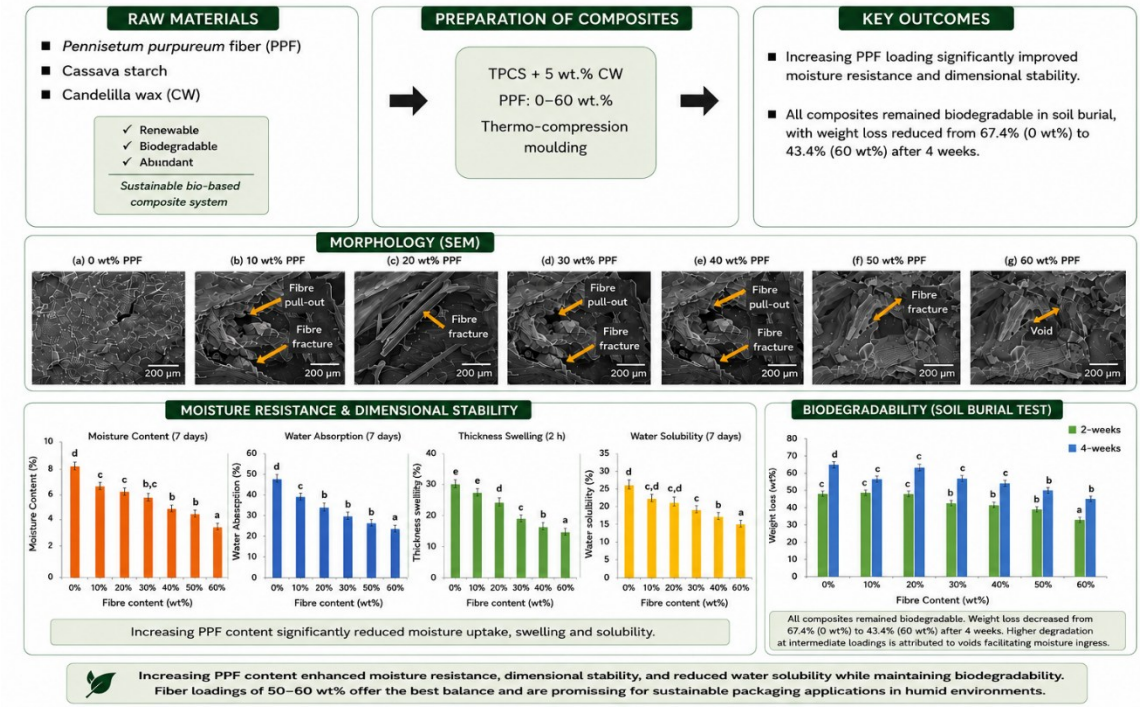
Moisture Resistance, Dimensional Stability, and Biodegradation Behavior of *Pennisetum purpureum* Fiber-reinforced Thermoplastic Cassava Starch/Candelilla Wax Composite

Mohd Fairus Kayat [a], Ridhwan Jumaidin [b,*] Lailatul Harina Paijan,[a,*] Fahmi Asyadi Md Yusof,[c] Zatil Hafila Kamaruddin,[d] Mohammad Khalid Wahid,[a] and Melbi Mahardika [e]

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GRAPHICAL ABSTRACT



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The practical use of starch-based biodegradable composites is often limited by high moisture sensitivity and dimensional instability in humid environments. This study investigates the effect of *Pennisetum purpureum* fiber (PPF) loading on the physical properties, moisture behaviour, and biodegradation performance of thermoplastic cassava starch/candelilla wax (TPCS/CW) composites prepared by thermo-compression moulding with fiber contents ranging from 0 to 60 wt%. Increasing fiber loading reduced density and significantly improved resistance to moisture-related deterioration. Moisture content, water absorption, thickness swelling, and water solubility decreased progressively with higher PPF content, indicating enhanced dimensional stability and reduced water permeability. After seven days of exposure, equilibrium moisture absorption decreased from 8.3% in neat TPCS/CW to 3.8% at 60 wt% PPF, while water solubility decreased from 29.2% to 13.1%. Soil burial testing confirmed that all composites remained biodegradable, although higher fiber loading moderated the degradation rate, with weight loss reduced from 67.4% in the neat matrix to 43.4% at 60 wt% after four weeks. Slightly higher degradation at intermediate fiber contents was attributed to interfacial voids that facilitated moisture ingress. Overall, PPF incorporation improved moisture resistance and structural stability while preserving biodegradability, supporting the potential of this fully bio-based composite for biodegradable packaging films, disposable packaging liners, and paperboard coating applications requiring moderate moisture resistance under humid conditions.

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Keywords: Pennisetum purpureum fiber; TPCS; Candelilla wax; Moisture absorption; Water solubility; Thickness swelling; Soil burial

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INTRODUCTION

The extensive use of petroleum-based plastics has resulted in serious environmental concerns due to their resistance to degradation and long-term accumulation in natural ecosystems. Growing awareness of plastic pollution and sustainability issues has intensified research efforts toward the development of biodegradable materials derived from renewable resources (Cataño *et al.* 2023; Arruda *et al.* 2025). Among these materials, starch-based polymers have attracted significant attention because of their abundance, biodegradability, low cost, and availability from agricultural sources such as cassava, corn, and potato (Syazmini et al. 2023; Wang et al. 2023).

Cassava starch is particularly appealing for sustainable material development in tropical regions because of its widespread cultivation and established supply chain. However, native starch exhibits several inherent limitations, including high hydrophilicity, poor mechanical strength, and limited thermal stability, which restrict its use in practical applications (Hazrati et al. 2021a; Wang et al. 2023). Plasticisation using agents such as glycerol enables the transformation of starch into thermoplastic starch (TPS), allowing processing under heat and pressure (Hazrati *et al.* 2021b; Syazmini *et al.* 2023). Cassava starch differs from other commonly used starch sources, such as corn and potato starch, in terms of amylose-to-amylopectin ratio, granule morphology, gelatinization behaviour, and moisture sensitivity, all of which influence the formation and performance of thermoplastic starch matrices (Hazrati *et al.* 2021a; Wang *et al.* 2023b). These characteristics can affect intermolecular interactions with reinforcing fibers and the effectiveness of hydrophobic additives in improving moisture resistance and dimensional stability. Consequently, findings reported for other starch systems cannot always be directly extrapolated to cassava starch-based composites, highlighting the need for dedicated investigations involving cassava starch formulations intended for biodegradable materials exposed to humid environments (Syazmini *et al.* 2023; Wang *et al.* 2023b).

