

Effect of alkaline treatment on mechanical, thermal, and biodegradation properties of bamboo filler-reinforced corn starch films

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ABSTRACT

Although starch-based bioplastics are becoming more popular as environmentally friendly substitutes for petroleum-based plastics, their limited mechanical strength and thermal stability restrict their usefulness. To examine the effects of bamboo filler loading and chemical treatment on mechanical, thermal, and biodegradation properties, corn starch films reinforced with untreated and alkaline-treated bamboo filler were prepared by solution casting. To improve bamboo filler-corn starch matrix adhesion and eliminate surface impurities, bamboo filler was treated with 5 wt% sodium hydroxide. Tensile testing (ASTM D882), thermogravimetric analysis (TGA), and biodegradability tests in freshwater and saltwater environments were used to characterize composite films with 2–10 wt% bamboo filler. The findings demonstrated that, while retaining a degree of flexibility, NaOH-treated bamboo filler increased tensile strength, with a maximum increase of approximately 52.8% when compared to untreated bamboo filler composites. All films had similar degradation onset temperatures ($\sim 300^{\circ}\text{C}$), according to thermal analysis; treated bamboo filler composites showed a shallower DTG peak, indicating better thermal uniformity. In freshwater, the immersion tests showed increased visible microbial colonization, whereas no discernible microbial growth was observed in simulated seawater. Overall, alkaline treatment successfully improved the mechanical and thermal performance of starch-based composites without sacrificing biodegradability, underscoring their potential as eco-friendly packaging materials.

Keywords: Alkaline Treatment, Biocomposites, Biodegradability, Mechanical Properties, Thermal Stability

1. INTRODUCTION

Because of the persistence and toxicity of microplastics, the growing amount of plastic waste in aquatic and marine environments has caused significant environmental and ecological concerns [1-2]. The urgent need for biodegradable alternatives is highlighted by the fact that conventional petroleum-based plastics resist natural degradation and contribute to long-term environmental contamination [3]. Because of their affordability, biodegradability, and renewability, starch-based bioplastics have become attractive options for disposable and short-lived applications [4-5].

Because corn starch is readily available and has film-forming ability, it is frequently used in the production of biodegradable films. However, starch-based films have intrinsic disadvantages that limit their usefulness, such as low mechanical strength, poor thermal stability, and high moisture sensitivity [6-7]. In order to increase flexibility, plasticizers such as glycerol are frequently added, but this can further reduce in stiffness and tensile strength [5]. Natural fiber reinforcement has been thoroughly investigated as a way to address these limitations.

A popular method for improving the mechanical performance of starch-based bioplastics while preserving biodegradability is reinforcement with natural fibers [8-9]. Because of their high cellulose content, low density, and renewability, agricultural and plant-based fibers like kenaf, bamboo, palm, and sugarcane bagasse have been studied as reinforcing fillers [10-11]. Several studies indicate that, if sufficient fiber dispersion and interfacial bonding are achieved, low-to-moderate natural fiber loadings can enhance tensile strength and stiffness in starch-based composites [7, 12]. However, because of their poor compatibility with the starch matrix, untreated natural fibers frequently result in inconsistent or diminished mechanical properties [13]. Ineffective stress transfer is caused by the presence of lignin, hemicellulose, waxes, and other surface impurities, which restrict hydrogen bonding and encourage fiber agglomeration [14].

Bamboo is known as the fastest-growing plant on Earth. Certain species of bamboo can grow up to 90 cm in a single day [15]. For this reason, bamboo is a renewable and sustainable resource for various applications such as construction, textiles, and daily goods such as furniture, chopsticks, and cutting boards [15]. Traditionally, bamboo

shoots are consumed locally in Malaysia [16]. Their discarded shells create a disposal issue in the local communities [17]. Therefore, bamboo shoot shell was investigated as a filler for corn starch-based films in this study. Before it was included in the corn starch-based films, bamboo shoot shell was first mechanically ground. However, lignin, hemicellulose, waxes, and oils are surface contaminants found in untreated bamboo filler that prevent interfacial adhesion with hydrophilic starch matrices. Reduced mechanical performance and ineffective stress transfer are the outcomes of poor bamboo filler-corn starch matrix bonding. Alkaline (NaOH) treatment is one of the best ways to improve the compatibility between natural fibers and matrices. Alkaline treatment improves mechanical interlocking and interfacial adhesion by removing non-cellulosic components, increasing fiber surface roughness, and exposing cellulose hydroxyl groups [18-19].

