treatment of bamboo fibers with low NaOH concentrations of 6%–10%, improved wettability, as shown by increased surface roughness and contact angles [29], and provided thermal stability to bamboo fibers [30].

Therefore, the purpose of this study is to analyze the wettability properties of PaBFeR and FR-P under the treatment of various NaOH concentrations to obtain the compatibility values between the matrix and the fibers in the application of PaBFeR-reinforced flame retardant composites. This study introduces the results of a new study on the compatibility of PaBFeR and FR-P resins. By understanding *the wettability*

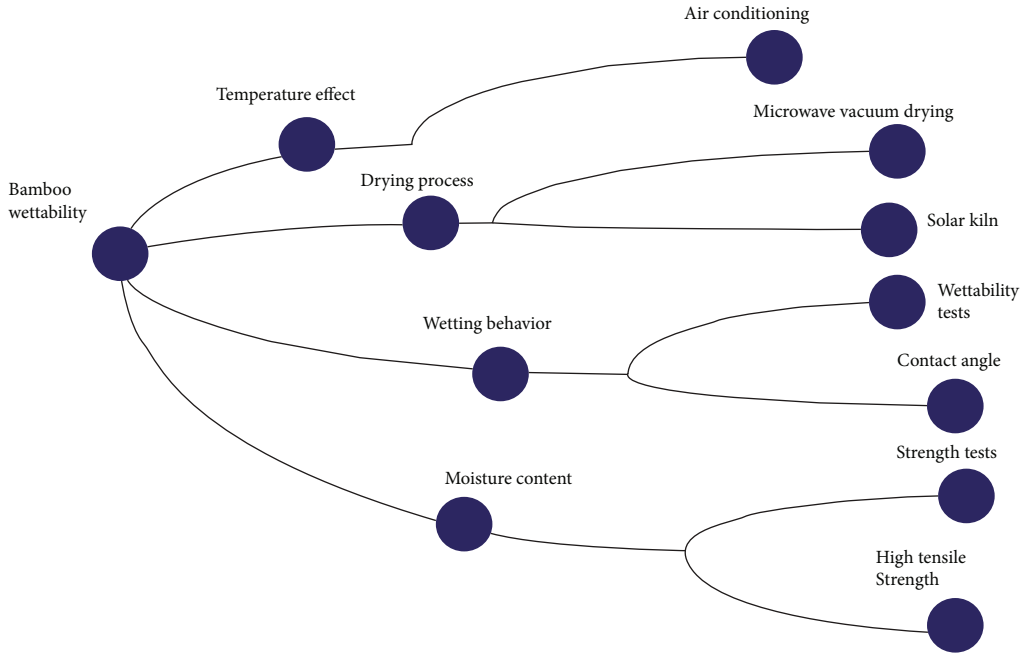

Figure 1: Wettability research concept in bamboo.

characteristics under certain treatments, this research contributes to developing good-performing refractory composite materials.

2. Materials and Methods

2.1. Materials. The fiber material used in this study is PaBFeR obtained from the cultivation of parring bamboo (*Gigantochloa atter*) or PaB in Maros Regency, South Sulawesi Province, Indonesia. The PaB used in this study was 3 years old; the stem segment was taken above 100 cm from the ground level along 120 cm. The PaB material used is shown in Figure 3.

The resin material used in this study is a liquid FR-P resin, with the brand Merryhill FR-P supplied by Alensy Wooden, and the Super Catalyst Butanox M50. This resin consists of an unsaturated polyester base and additives added to improve fire resistance. The resin:catalyst ratio is 100:1 with a volume of 100 mL each time the test material is manufactured. The FR-P resin and catalyst used are shown in Figure 4.

The tools used in this study were wooden saws, bamboo splitters, bamboo slicing machines, water heaters, stainless steel containers, bamboo extraction machine rolling type, droppers, and wettability instrument test (macro camera, magnifying glass, and fiber stretch support).