Despite some improvements, TPS remains highly sensitive to moisture and suffers from dimensional instability, particularly under humid conditions commonly encountered in tropical climates (Kamaruddin *et al.* 2023; Arruda *et al.* 2025). To overcome these limitations, reinforcement with natural fibers and modification with hydrophobic additives have been widely explored. Natural fibers offer advantages such as renewability, biodegradability, low density, and the ability to enhance stiffness and dimensional stability by restricting polymer chain mobility (Azlin *et al.* 2020; Schutz *et al.* 2024). However, the effectiveness of fiber reinforcement is strongly influenced by fiber loading, dispersion, and interfacial compatibility. Excessive fiber content can lead to fiber agglomeration and void formation, which negatively affect moisture resistance and mechanical reliability (Fazeli *et al.* 2019; Ibrahim *et al.* 2020). Candelilla wax (CW), a plant-derived wax with high hydrophobicity and thermal stability, has been increasingly incorporated into bio-based materials to improve moisture resistance and barrier performance. CW is widely used in food and pharmaceutical applications and is approved as a food additive (E 902) in the European Union and classified as Generally Recognised as Safe (GRAS) in the United States (Aranda-Ledesma *et al.* 2022; Kowalczyk *et al.* 2024). Previous studies have demonstrated that CW incorporation into starch- and protein-based systems can effectively reduce moisture uptake and improve dimensional stability without compromising biodegradability (Kowalczyk *et al.* 2024; Núñez-García *et al.* 2022). *Pennisetum purpureum* (Napier grass) is a fast-growing perennial grass widely cultivated in tropical and subtropical regions. Owing to its high biomass yield and favourable lignocellulosic

composition, it has gained increasing attention as a reinforcement material for biodegradable composites (Beber *et al.* 2025; Islam *et al.* 2023). The properties of *Pennisetum purpureum* fibers are influenced by harvesting age, which affects fiber rigidity, lignin content, and compatibility with hydrophilic polymer matrices (Dresch *et al.* 2025; Meel *et al.* 2025). At early growth stages, the fibers exhibit improved interaction with starch-based matrices while maintaining sufficient structural reinforcement.

In addition to improving moisture resistance, starch-based composites are increasingly being explored for packaging-related applications where moderate barrier performance and biodegradability are required. Such applications include biodegradable packaging films, disposable packaging liners, and coating layers for paper-based packaging materials. In these applications, dimensional stability, reduced water sensitivity, and environmental sustainability are often more critical than achieving the high barrier performance associated with conventional petroleum-based plastics. Therefore, enhancing the moisture resistance and structural stability of starch-based composites remains an important step toward expanding their practical use in sustainable packaging systems (Núñez-García *et al.* 2022; Kowalczyk *et al.* 2024).

Although natural fiber-reinforced starch composites and wax-modified starch systems have been widely reported, limited studies have systematically examined the combined effects of *Pennisetum purpureum* fiber and candelilla wax on the physical durability and environmental performance of thermoplastic cassava starch matrices. Furthermore, the influence of fiber loading on density, moisture behaviour, dimensional stability, and biodegradation performance remains insufficiently understood for cassava starch-based composite systems.

It should be noted that the mechanical performance of the same TPCS/CW/PPF composite system has been comprehensively reported in a recent companion study by the present authors (Kayat *et al.* 2026). Using identical formulations, fibre loadings (0 to 60 wt%), and processing conditions, the incorporation of PPF significantly improved tensile, flexural, and impact properties, with optimum performance achieved at 50 wt% fibre loading. The present study therefore complements those findings by focusing specifically on density, moisture behaviour, water absorption, dimensional stability, and biodegradation performance, which are equally important for assessing the practical applicability of starch-based biodegradable composites.

Therefore, this study aimed to evaluate the influence of *P. purpureum* fiber loading on the density, moisture behaviour, water absorption, and related physical properties of thermoplastic cassava starch/candelilla wax (TPCS/CW) composites. By examining how fiber incorporation affects these key parameters, this study sought to provide insight into the development of lightweight and environmentally durable fully bio-based composites suitable for sustainable material applications.

EXPERIMENTAL

Materials

Pennisetum purpureum grass was collected from Ladang Napier Selatan, Rembau, Negeri Sembilan, Malaysia. The grass was harvested at 30 days of age, corresponding to the early vegetative stage commonly used for fiber extraction (Beber *et al.* 2025; Meel *et al.* 2025). The harvested stems were washed and subjected to water retting for 14 days. The fibers were separated from the retted stems, rinsed, sun-dried for 48 h, and oven-dried at

85 °C for 24 h to remove residual moisture (Dresch *et al*. 2025). The dried fibers were cut into short fibers with an average length of approximately 1 cm. Cassava starch powder was supplied by THC Sdn. Bhd. (Malaysia). Glycerol (99.5% purity) was obtained from QReC Chemicals Sdn. Bhd. (Malaysia) and used as a plasticiser. Candelilla wax was supplied by Evergreen Engineering & Resources Sdn. Bhd. (Malaysia).

Composite Fabrication

Thermoplastic cassava starch/candelilla wax (TPCS/CW) composites reinforced with *Pennisetum purpureum* fiber (PPF) were prepared using compression moulding. The matrix formulation consisted of 65 wt% cassava starch, 30 wt% glycerol, and 5 wt% CW. Fiber contents of 0, 10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%, and 60 wt% were incorporated relative to the total composite weight. All components were mixed using a Panasonic MX-GM1011 dry mixer at 1200 rpm for 5 min. The mixtures were compression moulded at 150 °C for 60 min under a load of 10 tonnes, corresponding to an applied pressure of approximately 7.55 MPa based on the mould cavity area of 130 mm × 100 mm, using a GOTECH GT7014-P30 C hydraulic moulding press. The fabricated sheets had an average thickness of 3 mm and were stored in a desiccator prior to testing.

Scanning Electron Microscope

The fractured surfaces of the tensile-tested specimens were examined to evaluate the surface morphology and fiber–matrix interaction. Microstructural analysis was carried out using a Schottky Field Emission (Japan) scanning electron microscope (SEM) operated at an accelerating voltage of 10 kV. Prior to observation, the fractured samples were cut into suitable dimensions and sputter-coated with a thin layer of gold to improve electrical conductivity and prevent surface charging during imaging. The tensile specimens were stored in sealed zip-lock bags and subsequently placed in a desiccator containing silica gel prior to SEM examination to minimise contamination and moisture uptake from the environment.

Density

The density of TPCS/CW/PPF composite was determined following the ASTM D1895 standard. Five specimens with dimensions of 10 × 10 × 3 mm were prepared for each formulation. Prior to measurement, all samples were oven-dried at 105 °C for 24 h to remove residual moisture. The dried specimens were then stored in a desiccator containing silica gel to prevent moisture uptake before testing. The mass and volume of each specimen were measured using a precision analytical balance and an electronic densimeter (H-300S, Hildebrand, Germany). The density of the composites was calculated as the ratio of mass to volume using Equation (1).

$$Density\ \left(\frac{g}{cm3}\right) = \frac{Mass\ (g)}{Volume\ (cm3)} \quad (1)$$

