PREPARATION AND CHARACTERIZATION OF SAGO WASTE-DERIVED CELLULOSE/PVA BIOFILMS FOR SUSTAINABLE PACKAGING APPLICATIONS

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Sago waste-derived cellulose (SWC)/polyvinyl alcohol (PVA) biofilms were prepared via solution casting for potential biodegradable packaging applications. Sago waste fibers were treated with different sodium hydroxide (NaOH) concentrations (1-3 M) to improve cellulose exposure and interfacial adhesion within the biofilm matrix. The effects of alkali treatment, SWC loading, and glycerol content on the structural, thermal, moisture absorption, and mechanical properties of the biofilms were investigated using SEM, FTIR, TGA, and tensile analyses. SEM and FTIR results confirmed the reduction in hemicellulose and lignin contents after alkali treatment, resulting in rougher fiber surfaces and improved cellulose characteristics. The incorporation of glycerol increased the flexibility and moisture absorption capacity of the films, while excessive glycerol reduced thermal stability. Among the prepared samples, the 2M NaOH-treated SWC biofilm exhibited the best mechanical performance, with a tensile strength of 16.66 MPa, due to improved fiber–matrix interaction. The results demonstrate that alkali-treated sago waste/PVA biofilms have promising potential as sustainable biodegradable materials for food packaging applications.

Keywords: sago waste, biofilm, preparation, characterization

INTRODUCTION

Petroleum-based plastics are widely used in packaging applications due to their excellent mechanical strength, lightweight nature, low production cost, and good heat-sealing properties. However, most conventional plastics are derived from non-renewable petrochemical resources and exhibit poor biodegradability, resulting in severe environmental pollution and long-term accumulation in landfills and aquatic environments.[1,2] The continuous increase in plastic consumption, combined with their slow natural degradation rate, has intensified global concerns regarding sustainable waste management and environmental preservation.

To address these environmental issues, increasing research efforts have focused on the development of biodegradable polymers derived from renewable biomass resources.[2] Biodegradable plastics, commonly known as bioplastics, can be produced from natural materials, such as starch, cellulose, lignin, proteins, and other bio-based compounds.[3] Compared to conventional petroleum-based plastics, biodegradable materials can undergo microbial degradation under suitable environmental conditions, thereby reducing long-term plastic accumulation and environmental impact. Owing to their biodegradability, renewability, and eco-friendly characteristics, bioplastics have emerged as promising alternatives for sustainable packaging applications.

Among various renewable materials, lignocellulosic biomass has attracted considerable attention as a potential raw material for biodegradable film development due to its abundance and cellulose-rich composition.[4] Sago waste, locally known as *sago hampas*, is a solid fibrous residue generated during starch extraction from the pith of the sago palm (*Metroxylon sagu*). Following starch extraction, a substantial amount of residual fibrous material, containing lignocellulosic components including cellulose,

hemicelluloses, and lignin, remains. The cellulose in this residue offers an opportunity to use it as a renewable raw material for bio-based material development rather than disposing of it as agricultural processing waste. However, the presence of non-cellulosic components and the hydrophilic nature of the cellulose-rich material may limit its direct application in film formulations.[5,6]

Despite their environmental advantages, pure bio-based films generally exhibit poor mechanical strength, brittleness, and high moisture sensitivity, which restrict their practical applications in food packaging materials.[5] In addition, cellulose-based films often possess limited barrier and flexibility properties due to the hydrophilic nature of cellulose.[6] Therefore, chemical modification and plasticizer incorporation are commonly employed to improve the structural and mechanical performance of biofilms. Alkali treatment is widely used to remove hemicelluloses, lignin, and surface impurities from natural fibers, thereby enhancing cellulose exposure and improving fiber–matrix interaction. Furthermore, plasticizers, such as glycerol, sorbitol, and polyethylene glycol, are frequently incorporated to increase film flexibility and reduce brittleness. Among these plasticizers, glycerol is widely used due to its compatibility, non-toxicity, and effectiveness in improving the flexibility and transparency of biofilms.[5]

Therefore, this study aimed to prepare and characterize sago waste-derived cellulose (SWC)/polyvinyl alcohol (PVA) biofilms for potential biodegradable food packaging applications. The effects of sodium hydroxide (NaOH) treatment, SWC loading, and glycerol content on the structural, thermal, moisture absorption, and mechanical properties of the biofilms were investigated using SEM, FTIR, TGA, and tensile analyses.

EXPERIMENTAL

Materials

Sago waste was collected from a local factory in Pusa, Sarawak, Malaysia. The waste was collected and dried in an oven at 70 °C for 20 hours. The dried waste was cleaned, washed thoroughly, and rinsed 3–5 times with distilled water to remove starch. The rinsed sago fibers were then dried again in an oven at 70 °C for 20 hours and stored in a zip-and-lock bag for further use. Standard analytical grade chemicals, including sodium hydroxide (NaOH, Merck & Co.), glycerol ($C_3H_8O_3$, HmBG), copper (II) sulphate ($CuSO_4$, R&M Chemicals), which was used to establish the controlled humid environment for the moisture absorption testing, and polyvinyl alcohol (PVA, Aldrich Resources Berhad), were purchased from local chemical suppliers and used without modification. The standard solutions were prepared using distilled water.