2.2. Preparation of Fibers. The PaB cutting follows the specified dimensions, which is 120 cm using a saw, and then divided by 3 so that the dimensions of the PaB material obtained are 40 cm. Next, the PaB is split using a bamboo splitting tool, as shown in Figure 5; after which it is formed into PaB strips using a slicing machine, as shown in Figure 6. The obtained bamboo strips are $40 \times 20 \times 2$ mm. Before the extraction process begins on the bamboo fiber extraction machine, as shown in Figure 7, the PaB strips are washed for delignification using NaOH solutions with concentrations of 3%, 5%, 7%, and 11%. The washing durations are 45, 90, and 135 min, respectively, as shown in Figure 8. Additionally, a set of strips is left untreated as a control.

2.3. Alkaline Treatment. In this study, the alkaline treatment was conducted to reduce lignin, which acts as an adhesive between cellulose in PaB. This process, known as delignification, utilized NaOH solutions with varying concentrations and soaking durations at a controlled temperature of 85°C.

2.4. Contact Angle Measurement. In this study, the measurement of the contact angle was used in the sessile drop method in accordance with ASTM D7334-08 (2022), as illustrated in Figure 9.

A single fiber of PaBFeR, stretched to 70 cm in length, was used for each experimental condition. Contact angle measurements were taken from seven (7) droplets of FR-P resin applied using a drop pipette onto the single PaBFeR fiber. Each droplet was measured individually after being left to settle for 2 min, using a macro camera with 40× magnification. The captured images were then analyzed using ImageJ software to calculate the contact angle. Data were collected based on the minimum (x) and maximum (y) contact angle ranges ($x^\circ \leq \theta \leq y^\circ$) observed in seven droplets on each PaBFeR. The optimal value approach was carried out based on averages (AVG ± STDEV), and the results were compared with the ASTM D7334-2022 standard.

3. Result and Discussion

The measurement of the contact angle is obtained as shown in Table 1.

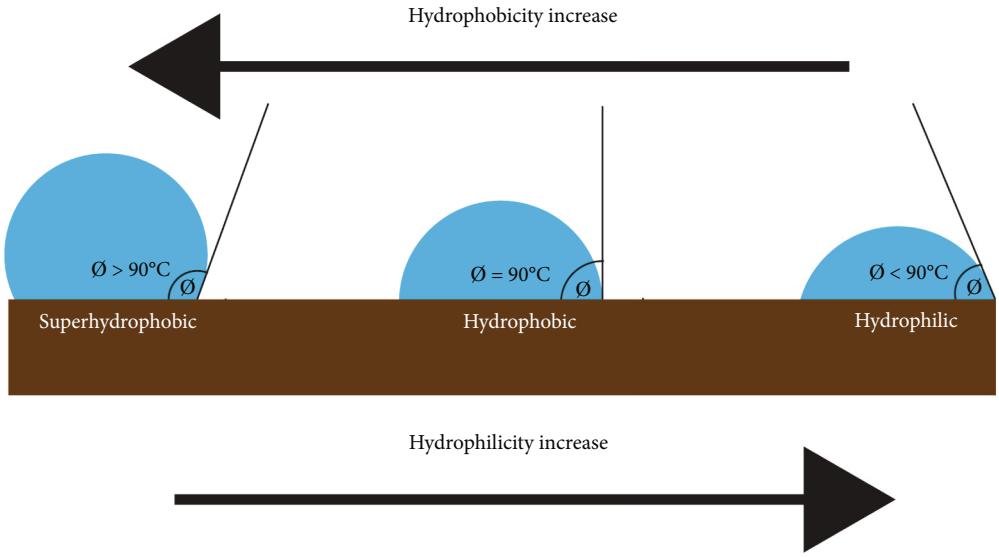

Figure 2: Wettability on the fiber surface.

