



Effect of Pepper Stem Fiber Orientation on Mechanical Properties, Water Absorption, and Biodegradation of Cassava Bioplastic Films

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ABSTRACT

Plastic pollution continues to pose a serious environmental challenge due to the persistence of petroleum-based polymers in ecosystems. Starch-based bioplastics offer a biodegradable alternative; however, their practical application is limited by low mechanical strength and high water absorption. This study investigates the effect of pepper stem waste fiber (*Piper nigrum*) reinforcement and fiber orientation (aligned vs. random) on the mechanical properties, water absorption, and biodegradation behavior of cassava starch films. Bioplastic films were prepared in three formulations: control (without fibers), aligned fiber reinforcement (AFR), and random fiber reinforcement (RFR). Structural and performance evaluations were conducted using X-ray diffraction (XRD), tensile testing, water absorption analysis, and soil burial biodegradation (8 cm depth). XRD patterns confirmed semi-crystalline starch structures with characteristic peaks at $2\theta \approx 17^\circ$, 19° , and 22° . Tensile strength increased from 0.6957 MPa (control) to 0.7873 MPa (AFR), corresponding to a 13.2% improvement, indicating enhanced load transfer in aligned fiber structures. Water absorption decreased from 70% (control) to 50% (RFR), showing a 28.6% reduction, likely due to increased tortuosity of water diffusion pathways. Soil burial testing showed rapid degradation, reaching 28.37% mass loss by day 3 and 100% mass loss/disintegration by day 6 under the tested conditions. Overall, pepper stem fibers improve the performance of cassava starch films while maintaining rapid biodegradability, highlighting their potential for environmentally friendly disposable and packaging applications.

1. Introduction

Plastic pollution remains one of the most pressing environmental challenges worldwide due to the persistence and accumulation of petroleum-based polymers in terrestrial and marine ecosystems [1,2]. Conventional plastics degrade extremely slowly and contribute to long-term environmental contamination, biodiversity loss, and microplastic entry into food chains [3]. In Indonesia, plastic waste generation continues to increase annually, intensifying the need for sustainable alternatives that can reduce environmental burdens [4,5].

Bioplastics derived from renewable resources have received growing attention as potential substitutes for synthetic plastics [6,7]. Among these, starch-based materials are particularly attractive because starch is abundant, inexpensive, and capable of forming continuous films [8,9]. Cassava starch is widely available in tropical regions and offers strong potential for biodegradable packaging and disposable materials [10,11]. However, cassava starch films generally exhibit low tensile strength and high water absorption due to the hydrophilic nature of starch molecules and the porous microstructure formed during film casting [12,13]. These limitations restrict the use of cassava starch films in applications

that require mechanical integrity and moisture resistance.

To address these weaknesses, researchers have explored reinforcement using natural fibers, which can improve stiffness and strength through stress transfer from matrix to fiber [14]. Agricultural residues such as bagasse [15], bamboo fibers [16], and cellulose-based reinforcements have been widely studied [17,18], often producing tensile strengths above 6 MPa and water absorption values below 10% when combined with optimized fiber fractions, chemical modification, and/or hydrophobic additives [19,20,21]. While these high-performance systems demonstrate strong potential, they typically involve additional processing complexity and matrix treatments. In contrast, the present study focuses on a simple, low-cost reinforcement strategy using agricultural waste fibers without chemical modification of the cassava starch matrix; therefore, the mechanical and water resistance values are expected to be lower than chemically optimized composites, but still meaningful for disposable and short-lifetime applications.

Pepper stem waste (*Piper nigrum*) is an abundant but underutilized agricultural residue in

pepper-producing regions such as South Sulawesi, Indonesia. This biomass is cellulose-rich and offers a promising reinforcement source for biodegradable films, while simultaneously supporting waste valorization and circular economy practices [21]. In addition to fiber composition, the orientation of fibers is known to influence composite performance. Mechanistically, aligned fibers are expected to improve tensile strength by enabling more efficient load transfer along the direction of stress, whereas random fiber networks may reduce water absorption by creating tortuous diffusion pathways that hinder water penetration [22,23]. Despite this mechanistic expectation, systematic studies on how fiber orientation affects the performance of cassava starch films reinforced specifically with pepper stem waste fibers remain limited.