Moisture Content

Moisture content analysis was carried out using five specimens for each composite formulation. The samples were initially weighed to obtain their initial mass (Wi) and subsequently oven-dried at 105 °C for 24 h to achieve a constant weight. After drying, the final mass (W_f) of each specimen was recorded. The moisture content was determined based on the mass loss during drying, following the procedure reported by Ilyas *et al*. (2017). The percentage moisture content was calculated using Eq. 2:

$$Moisture\ content\ (\%) = \frac{Wi-Wf}{Wi} \text{ x } 100 \quad (2)$$

Water Absorption

Water absorption behaviour of the TPCS/CW/PPF composites was evaluated using five specimens with dimensions of 10 × 10 × 3 mm. Prior to testing, all samples were oven-dried at 105 ± 2 °C for 24 h in an air-circulating oven to eliminate residual moisture. After drying, the initial mass (W_i) of each specimen was recorded. The specimens were then fully immersed in distilled water at ambient temperature (23 ± 1 °C) for 2 h. Upon removal from the water, surface moisture was gently wiped off, and the final mass (W_f) was measured immediately. The percentage water absorption was calculated based on the change in mass using Eq. 3:

$$Water\ absorption\ (\%) = \frac{Wi-Wf}{Wi}\ x\ 100 \quad (3)$$

Thickness Swelling

Thickness swelling was evaluated based on the water immersion procedures Thickness swelling was evaluated in accordance with ASTM D5229 (2020) and adapted from previous studies (Jawaid *et al.* 2011; Kamaruddin *et al.* 2023). Five specimens with dimensions of 10 × 10 × 3 mm³ were prepared, lightly cleaned, and oven-dried at 105 ± 2 °C for 24 h prior to testing. The initial thickness of each specimen (T_i) was measured before immersion. The specimens were then immersed in 30 mL of distilled water at room temperature (23 ± 1 °C) for 2 h, after which the final thickness (T_f) was recorded. The percentage of thickness swelling was calculated using Eq. 4:

$$Thickness\ swelling\ (\%) = \frac{Ti-Tf}{Ti}\ x\ 100 \quad (4)$$

Moisture Absorption

Moisture uptake was evaluated by placing the TPCS/CW composites in a sealed climate chamber (Memmert GmbH, Germany) maintained at 25 ± 2 °C and 75 ± 2% relative humidity. Five specimens with dimensions of 10 × 10 × 3 mm³ were prepared and pre-dried at 105 ± 2 °C for 24 h prior to testing. The specimen masses were recorded before and after exposure, denoted as W_i (initial mass) and W_f (final mass), and used to calculate moisture uptake according to Eq. 5. The measurements were continued until the samples reached equilibrium moisture content.

$$Moisture\ Absorption\ (\%) = \frac{Wf-Wi}{Wi}\ x\ 100 \quad (5)$$

Water Solubility

Water solubility was determined using a procedure adapted from a previous study (Reddy *et al.* 2018). Five specimens with dimensions of 10 × 10 × 3 mm³ were first oven-dried at 105 ± 2 °C for 24 h to obtain the initial mass (W_i). The dried specimens were then immersed in 30 mL of distilled water and gently agitated for 24 h. After immersion, the specimens were recovered by filtration, excess surface water was removed using filter paper, and the samples were oven-dried again at 105 ± 2 °C to a constant mass, recorded as the final mass (W_f). The percentage of water solubility was calculated using Eq. 6:

$$Water\ Solubility\ (\%) = \frac{Wi-Wf}{Wi}\ x\ 100 \quad (6)$$

Soil Burial

Biodegradation was evaluated using a soil burial test adapted from the method reported in previous studies (Su *et al.* 2023). The average environmental conditions during the test were a temperature of 26 ± 4 °C, relative humidity of 76 ± 4%, and a soil pH of approximately 6.5. Soil moisture content was not quantitatively monitored during the burial period. However, distilled water was periodically added throughout the experiment to maintain consistently moist soil conditions favourable for microbial activity and biodegradation. Each specimen was enclosed in a fine iron mesh prior to burial to facilitate retrieval while allowing contact with moisture and microorganisms. Before burial, the specimens were oven-dried at 105 °C for 24 h and weighed to obtain the initial mass (W_i). The samples were buried for periods of 2 and 4 weeks, after which they were gently recovered, rinsed with distilled water to remove adhering soil, and oven-dried again at 105 °C for 24 h to determine the final mass (W_f). The percentage of weight loss due to biodegradation was calculated using Eq. 7:

$$Weigth\ loss\ (\%) = \frac{Wi-Wf}{Wi}\ x\ 100 \quad (7)$$

Statistical Analysis

Statistical analysis was carried out to evaluate the significance of differences among the mean values of the measured properties. A one-way analysis of variance (ANOVA) was conducted at a significance level of $P < 0.05$. When statistically significant differences were observed, Duncan's multiple range test was employed as a post hoc analysis to compare the individual group means at the same confidence level.

RESULTS AND DISCUSSION

Density

The influence of *Pennisetum purpureum* fiber (PPF) loading on the density of TPCS/CW composites is shown in Fig. 1. A clear and consistent reduction in density was observed as the fiber content increased from 0 to 60 wt%. The composite without fiber exhibited the highest density, approximately 1.33 g/cm³, while progressive fiber incorporation led to a gradual decrease in density, reaching a minimum value of about 1.10 g/cm³ at 60 wt% PPF. Overall, this corresponds to a reduction of roughly 17% across the investigated fiber loading range.

This decreasing trend can be primarily attributed to the lower intrinsic density of PPF compared to the TPCS/CW matrix. As the fiber loading increases, a larger fraction of the relatively dense starch–wax matrix is replaced by the lighter lignocellulosic fiber, while the overall composite volume remains largely unchanged. Consequently, the mass per unit volume decreases, resulting in a lower composite density. Similar density reductions have been widely reported in natural fiber-reinforced starch-based composites, where lightweight fibers displace a portion of the polymeric matrix material (Kamaruddin et al. 2022; Kibet et al. 2025). In addition to the density contrast between fiber and matrix, the internal structure of lignocellulosic fibers also contributes to this behaviour. The PPF have a hollow lumen and a porous microstructure, which further reduce their effective density. It is also possible that a portion of the fiber lumens remained partially unfilled by the

starch–wax matrix during composite fabrication. Such internal void spaces would contribute to a lower effective density by increasing the composite volume without a proportional increase in mass. Although lumen occupancy was not directly examined in the present study, the hollow cellular structure of lignocellulosic fibers is widely recognised as an important factor contributing to the lightweight nature of natural fiber-reinforced composites (Olhan *et al.* 2021; Kibet *et al.* 2025).As the fiber fraction increases, these structural features increasingly influence the overall composite, leading to a more pronounced reduction in density. Comparable trends have been documented in thermoplastic starch composites reinforced with banana, kenaf, and sisal fibers, where density reductions in the range of 10% to 20% were achieved at higher fiber contents (Kibet *et al.* 2025).

The present findings are also consistent with the observations reported by Bolaños *et al*. (2025), who noted that the incorporation of low-density natural fibers into starch-based matrices results in a noticeable decrease in composite density. From a practical standpoint, the reduction in density associated with higher fiber loadings is advantageous for the development of lightweight biodegradable materials. Lower density can improve handling efficiency and reduce transportation costs, while maintaining the sustainability benefits inherent to bio-based composite systems (Olhan *et al.* 2021; Kibet *et al.* 2025).