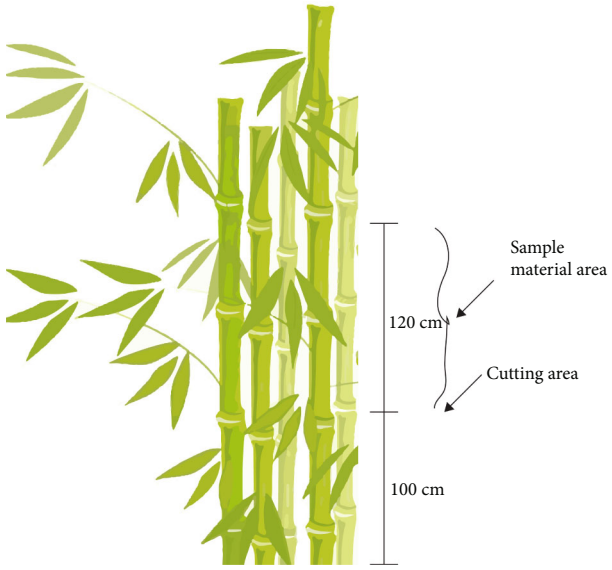

Figure 3: PaB cutting area and sample material area.



Figure 4: Flame retardant polyester (FR. P) Merryhill brand and Super Catalyst Butanox M-50.

The contact angle measurement method ensures precision by analyzing seven droplets per experimental condition. The range of values (e.g., 32°–41° for the control sample) reflects the variability observed across all seven measurements. For each condition, the minimum and maximum contact angles were extracted directly from the individual droplet measurements. This variability highlights the influence of fiber surface treatment on resin wettability.

The control/normal sample has an average contact angle of 36.71° ± 2.71°, with a range of 32°–41°. For samples with a 3% NaOH solution for 45 min, the average contact angle was 33.57° ± 7.44°, with a range of 21°–46°. A 90-min 3% NaOH sample showed an average contact angle of 36.29° ± 4.8°, with a range of 26°–46°, while for a time of 135 min, the average value was 35.57° ± 2.77° with a range of 31°–41°. At a concentration of 5% NaOH for 45 min, the average contact angle was 37.86° ± 2.36°, with a range of 33°–40°, which increased to 43.71° ± 4.46°, with a range of 39°–50°, at 90 min, and decreased slightly to 40.86° ± 4.58°, with a range of 34°–47° at 135 min. In the treatment with a NaOH concentration of 7%, the sample with a time of 45 min had an average contact angle of 37.43° ± 3.06° with a range of 33°–43°, while at 90 min the average value was 37.86° ± 2.36° with a range of 34°–41°, and at 135 min, it decreased to 33° ± 3.30° with a range of 29°–39°. In the treatment with a NaOH concentration of 11%, the average contact angle at 45 min was 39° ± 2.51° with a range of 35°–42°, while at 90 min, it



(a) (b)

Figure 5: (a) The process of splitting bamboo using a bamboo splitting tool. (b) Output of bamboo splitting products.



(a) (b)

Figure 6: (a) Bamboo slicing machine. (b) Bamboo strip $40 \times 20 \times 2$ mm.

increased to $44.86^{\circ} \pm 1.88^{\circ}$ with a range of 42°–48°, and at 135 min, it was $39.57^{\circ} \pm 5.15^{\circ}$ with a range of 32°–48°.

A comparison of the range of contact angle values for each PaBFeR sample based on the variation in NaOH concentration is shown in Figure 10.

Figure 10a shows 45 min, an increase in NaOH concentration showing a relatively small change in the contact angle, with all treatments remaining below 45° except for a 3% NaOH sample. The 3% NaOH sample was significantly below 45° and had the least contact angle, but there was a point on the PaBFeR with a contact angle value exceeding 45°. This shows that PaBFeR fibers still have good wettability in a short time. In contrast, NaOH treatment has not significantly influenced the surface properties of the fibers that have not reacted optimally to the fiber surface. When the duration is extended to 90 min (Figure 10b), the variation in the contact angle becomes more significant. NaOH of 5% causes the contact angle to exceed 45°, signaling a decrease in wettability, while NaOH of 11% is close to that limit. This suggests that the longer the duration of the treatment, the more likely it is that chemical changes on the surface of the fibers can affect the hydrophobic properties of the fibers. At a duration of 135 min (Figure 10c), this pattern continued with 7% NaOH showing a decrease in wettability (contact angle lower than 35°), while NaOH 11% resulted in a contact angle reaching 48°, indicating a more significant decrease in wettability.