To date, the effect of pepper stem waste fiber orientation (aligned versus random) on the mechanical properties, water absorption, and biodegradation behavior of cassava starch films has not been clearly established.

Therefore, this study investigates cassava starch films reinforced with pepper stem waste fibers using aligned and random fiber configurations, and evaluates their structural characteristics (XRD), tensile strength, water absorption, and soil burial biodegradation. The results are expected to provide an orientation-based understanding of performance changes in waste-fiber-reinforced starch films and support the development of environmentally friendly disposable materials.

2. Materials and Methods

2.1 Materials

Cassava starch was used as the polymer matrix (analytical grade). Glycerin was used as a plasticizer (Merck, analytical grade), and glacial acetic acid was used as a solvent/compatibilizer (Merck, analytical grade). Sodium hydroxide (NaOH, Merck, analytical grade) was used for alkali treatment of pepper stem fibers. Pepper stem waste (*Piper nigrum*) was collected from Towuti, Luwu Timur, South Sulawesi, Indonesia. Distilled water was used for all preparations and testing.

2.2 Fiber Preparation

Pepper stem waste was washed thoroughly with distilled water to remove dirt and dried at room temperature. The dried stems were cut into 1–2 cm pieces and treated in 5% (w/v) NaOH solution at 60 °C for 2 h to remove hemicellulose and lignin, which increases fiber surface roughness and improves interfacial bonding with starch matrices. After treatment, fibers were rinsed repeatedly with distilled water until neutral pH, oven-dried at 60 °C for 24 h, and ground to an average fiber length of approximately 3–5 mm prior to use.

2.3 Film Preparation

Cassava starch (5 g) was dissolved in 100 mL distilled water under magnetic stirring. Glycerin (1.5

mL) and acetic acid (1 mL) were added and heated while stirring until gelatinization occurred (~5 min). The gelatinized slurry was poured into circular polystyrene molds (diameter: 9 cm) to form films. Three formulations were prepared (Fig. 1): (i) control films without fibers, (ii) aligned fiber reinforcement (AFR), and (iii) random fiber reinforcement (RFR). For AFR films, pepper stem fibers (0.50 g, corresponding to 10 wt% relative to starch mass) were arranged manually in a single direction on the mold surface prior to casting, and the slurry was poured gently to preserve the orientation. For RFR films, the same amount of pepper stem fibers (0.50 g, 10 wt%) was dispersed directly into the slurry and mixed for 2 min to achieve random distribution before casting. Films were dried at room temperature (27 ± 2 °C) for 24 h and conditioned for 24 h at $50 \pm 5\%$ relative humidity before testing. The final average film thickness was 0.30 ± 0.05 mm.

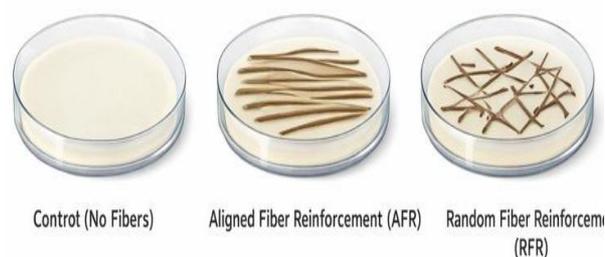


Fig 1: Pepper Stem Fiber Orientation

2.4 X-Ray Diffraction (XRD)

X-ray diffraction (XRD) analysis was performed using a Shimadzu 7000 diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5405$ Å), operated at 30 kV and 10 mA. The diffraction patterns were recorded over a scanning range of $2\theta = 15\text{--}70^\circ$ with a step size of 0.02° and a scanning speed of $2^\circ/\text{min}$. The XRD patterns were used to identify characteristic crystalline peaks of starch and to observe structural changes after fiber reinforcement.