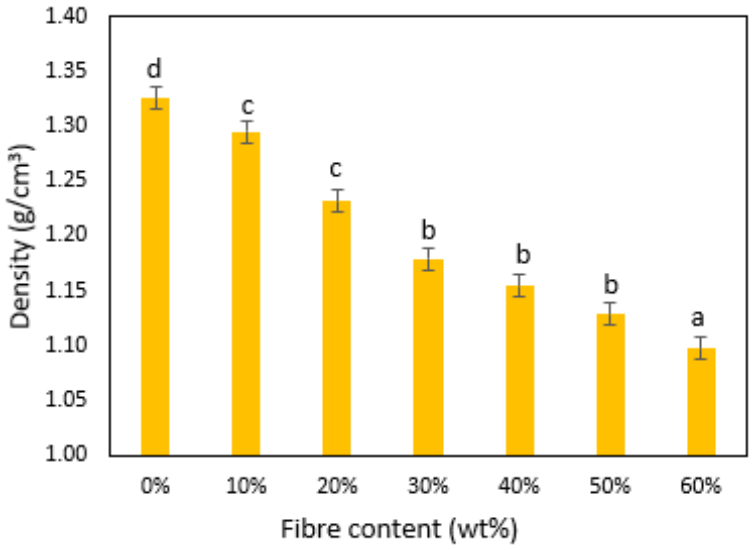


Fig. 1. Density of TPCS/CW/PPF biocomposites

Scanning Electron Microscopic Analysis

The fractured surfaces of the TPCS/CW/PPF composites were examined using SEM, and the corresponding micrographs are presented in Figs. 2a to 2g. Clear differences in surface morphology were observed between the neat matrix and the fiber-reinforced systems. The neat TPCS/CW sample exhibited a relatively smooth and continuous fracture surface, indicating a uniform structure without reinforcing phases, as shown in Fig. 2a. This compact morphology reflects effective plasticisation of cassava starch by glycerol, CW and good matrix continuity in the absence of fibers.

With the addition of PPF, the fracture behaviour changed noticeably. As shown in Figs. 2b to 2f, features such as fiber pull-out, fiber fracture, and localised interfacial debonding became evident.

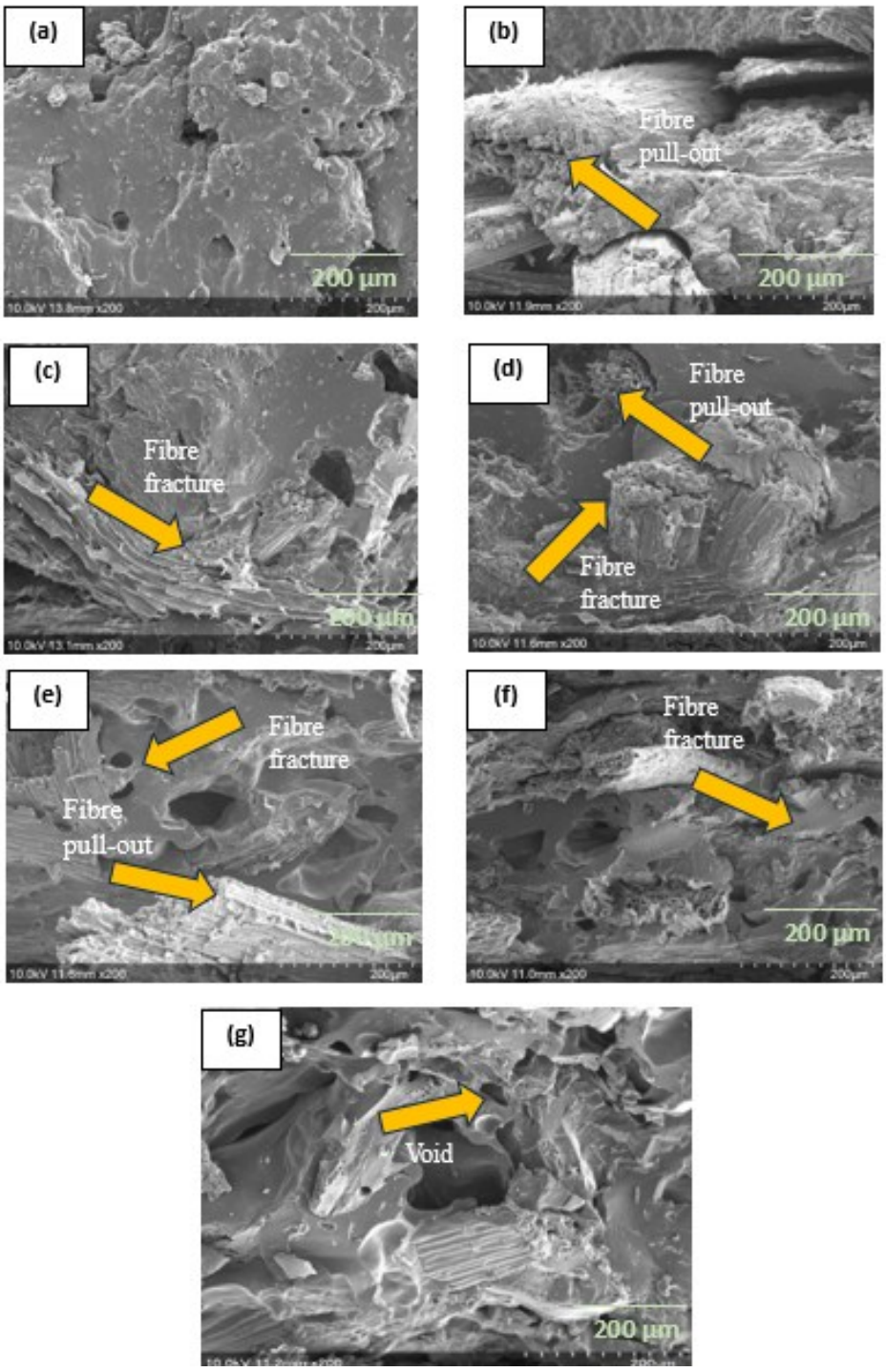


Fig. 2. SEM micrographs of the fractured surfaces of TPCS/CW composites reinforced with varying contents of *Pennisetum purpureum* fiber (PPF): (a) neat TPCS/CW, (b) TPCS/CW/PPF at 10 wt%, (c) 20 wt%, (d) 30 wt%, (e) 40 wt%, (f) 50 wt%, and (g) 60 wt%.

The presence of fractured fibers embedded within the matrix suggests that stress was successfully transferred from the starch phase to the reinforcing fibers during tensile loading. In several regions, fibers were broken rather than completely pulled out, which indicates relatively strong interfacial adhesion between PPF and the TPCS matrix. Such

behaviour is consistent with hydrogen bonding interactions between the hydroxyl groups of cellulose and starch, which enhance load transfer efficiency (Jagadeesh *et al.* 2021; Nurazzi *et al.* 2021). At higher fiber contents, particularly at 60 wt%, the fracture surfaces revealed more pronounced voids and regions of fiber agglomeration, as illustrated in Fig. 4g. The appearance of voids indicates incomplete wetting of fibers by the matrix and non-uniform dispersion at elevated loadings. These structural imperfections can act as stress concentration points, which reduce mechanical integrity despite the higher fiber fraction. Similar behaviour has been reported in starch-based biocomposites reinforced with lignocellulosic fibers, where excessive fiber incorporation disrupts matrix continuity and limits effective stress transfer (Fazeli *et al.* 2019; Ibrahim *et al.* 2020; Kamaruddin *et al.* 2023).

Overall, the SEM analysis confirms that moderate PPF loading promotes improved fiber–matrix interaction and efficient stress transfer, while excessive fiber incorporation introduces microstructural defects that compromise structural cohesion.