In general, the effect of duration and concentration of natural fiber washing with NaOH significantly affects the wettability of natural fibers [31–34] as well as bamboo fibers



(a) (b)

Figure 7: (a) Bamboo fiber extraction machine. (b) PaBFeR bamboo fiber output.



Figure 8: PaB delignification process.

[33, 35, 36] and specifically in the PaBFeR that has been studied. The longer duration and concentration of NaOH leaching in PaBFeR affect the morphological properties of the fiber surface. A comparison of the contact angle values of each PaBFeR sample based on the variation in wash duration is shown in Figure 11. Figure 11a shows a NaOH concentration of 3%, and the contact angle (θ) shows a relatively stable range of values. At 45 min, θ ranges from 21° to 46°; however, there is a decrease seen at 90 min with θ between 26° and 40°. This suggests that increasing the wash time at low concentrations does not significantly improve wettability and that, over a longer period, the chemical reaction occurs less efficiently or there is damage to the fibers.

In Figure 11b, a concentration of 5% shows a more consistent increase in wettability with a wider range of θ. At 45 min, θ ranges from 33° to 40°, while at 90 min, θ increases to 39°–50°. This indicates that a concentration of 5% NaOH is quite effective in improving wettability, with a longer wash time having a greater effect in lowering the contact angle. However, at 135 min, θ again decreased from 34° to 47°, indicating the limit of the effectiveness of this concentration or the potential for degradation of fiber quality due to overtreatment.

Figure 11c, which is a concentration of 7%, shows the most significant change in wettability. At 45 min, θ is in the range of 33°–43°, and at 90 min, θ decreases slightly to 34°–41°. However, at 135 min, θ reached the lowest range of 29°–39°, suggesting that this concentration optimally modifies the surface of the fibers to achieve the best wettability but also has the potential to damage the fibers if it takes too long.

Figure 11d shows an 11% concentration, and there is a clear increase in wettability. At 45 min, θ ranges from 35° to 42°, and at 90 min, θ increases further to 42°–48°. At 135 min, the θ range becomes the widest, between 32° and 48°. This suggests that high concentrations of NaOH can significantly improve the wettability of PaBFeR fibers, although there may be a risk of damage to the fibers if the wash time is too long.

According to the ASTM D7334-08 (2022) standard, the contact angle value below 45° indicates a good wettability value. This wettability value is assessed based on measurements at several different points on the fiber surface to overcome local variations (anisotropic). Therefore, it is very important to look at the minimum and maximum contact angle ranges. Furthermore, the optimal sample is assessed based on the average value and significance of the contact angle of the fiber sample.

This study shows that the wetting properties according to the ASTM D7334-08 (2022) standard in PaBFeR after applying NaOH treatment to the composite show that the average value of the contact angle looks appropriate. In addition, some samples showed values that were likely to change from normal samples. It can be seen that NaOH immersion,

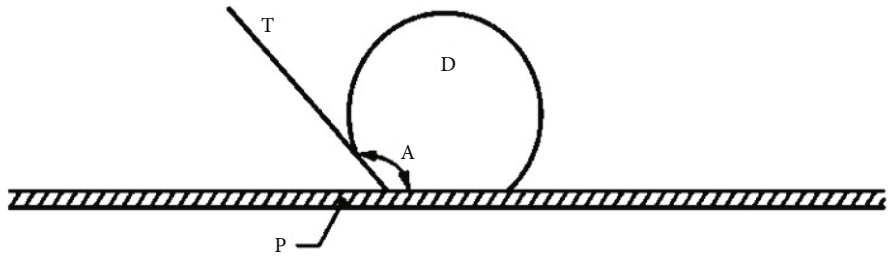

Figure 9: ASTM D7334-2022 contact angle measurement.