When added to polymer matrices, NaOH-treated fibers show enhanced tensile strength, modulus, and thermal behavior, according to numerous studies [20-21]. Similar gains have been documented for bamboo fibers, where load-bearing efficiency and fiber crystallinity are improved by alkaline treatment [22-23]. The need for optimal treatment parameters is highlighted by the fact that excessive fiber loading or overly harsh treatment conditions may deteriorate cellulose structure and impair composite performance [24-25].

When assessing the processing and application potential of starch-based bioplastics, thermal stability is a crucial consideration. Starch-based materials usually show significant thermal decomposition between 270°C and 350°C [26]. Thermogravimetric analysis (TGA) is frequently used to evaluate degradation behavior. Due to the presence of thermally unstable components like hemicellulose and lignin, the addition of untreated natural fibers frequently results in heterogeneous degradation behavior [27]. Alkaline-treated fibers, on the other hand, typically improve thermal uniformity, leading to more predictable degradation profiles and narrower DTG peaks [28-29]. Chemical treatment improves thermal coherence and decreases residual char formation, even though fiber reinforcement rarely raises the degradation onset temperature noticeably [11].

Because of the growing amount of plastic pollution in freshwater and marine environments, biodegradability studies under aquatic conditions are crucial. One distinguishing feature of starch-based bioplastics is their biodegradability. However, environmental factors have a significant impact on the degradation behavior. According to studies, microbial activity and enzymatic hydrolysis cause starch-based materials to easily break down in freshwater settings [3]. By improving water absorption and surface roughness, natural fibers can hasten degradation even more by promoting microbial attachment [30]. In contrast, osmotic stress and decreased microbial efficiency in marine and saline environments frequently prevent biodegradation [1]. The significance of assessing material performance under various aquatic environments is highlighted by earlier research that found delayed or

suppressed degradation of biodegradable polymers in seawater as opposed to freshwater conditions [2].

The combined effects of bamboo filler loading and alkaline treatment on mechanical, thermal, and biodegradation behavior under freshwater and seawater conditions have received limited attention, despite the fact that natural fiber-reinforced starch bioplastics have been the subject of much research. Moreover, despite their high cellulose content and availability as agricultural waste, bamboo shoot shell filler is still underutilized. Thus, the purpose of this study is to examine the effect of bamboo filler loading and alkaline treatment on the mechanical, thermal, and biodegradation characteristics of corn starch-based films. To evaluate their suitability as sustainable substitutes for traditional plastics, the performance of untreated and NaOH-treated bamboo filler composites is compared.

2. MATERIALS AND METHODS

2.1. Materials

The food-grade corn starch (Cap Bintang, THC Sdn. Bhd., Malaysia) was selected as the polymer matrix. To increase compatibility within the starch matrix, glycerol (HmbG) was used as a plasticizer and acetic acid (HmbG) was utilized as a co-plasticizer. The natural filler was made from bamboo shoot shells that were gathered from Kampung Baru Pauh Setia, Arau, Perlis, Malaysia. Sodium hydroxide pellets (HmbG) were used for alkaline treatment. To prepare simulated seawater for biodegradation testing, aquarium-grade sodium chloride (Ki-Ki, Kwaquatic Supplies Sdn. Bhd., Malaysia) was utilized.

2.2. Preparation of Bamboo Filler

The fresh bamboo shoot shells were collected from the local bamboo shoot collectors. The surfaces of the bamboo shoot shells were cleaned thoroughly to remove impurities. The cleaned bamboo shoot shells were subsequently dried under the sun. Next, the bamboo shoot shells were subjected to oven drying for 5 h at 80°C to ensure the residual moisture was removed. Finally, the dried bamboo shoot shells were mechanically ground and sieved through a 100-mesh sieve to obtain bamboo shoot shell filler (BSSF).