Moisture Content

The moisture content values of the TPCS/CW/PPF biocomposites at varying fiber loadings are shown in Fig. 3. The results indicate a gradual decrease in moisture content as the amount of PPF increased. The control sample without fiber exhibited the highest moisture content of 8.6%, whereas the composite containing 60 wt% PPF recorded the lowest value of 5.0%. This consistent downward trend demonstrates that fiber incorporation plays a significant role in reducing the overall moisture retention of the material. The reduction in moisture content can be attributed to the relative hydrophobic character of PPF compared to the TPCS/CW matrix. While lignocellulosic fibers are typically considered hydrophilic due to the abundance of hydroxyl groups in cellulose and hemicellulose, PPF also contains lignin and surface wax components that contribute to a partially hydrophobic behaviour. These constituents reduce the tendency of the composite to retain moisture. As the fiber content increases, the proportion of starch within the composite decreases, and since starch is the most hygroscopic component, the overall moisture content correspondingly declines. This behaviour is consistent with previous findings, where the incorporation of fibers rich in lignin and wax into starch-based systems resulted in reduced moisture retention (Kamaruddin *et al.* 2023). Similar observations were also documented in kenaf- and sisal-reinforced thermoplastic starch composites, where hydrophobic components within the fibers lowered the affinity of the material towards water absorption (Nurazzi *et al.* 2021). The present results therefore support the role of fiber composition in controlling the moisture behaviour of starch-based biocomposites. The present results therefore support the role of fiber composition in controlling the moisture behaviour of starch-based biocomposites.

In addition to the chemical contribution of lignin and waxy constituents, the reinforcing effect of PPF may also contribute to the reduction in moisture content. The embedded fibers can act as a structural framework within the composite, restricting matrix swelling and reducing the mobility of starch chains during moisture exposure. Such reinforcement may decrease the availability of free volume and limit water diffusion pathways within the matrix, thereby reducing moisture retention and improving dimensional stability. Similar observations have been reported in starch- and natural fiber-based composites, where effective fiber–matrix interactions contributed to reduced water uptake and swelling behaviour through physical restraint mechanisms (Ibrahim *et al.* 2020; Nurazzi *et al.* 2021; Kamaruddin *et al.* 2023).

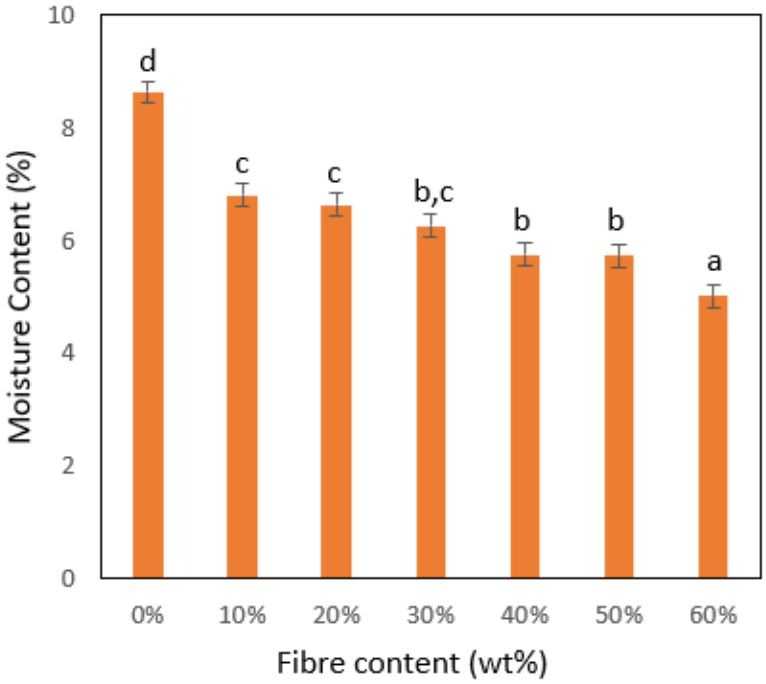


Fig. 3. Moisture content of TPCS/CW/PPF biocomposites

Water Absorption

Water absorption is a critical parameter in assessing the suitability of TPS-based materials for applications in humid or wet environments. The water absorption behaviour of the TPCS/CW/PPF biocomposites is shown in Fig. 4. A clear decreasing trend was observed as the fiber loading increased. The composite without fiber exhibited the highest water absorption value of 52.2%, whereas the sample containing 60 wt% PPF recorded the lowest value of 31.8%. This progressive reduction indicates that fiber incorporation significantly influenced the composites' resistance to water uptake. The observed decline in water absorption followed a similar pattern to that recorded for moisture content. This behaviour can be attributed to the partially hydrophobic characteristics of PPF. Although lignocellulosic fibers contain hydroxyl groups in cellulose and hemicellulose that promote hydrophilicity, PPF also contains lignin, surface waxes, and fatty substances. These constituents reduce the affinity of the composite towards water and restrict moisture penetration. Furthermore, increasing the fiber fraction reduces the relative amount of starch, which is the most water-sensitive component in the system. As a result, the overall tendency of the composite to absorb water decreased. In addition to compositional effects, the microstructural arrangement of the composite plays an important role. Higher fiber loading enhances fiber–matrix interaction, which can lead to improved interfacial bonding and a more compact internal structure. Such structural densification reduces pore connectivity and limits the pathways available for water ingress. This explanation aligns with the findings of Kamaruddin *et al*. (2023), who reported reduced water uptake in starch-based composites reinforced with hydrophobic natural fibers due to decreased porosity. Espigulé *et al*. (2013) also observed that rape fiber reinforcement restricted the swelling of starch macromolecules, thereby preventing excessive water absorption. Similarly, Nurazzi *et al*. (2021) highlighted that lignocellulosic fibers can improve water resistance through barrier effects and partial encapsulation of hydrophilic starch regions.

More recent studies further support this interpretation. It has been demonstrated that improved fiber–matrix adhesion in sugar palm fiber composites reduced micro-void formation and limited water penetration. Mohammed *et al*. (2023) emphasised that natural fibers, when properly incorporated, can function as physical barriers to moisture transport and enhance dimensional stability. Wang *et al.* (2023a) also reported that plant fibers containing lignin and wax components reduce water uptake through both chemical composition and structural barrier mechanisms. Overall, these findings confirm that the reduction in water absorption observed in TPCS/CW/PPF composites is governed by the combined effects of fiber composition and improved interfacial bonding.