2.5 Water Absorption Test

Water absorption was evaluated by immersing dried film specimens ($30 \text{ mm} \times 10 \text{ mm} \times 0.30 \text{ mm}$) in distilled water at room temperature (27 ± 2 °C) for 24 h. Prior to immersion, the specimens were dried to constant mass and weighed (W_1). After immersion, samples were removed, gently wiped with tissue paper to remove surface water, and immediately weighed again (W_2). Water absorption (%) was calculated using the following equation [8].

$$\text{Water absorption}(\%) = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

2.6 Biodegradation Test

Biodegradation was evaluated using a soil burial mass-loss method. Garden loamy soil was used as the biodegradation medium, with moisture content maintained at approximately 20–25% and pH in the range of 6.5–7.0. Film specimens ($30 \text{ mm} \times 10 \text{ mm}$)

were buried at a depth of 8 cm and retrieved after 3 and 6 days. After retrieval, the samples were washed gently with distilled water to remove soil particles, then oven-dried at 60 °C until constant mass before weighing. Percentage mass loss was calculated relative to the initial dry mass. In this study, the term “100% mass loss/disintegration under tested soil burial conditions” is used when specimens could no longer be recovered in measurable form.

2.7 Tensile Strength Test

Tensile strength was measured using an IK-MT-30.71 tensile tester at room temperature. Film specimens (80 mm × 10 mm × 0.30 mm) were prepared and tested with a gauge length of 50 mm and a crosshead speed of 5 mm/min. Each formulation was tested in triplicate (n = 3). Tensile strength (MPa) was calculated as the maximum force divided by the initial cross-sectional area of the specimen. Results are reported as mean values; future studies should incorporate standard deviations and statistical analysis to strengthen comparisons among formulations.

3. Material and Methods

3.1 X-Ray Diffraction (XRD) Analysis

All samples exhibited semi-crystalline diffraction patterns (Fig. 2) with characteristic peaks of starch-based films at $2\theta \approx 17^\circ$, 19° , and 22° , which are commonly associated with the crystalline regions of amylose and amylopectin. These peaks indicate that the cassava starch matrix retained its semi-crystalline structure after film formation and fiber incorporation [24, 25]. Minor changes in peak intensity were observed between formulations, suggesting that fiber addition and orientation may influence the degree of molecular ordering, likely through differences in interfacial interactions and packing behavior during drying [26].

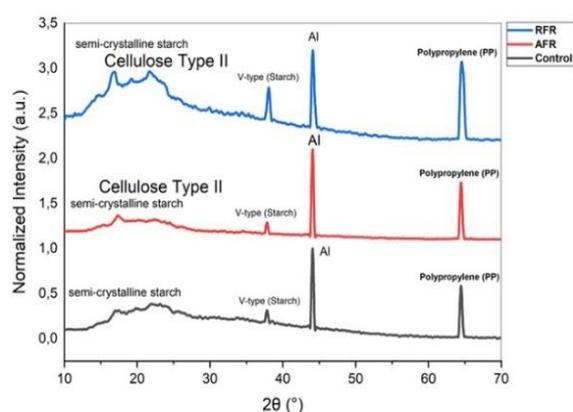


Fig 2: XRD patterns of bioplastic samples

In addition to the main starch peaks, weak reflections were observed at approximately $2\theta \approx 37^\circ$ and 44° . These peaks may originate from residual crystalline cellulose, naturally occurring inorganic impurities associated with pepper stem fibers, or experimental artifacts introduced during sample preparation [27, 28] (e.g., contributions from sample

holders or mounting materials). Because no complementary compositional characterization (such as FTIR or elemental analysis) was performed in this study, the attribution of these minor peaks remains tentative and should be interpreted cautiously. Therefore, these reflections are reported as minor additional peaks rather than definitive phase identification.