The alkaline treatment was conducted using 5 wt% sodium hydroxide (NaOH) solution. The cleaned bamboo shoot shells were submerged in the solution for 2 h at room temperature. Following treatment, the bamboo shoot shells were mildly neutralized with diluted acetic acid after being repeatedly rinsed with distilled water until the pH was nearly neutral. The treated bamboo shoot shells were dried under the sun for one day followed by drying in an oven for 5 h at 80°C. Finally, the treated bamboo shoot shells were ground and sieved through a 100-mesh sieve.

2.3. Preparation of Corn Starch-Based Composite Films

The solution casting method was used to prepare corn starch films. Corn starch (10 g) was dissolved in 185 mL of distilled water to create a starch slurry. Next, 5 mL of glycerol and 2 mL of a 5% acetic acid solution were added.

The mixture was continuously stirred and heated to approximately 85°C until it completely gelatinized and formed a homogeneous solution.

Bamboo shoot shell filler (BSSF) was added to the starch solution at filler loadings of 2, 4, 6, 8, and 10 weight percent (wt%) in relation to the starch content. The final mixtures were cast into Petri dishes after being agitated to guarantee even bamboo filler dispersion. The films were dried for at least 24 hours at room temperature with forced air circulation. A similar procedure was repeated by replacing the untreated filler with alkali-treated filler. For comparison, a control film without filler was also prepared.

2.4. Mechanical Testing

A universal testing machine (Instron 5569) was used to measure tensile properties, such as tensile strength, modulus of elasticity, and elongation at break, in compliance with ASTM D882. Film samples were cut into 80 mm x 15 mm rectangular strips. A crosshead speed of 10 mm/min was used for the tests. Each specimen's gripping areas were covered with double-sided adhesive tape to reduce slippage during testing. Five specimens were tested for each formulation, and the average results are presented.

2.5. Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was used to assess the films' thermal stability (PerkinElmer TGA 8000). In a nitrogen atmosphere, small samples of the control film and selected composite films with better mechanical performance were heated from 30°C to 600°C at a rate of 10°C/min. The following parameters were recorded: residual mass, maximum degradation temperature (T_{max}), and onset degradation temperature (T_{onset}).

2.6. Biodegradability Assessment

Immersion tests in aquatic environments were used to assess the biodegradability of the corn starch films. A 3.5 wt% sodium chloride solution was used to create simulated seawater, while distilled water was used to simulate freshwater conditions. After being cut into 20 mm x 20 mm samples, the film specimens were submerged in the appropriate media at room temperature. Visual observations of the degradation behavior were made every five days for a duration of 60 days. To evaluate the progression of degradation, changes in surface morphology, discoloration, and microbial colonization were photographically recorded.

3. RESULTS AND DISCUSSION

3.1. Mechanical Properties

Figure 1 shows a comparison of the mechanical characteristics of corn starch films filled with untreated bamboo filler at various loadings (2–10 wt%) with the unfilled control film. Because of its continuous starch matrix and efficient plasticization, the control film had the highest tensile strength (6.5 MPa). The tensile strength of untreated

bamboo filler composites, however, was lower and peaked at 4.1 MPa at 6 wt% before declining at higher loadings.