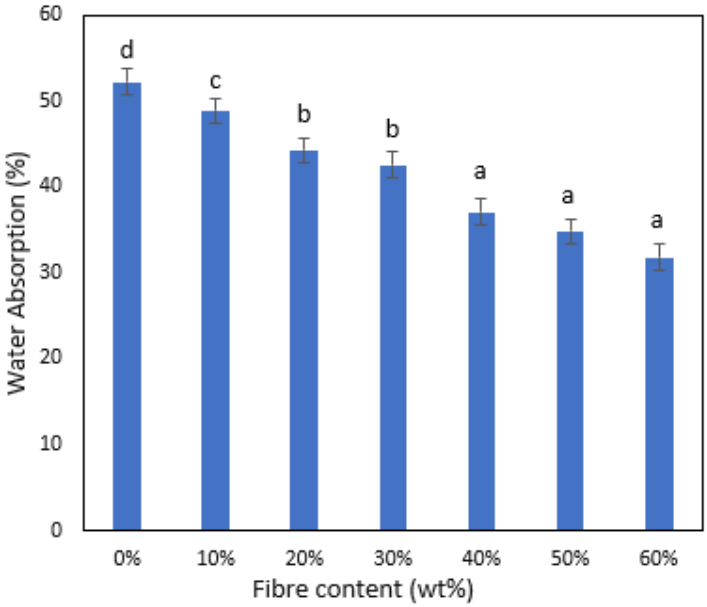


Fig. 4. Water absorption of TPCS/CW/PPF biocomposites

Thickness Swelling

The thickness swelling behaviour of the TPCS/CW/PPF composites was examined to evaluate their dimensional stability after water immersion. The results are presented in Fig. 5. A noticeable reduction in thickness swelling was recorded as the fiber content increased. The composite without fiber exhibited the highest swelling, whereas the sample containing 60 wt% PPF showed the lowest value. Overall, incorporating fiber within the investigated range resulted in an approximate 32% decrease in thickness swelling. The improvement in dimensional stability can be associated with the structural contribution of PPF within the composite matrix. Compared with starch, PPF fibers possess higher stiffness and lower hydrophilicity, which helps restrain matrix expansion when exposed to water. As fiber loading increases, the rigid fiber network provides resistance against swelling forces generated within the hydrophilic starch phase. In addition, the presence of lignin and surface wax components in PPF contributes to its relatively hydrophobic character, thereby limiting moisture diffusion and reducing volumetric expansion.

Similar behaviour has been reported in other lignocellulosic fiber systems. Ng *et al*. (2024) observed that increasing pineapple leaf fiber content in PALF/ramie composites led to reduced thickness swelling, which was attributed to the higher lignin content of the fiber and its ability to improve dimensional stability. The current findings follow a comparable trend, where the compositional characteristics of the fiber influence water interaction within the composite.

Consistent results have also been documented in starch-based composite systems. Munar *et al*. (2025) reported that incorporating lignocellulosic fibers into starch matrices significantly reduced swelling due to enhanced rigidity and improved fiber–matrix bonding. Likewise, Kamaruddin *et al.* (2023) demonstrated that *Cymbopogon citratus* fiber reduced thickness swelling and water uptake in starch composites. This was attributed to the effect to stronger interfacial adhesion and hydrophobic fiber constituents. Nurazzi *et al.* (2021) further emphasised that fibers with higher lignin content can improve dimensional stability by reducing the hygroscopic response of the composite structure.

In summary, the reduction in thickness swelling observed in TPCS/CW/PPF composites can be explained by three interrelated factors. First, the inherent stiffness and reduced hydrophilicity of PPF help limit matrix expansion. Second, lignin and waxy substances within the fiber act as barriers to moisture penetration. Third, improved fiber–matrix interaction reduces micro-void formation and restricts pathways for water ingress. These combined effects contribute to enhanced dimensional stability at higher fiber loadings.

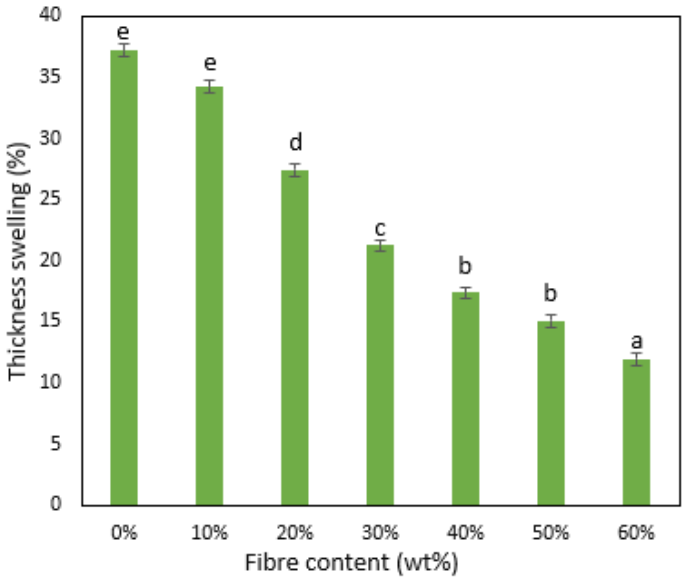


Fig. 5. Thickness swelling of TPCS/CW/PPF biocomposites

Moisture Absorption

Moisture absorption testing measures the amount of water taken up by a material when exposed to a controlled humid environment. This property is particularly important for starch-based composites intended for packaging or structural use, where resistance to moisture plays a decisive role in performance. The effect of PPF content on the moisture absorption behaviour of TPCS/CW composites over a period of 7 days is shown in Fig. 6. All samples exhibited a similar absorption profile. A substantial increase in moisture uptake was observed during the initial stage of exposure, followed by a more gradual rise until equilibrium was approached. For most compositions, the absorption rate began to slow down after approximately the third day and reached a relatively stable condition between day 5 and day 6. Because measurements were recorded at discrete time intervals, the exact time required to reach the initial stage of rapid moisture uptake could not be determined. Nevertheless, the results clearly indicate that the majority of moisture absorption occurred during the early exposure period before progressively approaching

equilibrium. This behaviour is typical of thermoplastic starch systems, where moisture diffusion is initially driven by the availability of free hydroxyl groups and accessible diffusion pathways before gradually slowing as these sites become occupied (Kamaruddin *et al.* 2022; He *et al.* 2024). After 7 days of exposure, the composite without fiber recorded the highest equilibrium moisture absorption of 8.31%. In contrast, increasing fiber loading resulted in progressively lower absorption values. The composite containing 60 wt% PPF exhibited the lowest equilibrium absorption at 3.85%. This consistent reduction indicates that fiber incorporation significantly improved the moisture resistance of the composites. Several mechanisms contribute to this improvement. First, the presence of lignin and surface wax components in PPF introduces partially hydrophobic regions within the composite, which act as barriers to moisture transport (Li *et al.* 2021). Second, higher fiber loading promotes improved fiber–matrix interaction, leading to reduced porosity and fewer continuous pathways for water diffusion (Mohammed *et al*. 2023). Third, lignin contributes to enhanced dimensional stability by limiting the swelling tendency of the starch matrix when exposed to moisture (Wang et al. 2023).

The present results are consistent with previous studies on natural fiber-reinforced starch systems. Mohammed *et al*. (2023) reported that hydrophobic fiber constituents and stronger interfacial bonding effectively reduce water uptake in polymer composites. Wang *et al*. (2023b) also highlighted the role of lignin and waxy components in restricting moisture ingress through both chemical composition and physical barrier effects. In TPS blends, Li *et al*. (2021) demonstrated that lignin-modified PBAT/TPS systems exhibited lower water absorption due to improved interfacial compatibility. Comparable behaviour was observed in starch composites reinforced with *Cymbopogon citratus* fiber, where the presence of lignin and waxes reduced moisture uptake (Kamaruddin et al. 2022). It also has been similarly reported that sugar palm fiber reinforcement lowered water absorption compared to neat starch matrices, confirming the contribution of lignocellulosic fibers to improved moisture resistance (Asyraf *et al.* 2022).