TABLE 1: PaBFeR contact angle measurement results.

No.	Sample code	Average contact angle (θ°)	Range contact angle (θ°)
1	N	$36.71^{\circ} \pm 2.71^{\circ}$	$32^{\circ} \leq \theta \leq 41^{\circ}$
2	NaOH3%45	$33.57^{\circ} \pm 7.44^{\circ}$	$21^{\circ} \leq \theta \leq 46^{\circ}$
3	NaOH3%90	$36.29^{\circ} \pm 4.8^{\circ}$	$26^{\circ} \leq \theta \leq 46^{\circ}$
4	NaOH3%135	$35.57^{\circ} \pm 2.77^{\circ}$	$31^{\circ} \leq \theta \leq 41^{\circ}$
5	NaOH5%45	$37.86^{\circ} \pm 2.36^{\circ}$	$33^{\circ} \leq \theta \leq 40^{\circ}$
6	NaOH5%90	$43.71^{\circ} \pm 4.46^{\circ}$	$39^{\circ} \leq \theta \leq 50^{\circ}$
7	NaOH5%135	$40.86^{\circ} \pm 4.58^{\circ}$	$34^{\circ} \leq \theta \leq 47^{\circ}$
8	NaOH7%45	$37.43^{\circ} \pm 3.06^{\circ}$	$33^{\circ} \leq \theta \leq 43^{\circ}$
9	NaOH7%90	$37.86^{\circ} \pm 2.36^{\circ}$	$34^{\circ} \leq \theta \leq 41^{\circ}$
10	NaOH7%135	$33^{\circ} \pm 3.30^{\circ}$	$29^{\circ} \leq \theta \leq 39^{\circ}$
11	NaOH11%45	$39^{\circ} \pm 2.51^{\circ}$	$35^{\circ} \leq \theta \leq 42^{\circ}$
12	NaOH11%90	$44.86^{\circ} \pm 1.88^{\circ}$	$42^{\circ} \leq \theta \leq 48^{\circ}$
13	NaOH11%135	$39.57^{\circ} \pm 5.15^{\circ}$	$32^{\circ} \leq \theta \leq 48^{\circ}$

both concentration variations and immersion duration, affects the wettability value of PaBFeR.

Although the NaOH7%135 sample had the lowest average contact angle of 33°, it was not selected as the most optimal. Based on the ASTM D7334-08 (2022) standard, contact angle values below 45° do indicate good wettability, but optimal wettability is not only determined by the average low contact angle value. The consistency and stability of the measurement results are also very important factors. The 7% 135-min NaOH sample had a standard deviation of ±3.30° and a fairly wide range of contact angles ($29^{\circ} \leq \theta \leq 39^{\circ}$), which showed a greater variation in results compared to other samples. In practical applications, especially for composite materials, wettability consistency is essential to ensure uniform adhesion of fibers and resin matrices. In addition, the mean decrease in the contact angle in the NaOH sample was 7% 135 min compared to the control sample of only 3.71°, with a higher standard deviation compared to the control (±3.30° vs. ±2.71°). This suggests that this treatment does not provide a significant increase in wettability in practice.

In contrast, the NaOH11%90 sample showed an average contact angle of 44.86°, which is close to the 45° limit with the lowest standard deviation (±1.88°) and a narrow range of contact angles ($42^{\circ} \leq \theta \leq 48^{\circ}$). These results indicate that the treatment provides more stable and consistent results across the fiber surface. In addition, a suboptimal sample of NaOH3%45 due to a much larger standard deviation value (±7.44) indicates a higher level of yield instability and a very wide range of contact angle values (21°–46°), which may indicate less consistent treatment. These results allow for further research on the microstructure of PaBFeR because the sample results are not optimal due to interfering factors in the PaBFeR fibers.