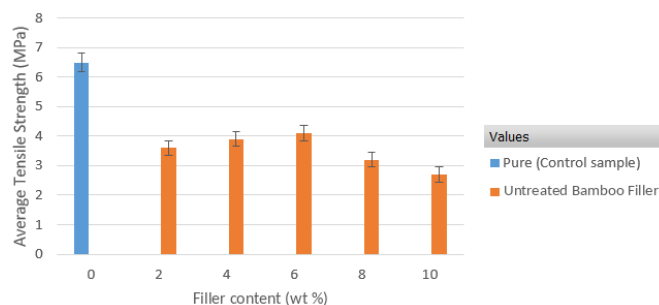


Figure 1. Tensile strength for unfilled and bamboo filler-filled composites

Poor interfacial adhesion between untreated bamboo filler and the starch matrix primarily caused the decrease in tensile strength. Waxes, hemicellulose, and residual lignin prevent hydrogen bonding and encourage bamboo filler agglomeration, which leads to ineffective stress transfer and early failure. As a low-aspect-ratio particulate filler rather than a continuous fiber, the ground bamboo shell is also less able to transfer stress efficiently, a limitation widely reported for particulate natural fillers regardless of matrix type [31]. Biopolymer composites reinforced with untreated natural fibers have shown similar patterns [13–14].

Figure 2 shows that at a low untreated bamboo filler loading, elongation at break increased from 36.18% for the control film to 42.88% at 2 wt%, indicating increased chain mobility as a result of weak bamboo filler–corn starch matrix interaction. However, elongation dropped as bamboo filler content increased, reaching 28.88% at 10 wt%, indicating embrittlement brought on by agglomeration and microstructural flaws.

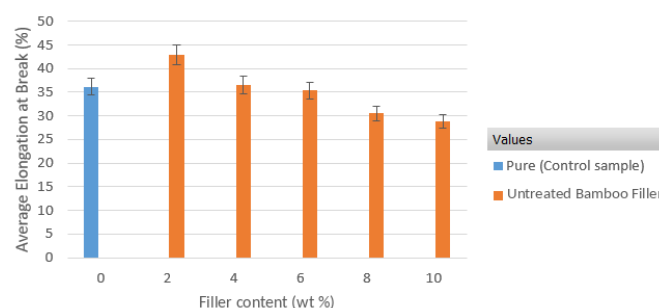


Figure 2. Elongation at break for unfilled and bamboo filler-filled composites

The modulus of elasticity in Figure 3 showed a similar pattern to tensile strength. Untreated bamboo filler did not effectively provide reinforcement, as evidenced by the control film's highest stiffness (89.66 MPa) and the untreated bamboo filler composites' noticeably lower modulus values (33.94–40.60 MPa). This is consistent with the same aspect-ratio limitation, since particulate fillers below their critical aspect ratio act mainly as fillers rather than reinforcement and typically lower rather than raise composite stiffness [31].

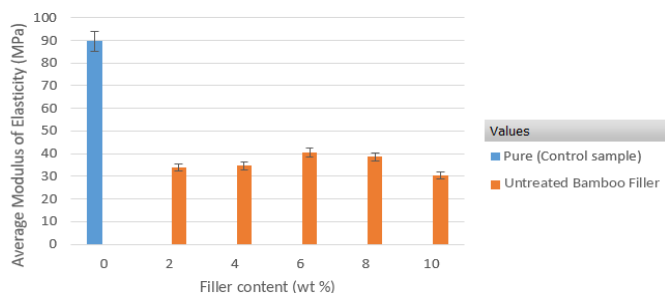


Figure 3. Modulus of elasticity for unfilled and bamboo filler-filled composites

3.2. Effect of Alkaline Treatment

Figure 4 shows that the mechanical performance of bamboo filler-reinforced starch films was enhanced by alkaline treatment with 5 wt% NaOH. Across all bamboo filler loadings, treated bamboo filler composites showed greater or comparable tensile strength than their untreated counterparts, with the two essentially tied at 4 wt%. Tensile strength increased by about 52.8% at 2 wt% bamboo filler loading, which was the largest improvement.