In summary, increasing PPF loading reduced the 7-day equilibrium moisture absorption from 8.31% in the control sample to 3.85% at 60 wt% fiber content. The reduction in hygroscopic behaviour can be attributed to the combined effects of hydrophobic fiber constituents, improved interfacial bonding, and reduced diffusion pathways within the composite. These findings suggest that higher PPF contents enhance the suitability of TPCS/CW biocomposites for applications in humid environments.

From a practical perspective, it is important to recognise that the objective of the present study was not to achieve the extremely low moisture absorption levels associated with conventional petroleum-based plastics, but rather to improve the environmental durability of starch-based biodegradable composites while preserving their biodegradability. Although the equilibrium moisture absorption values remained higher than those of synthetic packaging materials, the reduction from 8.31% to 3.85% represents a substantial improvement within the context of thermoplastic starch systems. Furthermore, a companion study on the same TPCS/CW/PPF formulations demonstrated significant improvements in tensile, flexural, and impact properties with increasing fiber content (Kayat *et al.* 2026). Therefore, the incorporation of PPF provided not only enhanced moisture resistance and dimensional stability, but also improved structural performance. This supports the potential use of these materials in lightweight biodegradable packaging and paper-based barrier coating applications where moderate moisture resistance and environmental sustainability are prioritised.

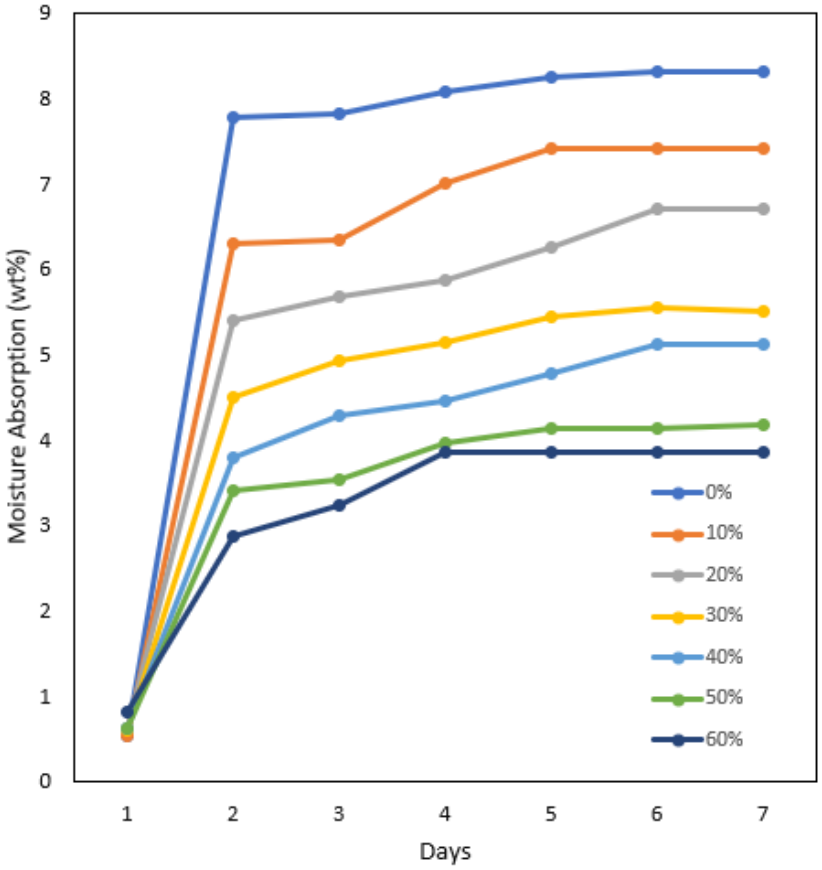


Fig. 6. Moisture absorption diagram of TPCS/CW/PPF composites

Water Solubility

Water solubility is a key parameter for determining the applicability of starch-based composites, particularly in environments where moisture exposure cannot be avoided. The water solubility values of TPCS/CW/PPF composites at different fiber loadings are shown in Fig. 7. The neat TPCS sample exhibited the highest solubility at 29.2%. With the incorporation of PPF, the solubility decreased progressively. At 10 wt% fiber loading, the value reduced to 26%, and continued fiber addition resulted in further reductions, reaching 13.11% at 60 wt%. This consistent downward trend indicates that fiber reinforcement significantly improved the resistance of the starch matrix to dissolution in water. The structural interactions between the fiber and the starch matrix can explain the reduction in solubility. As fiber loading increases, a more interconnected fiber–matrix network is formed, which limits the mobility of starch chains and reduces the accessibility of water molecules to the polymer structure. This behaviour aligns with reports that fiber reinforcement can decrease effective diffusion pathways and reduce the extent of solubilization in aqueous environments (Kamaruddin *et al.* 2023; Mohammed *et al.* 2023). Strong interfacial bonding and mechanical interlocking between fiber and matrix further restrict water penetration, as described in the interfacial bonding theory of natural fiber composites (Malkapuram *et al.* 2009; Musthaq *et al*. 2023). Similar findings have been reported in other lignocellulosic fiber-reinforced starch systems. Previous studies have observed that the incorporation of natural fibers reduced the disintegration of starch-based composites and improved their water resistance (Ibrahim *et al.* 2020; Kamaruddin *et al.*

2023). The relatively hydrophobic components present in cellulose-rich fibers also contribute to this effect by limiting moisture diffusion within the composite structure. Recent studies on modified starch systems provide further support. Choo *et al*. (2021) demonstrated that the inclusion of hydrophobic constituents in chitosan and acetylated starch films significantly reduced solubility. Previous studies have reported that hydrophobic modification strategies enhanced the barrier properties of starch-based films (Ng *et al.* 2024). It was similarly found that blending beeswax into thermoplastic starch matrices lowered both water solubility and moisture absorption (Diyana *et al*. 2021). These observations are consistent with the present findings, where increasing PPF content resulted in improved water resistance and reduced solubility of TPCS/CW/PPF composites.

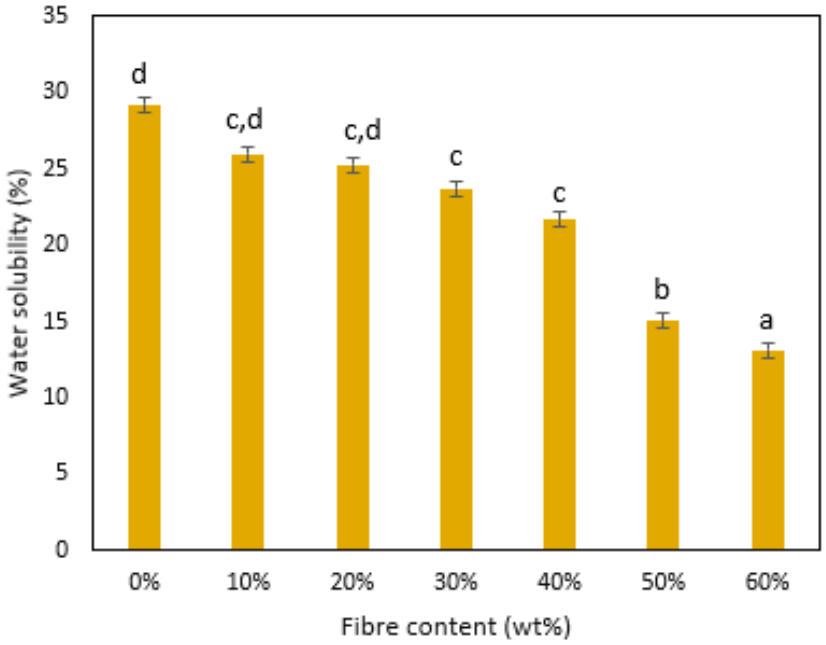


Fig. 7. Water solubility of TPCS/CW/PPF biocomposites

Soil Burial

The biodegradation behaviour of the composites was evaluated using soil burial testing, where weight loss serves as an indicator of microbial activity and moisture interaction within the material (Maran *et al.* 2014). The percentage weight loss of neat TPCS/CW and TPCS/CW/PPF composites after 2 and 4 weeks of burial is shown in Fig. 8. As expected, all samples exhibited higher weight loss with increasing burial duration, reflecting continued microbial action and sustained exposure to soil moisture.