A weak reflection was also observed at high angles around $2\theta \approx 64\text{--}65^\circ$, which coincides with the reference peak position commonly reported for polypropylene (PP). However, since PP is not a component of the formulation and cannot be generated from glycerin or starch under the present processing conditions, this reflection is most likely caused by contamination from polymer-based laboratory materials [29, 30] (e.g., plastic petri dishes, containers, or sample handling tools) or an instrument/reference-library annotation artifact. Thus, this peak was not assigned as a constituent phase of the cassava starch films, and the interpretation in this study focuses only on diffraction features attributable to the starch matrix and pepper stem fibers.

3.2 Biodegradation Behaviour

Biodegradation behavior of the cassava starch films was evaluated using a soil burial mass-loss method [8]. All formulations exhibited rapid disintegration under the tested conditions, reflecting the hydrophilic nature of starch-based matrices and their susceptibility to moisture-driven hydrolysis and microbial activity. After 3 days of burial, the samples showed an average mass loss of 28.37%, indicating that biodegradation had progressed substantially even within a short exposure period. By day 6, the films reached 100% mass loss/disintegration under the tested soil burial conditions, meaning that the specimens could no longer be recovered in measurable form for weighing (Fig. 3).

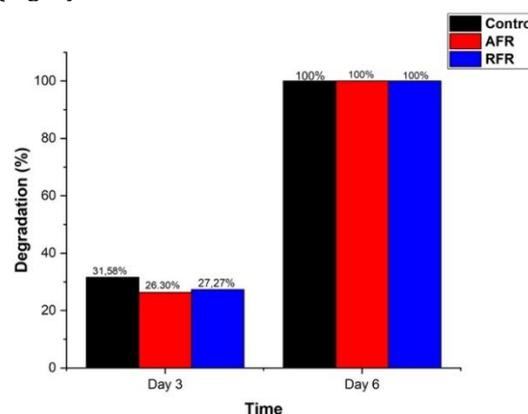


Fig 3: Biodegradation rate over 6 days

The incorporation of pepper stem fibers did not inhibit biodegradation. Fiber-reinforced films showed similar degradation trends to the control, which may be attributed to the biodegradable nature of the cellulose-based fibers. In the early stage, fiber-reinforced samples may exhibit slightly delayed

disintegration due to a denser structure that slows water penetration into the starch matrix; however, the overall biodegradation remained rapid [9, 31]. Within the observed time window (3–6 days), no clear difference was detected between aligned (AFR) and random (RFR) fiber configurations. Therefore, under the tested conditions, fiber orientation did not show an observable influence on biodegradation rate, although longer-term assessment and additional biodegradation indicators (e.g., CO₂ evolution or microbial activity measurements) would be required to support broader biodegradability claims beyond mass-loss/disintegration.

3.3 Water absorption

Water absorption results (Fig 4.) demonstrate that pepper stem fiber reinforcement and fiber orientation influence the moisture sensitivity of cassava starch films. The control film exhibited the highest water absorption (70%), which is expected due to the highly hydrophilic nature of starch and the presence of microvoids that facilitate rapid water uptake. Fiber-reinforced films showed reduced water absorption, particularly in the random fiber reinforcement (RFR) formulation, which exhibited the lowest value (50%). This reduction ($\approx 28.6\%$ relative to the control) suggests that randomly distributed fibers may improve the barrier behavior of the film by increasing the tortuosity of water diffusion pathways, thereby slowing moisture penetration through the matrix [3,15].

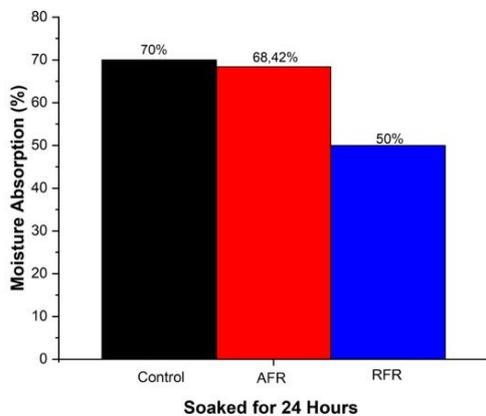


Fig 4: Water absorption of different composites

In contrast, the aligned fiber reinforcement (AFR) film exhibited water absorption of 68.42%, which is only slightly lower than the control. This behavior may be attributed to preferential moisture transport along aligned fiber–matrix interfaces, where continuous interfacial pathways can promote water ingress. Additionally, the aligned arrangement may introduce microchannels or interfacial discontinuities in the direction of fiber alignment, reducing the effectiveness of fiber reinforcement in restricting water diffusion compared to a randomly dispersed fiber network [16, 17, 18].