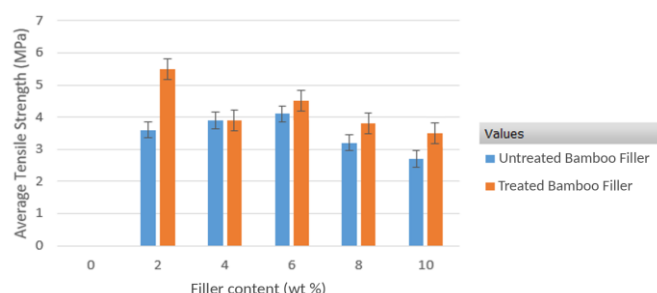


Figure 4. Tensile strength for untreated and treated bamboo filler-filled composites

The improvement is ascribed to the elimination of surface contaminants and greater cellulose hydroxyl group exposure, which enhanced stress transfer and bamboo filler-matrix adhesion. Figure 5 shows that the treated composites' elongation at break was more uniform and lower than that of the untreated composites, indicating decreased ductility as a result of stronger interfacial bonding. This trend is reported for most natural fiber composites, even though individual bamboo fibers themselves become more ductile after alkali treatment, suggesting that for a particulate filler the surrounding matrix constraint outweighs the filler's own change in ductility [32].

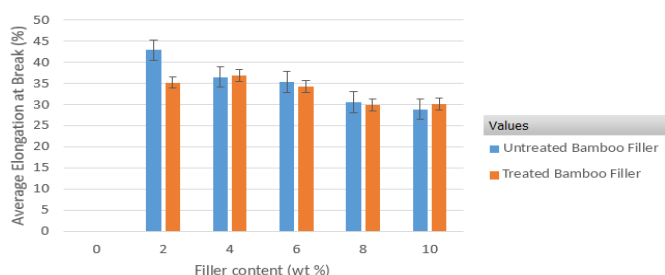


Figure 5. Elongation at break for untreated and treated bamboo filler-filled composites

Figure 6 shows that the treated composites' modulus of elasticity rose at low bamboo filler loading to 52.02 MPa at 2 wt%, but fell below the untreated composites at 4–8 wt% before recovering at 10 wt%. Similar decreases in composite modulus after alkali treatment have been reported for bamboo fiber composites, where the conversion of cellulose I to cellulose II during treatment is thought to lower the intrinsic stiffness of the fiber even as interfacial bonding improves [32–34]. Even at its highest, the treated modulus remained below that of the unfilled control (89.66 MPa), indicating that treatment narrows rather than reverses the stiffness limitation imposed by the filler's low aspect ratio.

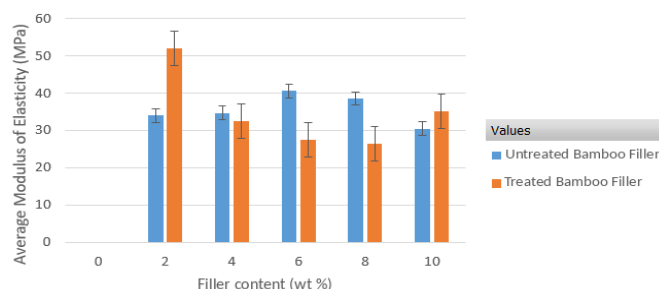


Figure 6. Modulus of elasticity for untreated and treated bamboo filler-filled composites

3.3. Thermal Stability

According to thermogravimetric analysis, all films experienced primary degradation between roughly 270 and 385 °C, with the onset of degradation occurring around 300 °C. Alkaline treatment and the addition of 2 wt% bamboo filler did not significantly change the starch matrix's degradation onset temperature.

Figure 7 shows that the untreated bamboo filler composite's peak degradation rate (−10.5%/min at 324°C) was similar to the unfilled control (−10.2%/min at 322°C), with the degradation window about the same width, indicating that untreated filler did not substantially change how the starch matrix decomposed. The treated composite in Figure 8 showed a distinctly shallower peak (−9.0%/min, still at 324°C), suggesting that alkaline treatment lowers the intensity of the main decomposition step, possibly by removing reactive non-cellulosic material, without shifting its timing. The treated composite also retained more residual mass at 600°C than the untreated one (10.95% versus 9.28%), closer to the 11.72% left by the pure control (Figure 9), suggesting the treatment partially restores char-forming behavior that untreated filler tends to reduce.