Treatment with NaOH at low concentrations does increase fiber wetting, while high concentrations tend to decrease wetting. This suggests that higher concentrations of NaOH may be too harsh and lead to a decrease in fiber surface quality, ultimately decreasing wettability [35, 37].

Heat treatment of longer soaking (similar to heat treatment) can help optimize wettability under certain conditions, but it can also decrease wettability when combined with too high concentrations of NaOH, in line with the reference that heat treatment can modify the chemical composition of fibers [38].

Effective surface modification with NaOH at low to moderate concentrations improves wettability and interaction with resins, according to references stating that surface modifications can improve wettability and resistance to water damage [39].

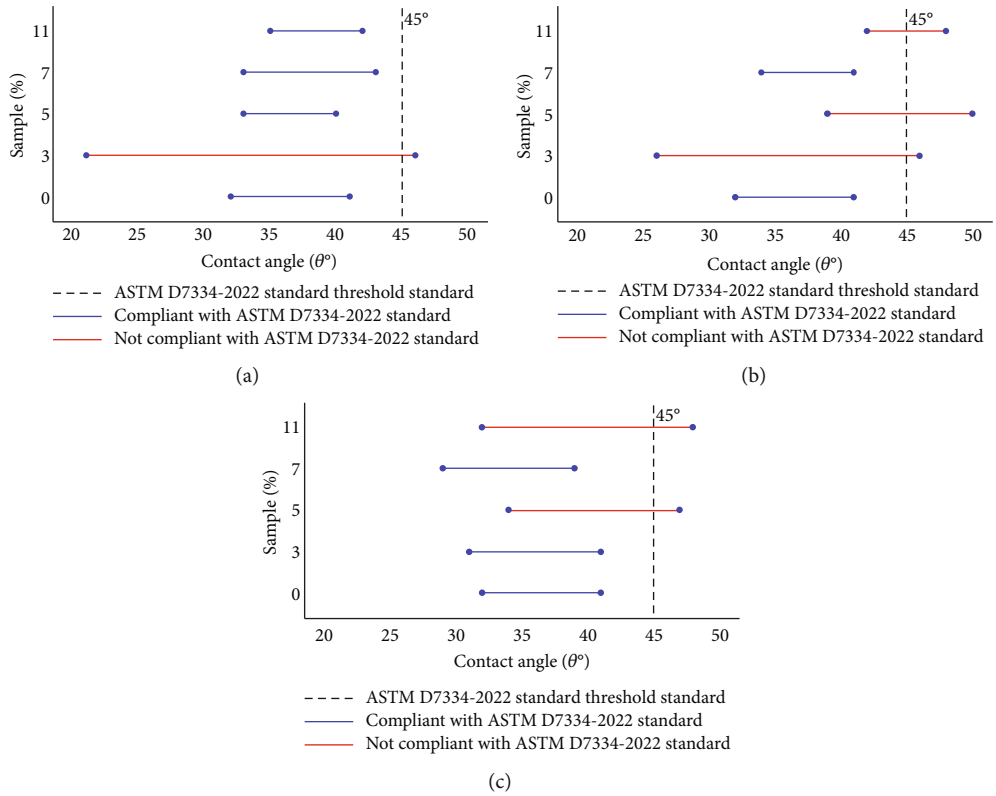

Figure 10: Range of contact angle PaBFeR variations in NaOH concentration for (a) 45 min, (b) 90 min, and (c) 135 min.