When compared with literature reports of much lower water absorption values (e.g., below 10%) [16, 18, 20], it is important to recognize that those high-

performance starch composites often rely on optimized fiber fractions, chemical modifications (such as crosslinking or hydrophobic treatments), or incorporation of water-resistant additives that reduce hydroxyl group accessibility and improve film density. In the present study, the formulation employs a simple, low-cost approach using agricultural waste fibers without chemical modification of the cassava starch matrix, which naturally retains high water affinity [22, 23]. Therefore, although the absolute absorption values remain relatively high, the observed reduction in RFR films indicates meaningful improvement and demonstrates the potential of pepper stem fibers—particularly with random orientation—to enhance the water resistance of cassava starch films for short-lifetime disposable applications.

3.4 Tensile Strength

The tensile strength results indicate that pepper stem waste fibers can enhance the mechanical performance of cassava starch films, although the magnitude of improvement is moderate. The control film exhibited the lowest tensile strength (0.6957 MPa), reflecting the intrinsic brittleness and weak intermolecular bonding of starch-based matrices. Fiber-reinforced films showed higher tensile strength values, reaching 0.7659 MPa for the random fiber reinforcement (RFR) film and 0.7873 MPa for the aligned fiber reinforcement (AFR) film. The increase from the control to AFR corresponds to an improvement of approximately 13.2%, suggesting that fiber addition contributes to improved stress-bearing capacity.

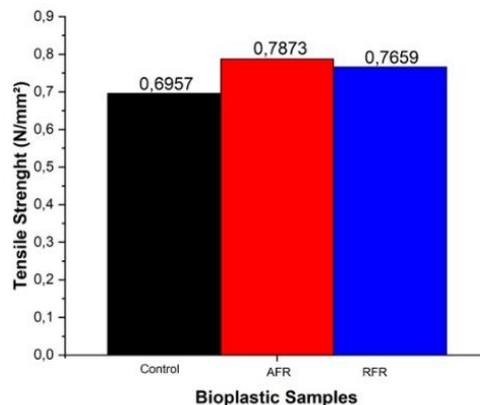


Fig 5: Tensile strength comparison between formulations

The slightly higher tensile strength of AFR compared to RFR can be explained by orientation-dependent load transfer mechanisms. In the aligned configuration, fibers are positioned in a consistent direction, which facilitates more efficient stress transfer from the starch matrix to the fibers along the loading axis, thereby improving tensile resistance. In contrast, randomly distributed fibers contribute to reinforcement through multidirectional bridging and crack deflection; however, the random arrangement

may reduce effective stress transfer along the tensile axis and may increase the likelihood of fiber entanglement or local agglomeration, which can limit the improvement in tensile strength [8, 13, 20].

4. Conclusion

This study shows that pepper stem waste fibers (*Piper nigrum*) can reinforce cassava starch films and that fiber orientation influences performance. Aligned fiber reinforcement (AFR) moderately increased tensile strength from 0.6957 MPa to 0.7873 MPa (~13.2%), consistent with improved load transfer along the alignment direction, while random fiber reinforcement (RFR) reduced water absorption from 70% to 50%, indicating enhanced moisture resistance through tortuous diffusion pathways. XRD confirmed the semi-crystalline structure of starch films with minor additional reflections that may relate to fiber residues or experimental artifacts, requiring further compositional verification. Soil burial testing indicated rapid biodegradation, reaching 28.37% mass loss by day 3 and 100% mass loss/disintegration by day 6 under the tested conditions. Future work should include longer biodegradation assessment, statistical analysis with error reporting, and optimization of fiber fraction and interfacial bonding to further improve film performance.

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