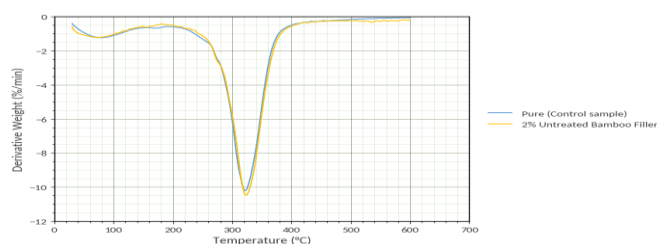


Figure 7. Derivative thermogravimetric curves of unfilled and 2% bamboo filler-filled composites

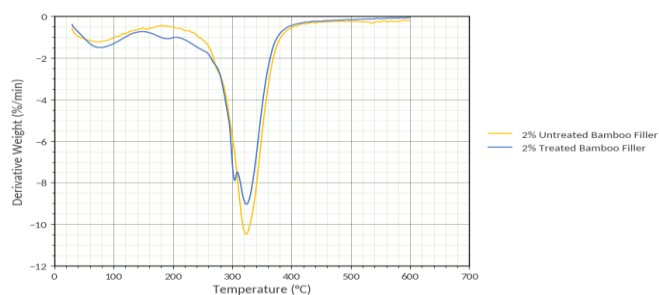


Figure 8. Derivative thermogravimetric curves of 2% untreated and treated bamboo filler-filled composites

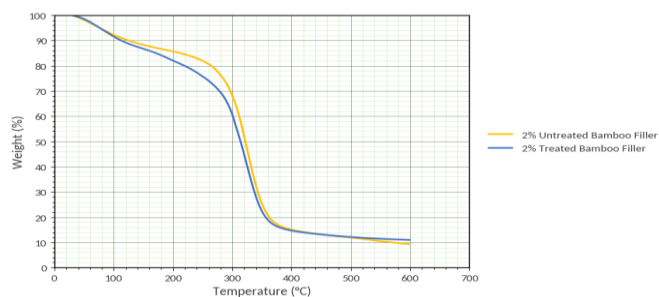


Figure 9. Thermogravimetric weight (%) curves of 2% untreated and treated bamboo filler-filled composites

3.4. Biodegradation Behavior

Microbial colonization in freshwater increased as bamboo filler content increased for both treated and untreated films, as shown in Table 1. Water absorption and microbial growth were encouraged by the hydrophilic properties of starch and bamboo filler in conjunction with glycerol plasticization.

Table 1. Biodegradability test Day 50, 55 and 60

Sample name	Day 50-F	Day 50-S	Day 55-F	Day 55-S	Day 60-F	Day 60-S
P						
U2						
U4						
U6						
U8						
U10						
T2						
T4						
T6						
T8						
T10						

Notably, films containing 4 wt% bamboo filler exhibited minimal microbial colonization, suggesting that ideal bamboo filler dispersion reduced surface porosity and nutrient accessibility. No discernible microbial growth was observed in the simulated seawater samples during the observation period.

Alkaline treatment appeared to have little effect on the visible deterioration of films during the immersion test.

CONCLUSION

Solution casting was used to successfully create corn starch-based films reinforced with bamboo filler, and the effects of bamboo filler loading and alkaline treatment were assessed. When compared to untreated bamboo filler composites, alkaline treatment with 5 wt% NaOH improved bamboo filler–corn starch matrix adhesion, leading to appreciable improvements in tensile strength and thermal uniformity. The intrinsic thermal limitation of starch-based matrices was confirmed by the fact that all films showed thermal degradation onset around 300°C. Studies on biodegradation showed that while no discernible microbial growth was observed in simulated seawater, freshwater environments had more microbial colonization. These results support the potential use of alkaline-treated bamboo filler-reinforced starch films as sustainable substitutes for traditional plastic packaging because they provide a balanced combination of mechanical performance, thermal stability, and biodegradability.

Future work could explore a wider range of alkaline treatment concentrations and soaking times to improve filler–matrix compatibility, and extend biodegradation testing to field or soil-burial conditions for a more realistic assessment. Compatibilizers or additional reinforcement types might also help offset the ductility loss seen at higher filler loadings, broadening potential packaging applications for these composites.

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