The neat TPCS/CW sample showed substantial degradation, with weight losses of 51.6% after 2 weeks and 67.4% after 4 weeks. These high values confirm the strong susceptibility of starch-rich matrices to microbial attack because of their hydrophilic nature and ease of enzymatic breakdown. In comparison, the incorporation of PPF significantly reduced the degradation rate. At 60 wt% fiber loading, weight loss values decreased to 34.9% and 43.4% after 2 and 4 weeks, respectively. This reduction indicates that fiber reinforcement improved the resistance of the composites to biodegradation. The enhanced stability can be attributed to the presence of crystalline cellulose and lignin within PPF, which are comparatively more resistant to microbial decomposition than starch (Surendren

et al. 2022). The biodegradation trend observed in the present study is also consistent with a simplified degradation mechanism in which the thermoplastic cassava starch matrix undergoes relatively rapid biodecomposition, whereas the lignocellulosic constituents of PPF degrade at a considerably slower rate (Surendren *et al.* 2022; Su *et al.* 2018). As the fiber loading increases, a larger proportion of the composite consists of cellulose, hemicellulose, and lignin, which generally exhibit greater resistance to microbial degradation over short burial periods than starch-rich matrices. Consequently, the overall weight loss decreased with increasing fiber content because the fraction of rapidly degradable starch was progressively reduced. Although lignocellulosic components are ultimately biodegradable, their degradation typically occurs over a longer timescale than thermoplastic starch, particularly under the relatively short burial duration investigated in this study.

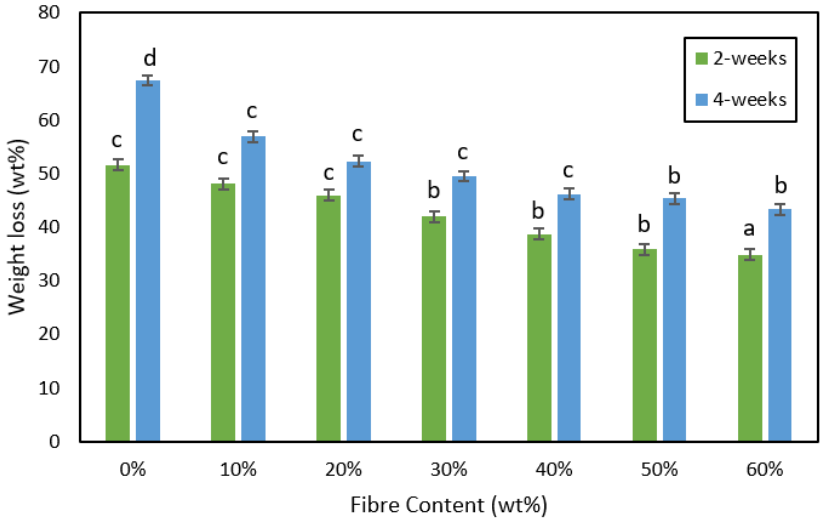


Fig. 8. Biodegradation weight loss of TPCS/CW/PPF composites after 2 and 4 weeks of soil burial

Interestingly, composites containing moderate fiber loadings, particularly at 20 wt%, exhibited relatively higher weight loss, with the TPCS/PPF-20% sample reaching 65.4% after 4 weeks. This behaviour may be associated with microstructural features such as voids and imperfect fiber–matrix interfaces that facilitate microbial penetration and accelerate degradation. It has previously been reported that cellulose-water interactions influence microbial accessibility, and loosely bonded regions within fiber-reinforced composites may promote biodegradation at intermediate fiber contents (Etale *et al.* 2023). At higher fiber loadings of 50 wt% to 60 wt%, the degradation rate decreased noticeably. The lower weight loss observed at these levels suggests that increased fiber packing enhanced fiber-matrix contact and hydrogen bonding, thereby restricting water diffusion and microbial access to the starch phase. A previous study by Su *et al*. (2018) highlighted that lignocellulosic reinforcement in starch systems can act as both a physical barrier and a stabilising component, reducing solubility and slowing biodegradation when fiber content is sufficiently high. The present findings are consistent with previous work on cassava starch composites reinforced with green coconut fibers, where fiber addition reduced degradation compared to neat starch matrices (Lomelí-Ramírez *et al*. 2014). Overall, the results demonstrate that PPF incorporation modifies the degradation behaviour of TPCS/CW composites. While intermediate fiber levels may create conditions that

facilitate microbial activity, higher fiber loadings, particularly at 60 wt%, enhance structural stability and improve durability under soil burial conditions.

CONCLUSIONS

This study examined the effect of *Pennisetum purpureum* fiber (PPF) loading on the physical performance, moisture behaviour, and biodegradation characteristics of thermoplastic cassava starch/candelilla wax (TPCS/CW) composites.

1. The incorporation of fiber from 0 to 60 wt% resulted in a progressive reduction in density, supporting the development of lightweight biodegradable materials.
2. Moisture-related properties showed clear improvement with increasing fiber content. Equilibrium moisture absorption after seven days decreased from 8.3% in the neat composite to 3.8% at 60 wt% fiber loading, while water solubility was reduced from 29.2% to 13.1%.
3. Thickness swelling and water absorption also declined consistently as fiber content increased. These improvements are associated with the presence of lignin and surface wax components in the fiber and with enhanced fiber–matrix interaction that limits water diffusion and pore connectivity.
4. Soil burial testing confirmed that all formulations remained biodegradable. However, fiber incorporation moderated the degradation rate. After four weeks, weight loss decreased from 67.4% in the neat matrix to 43.4% at 60 wt% fiber loading. Slightly higher degradation at intermediate fiber contents suggests that interfacial gaps may facilitate microbial access under certain compositions. At higher fiber loadings, improved structural integrity reduced moisture penetration and slowed degradation.
5. Overall, increasing PPF content enhanced dimensional stability and moisture resistance while maintaining the biodegradable nature of the TPCS/CW composites. Fiber loadings in the range of 50 to 60 wt% provide a favourable balance between durability and environmental degradability, indicating strong potential for sustainable packaging and applications exposed to humid conditions.
6. Based on the observed improvements in moisture resistance, dimensional stability, reduced water solubility, and maintained biodegradability, the developed TPCS/CW/PPF composites show potential for biodegradable packaging films, disposable packaging liners, and coating layers for paper-based packaging materials where moderate moisture resistance and environmental sustainability are required.

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