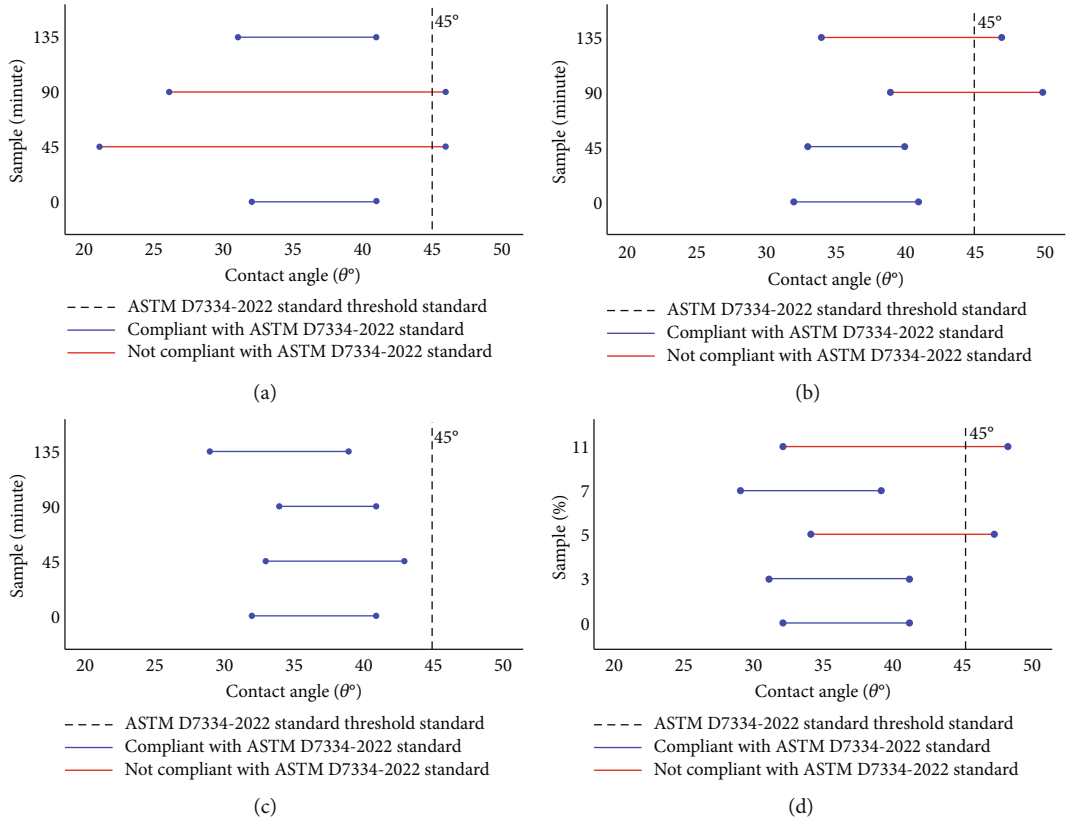

Figure 11: Range of contact angle PaBFeR variations in different times for (a) 3% NaOH, (b) 5% NaOH, (c) 7% NaOH, and (d) 11% NaOH.

PaBFeR fibers have good potential for use with FR-P resins in composite applications, especially when equipped with surface treatments such as alkali [40, 41]. Proper adjustments and modifications can minimize negative impacts and result in composites with good performance in terms of fire resistance. Further research is needed to optimize the formulation and test the performance of the composite under actual application conditions [42].

4. Conclusion

It was concluded that the PaBFeR NaOH11%90 sample was chosen as the optimal configuration because it had the highest average contact angle value of 44.86°, close to the maximum limit of good wettability, with a low standard deviation (±1.88) reflecting the consistency of the measurement, as well as a relatively narrow range of contact angle values ($42^\circ \leq \theta \leq 48^\circ$) which showed stable results. In contrast, the NaOH3%45 sample was declared the least optimal configuration because it had an average value of 33.57° contact angle with the highest standard deviation (±7.44), indicating large variation between measurements and a very wide range of contact angles ($21^\circ \leq \theta \leq 46^\circ$) indicating instability of the results.

Data Availability Statement

Data is available on request.

Conflicts of Interest

The authors declare no conflicts of interest.

Author Contributions

Massriyady Massaguni: formal analysis, investigation, methodology, conceptualization, writing, and editing. Zulkifli Djafar: supervision, methodology, conceptualization, project administrator, data curation, writing, and editing. Rozli Zulkifli: supervision, data curation, methodology, and conceptualization. Lukmanul Hakim Arma: supervision, data curation, methodology, and conceptualization.

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