Pretreatment of samples

The sago waste was pretreated using physical and chemical treatment before preparing the biofilm. The ground sago waste was soaked in three different concentrations of 1, 2, and 3 M NaOH solution for 20 minutes. The soaked sago waste was then rinsed with distilled water until a neutral pH of 7 ± 0.5 was achieved. The sago waste-derived cellulose (SWC) was dried at 60 °C for 20 hours and ground. The ground product was then sieved through a 200 µm mesh laboratory sieve and kept for further use.

Preparation of biofilms

The biofilms were prepared based on the methods reported by Sondari *et al*.,[4] with a few modifications. The sago waste biofilms were produced using the solution casting method. PVA solution was prepared by dissolving the PVA pellets (2.75, 3.00, and 3.25 g) in heated distilled water at 90 °C and continuously vigorously stirring to avoid the formation of clumps until the PVA pellets were completely dissolved. The PVA solution was then allowed to cool to 60 °C before introducing the desired SWC (0.25, 0.50, and 0.75 g) and glycerol (0.0, 1.5, and 3.0 mL) contents. The biofilms were prepared based on the formulations listed in Table 1.

In the sample denotations, N represents the NaOH concentration (M), P represents the SWC content (g), and G represents the glycerol content (mL). The mixture was stirred continuously for 20 minutes to obtain a uniform dispersion of SWC in the PVA solution. Then, 20 mL of the mixture was poured into a lubricant-sprayed 8.5 cm Petri dish using a dropper. The thickness of the film solution was measured to ensure uniform thickness.

The Petri dishes were left at room temperature for 2 hours to allow stabilization and bleeding of trapped bubbles before drying in an oven at 40 °C for 7 hours. The dried film was then equilibrated in a desiccator at room temperature before being kept for further use.

Characterization

The surface morphology of untreated and alkali-treated sago waste samples was analyzed using scanning electron microscopy (SEM, JEOL JSM-6390LA) at an accelerating voltage of 15 kV. Prior to analysis, the samples were sputter-coated with gold/palladium and observed under 100× and 600× magnifications.

FTIR spectra were recorded using a Shimadzu IRAffinity-1S spectrometer operating in ATR mode within the range of 4000-400 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans.

Thermal stability of the SWC biofilms was evaluated using thermogravimetric analysis (TGA, Shimadzu DTG-60H) under nitrogen atmosphere from

50 to 500 °C, at a heating rate of 10 °C min^{-1}. Moisture absorption tests were conducted under 98% relative humidity in a desiccator containing $CuSO_4$, and the moisture absorption percentage was calculated using Equation (1):

$$Moisture\ absorption = \frac{Soaked\ biofilm\ weight - Initial\ biofilm\ weight}{Initial\ biofilm\ sample\ weight} \times 100\% \quad (1)$$

Mechanical properties were determined using a universal testing machine (Shimadzu AGS-X) according to ASTM D638 Type V.

Table 1
Composition of SWC biofilms

PVA content (g)	NaOH concentration of SWC, N (M)	SWC content, P (g)	Glycerol content, G (mL)	Sample denotation
3.25	1	0.25	0.0	1N0.25P0.0G
			1.5	1N0.25P1.5G
			3.0	1N0.25P3.0G
3.00		0.50	0.0	1N0.5P0.0G
			1.5	1N0.5P1.5G
			3.0	1N0.5P3.0G
2.75		0.75	0.0	1N0.75P0.0G
			1.5	1N0.75P1.5G
			3.0	1N0.75P3.0G
3.25	2	0.25	0.0	2N0.25P0.0G
			1.5	2N0.25P1.5G
			3.0	2N0.25P3.0G
3.00		0.50	0.0	2N0.5P0.0G
			1.5	2N0.5P1.5G
			3.0	2N0.5P3.0G
2.75		0.75	0.0	2N0.75P0.0G
			1.5	2N0.75P1.5G
			3.0	2N0.75P3.0G
3.25	3	0.25	0.0	3N0.25P0.0G
			1.5	3N0.25P1.5G
			3.0	3N0.25P3.0G
3.00		0.50	0.0	3N0.5P0.0G
			1.5	3N0.5P1.5G
			3.0	3N0.5P3.0G
2.75		0.75	0.0	3N0.75P0.0G
			1.5	3N0.75P1.5G
			3.0	3N0.75P3.0G

RESULTS AND DISCUSSION

Morphological analysis

SEM micrographs of untreated and alkali-treated sago waste samples are presented in Figure 1. The untreated sago waste exhibited irregularly arranged fibers with the presence of residual starch granules and surface impurities. After alkali treatment, the fiber surfaces became rougher and more organized due to the partial removal of hemicelluloses, lignin, and other non-cellulosic components. The increased surface roughness can be beneficial in enhancing the interfacial interaction between the fibers and the polymer matrix. Among the treated samples, the 2 M NaOH-treated fibers exhibited a more uniform and well-defined cellulose structure compared to the 1 M-treated samples, indicating effective delignification without severe fiber damage. In contrast, the 3 M NaOH-treated fibers showed excessively rough and partially damaged surfaces, suggesting that excessive alkali concentration may deteriorate the fiber structure. The improved surface morphology observed in the 2 M NaOH-treated samples is consistent with the enhanced mechanical properties of the corresponding biofilms. Similar morphological changes following chemical treatment have been reported for cellulose derived from other agricultural residues. Ng *et al.*, for example, observed similar

changes in the surface morphology of orange-peel-derived cellulose following alkaline treatment and subsequent bleaching.[23]

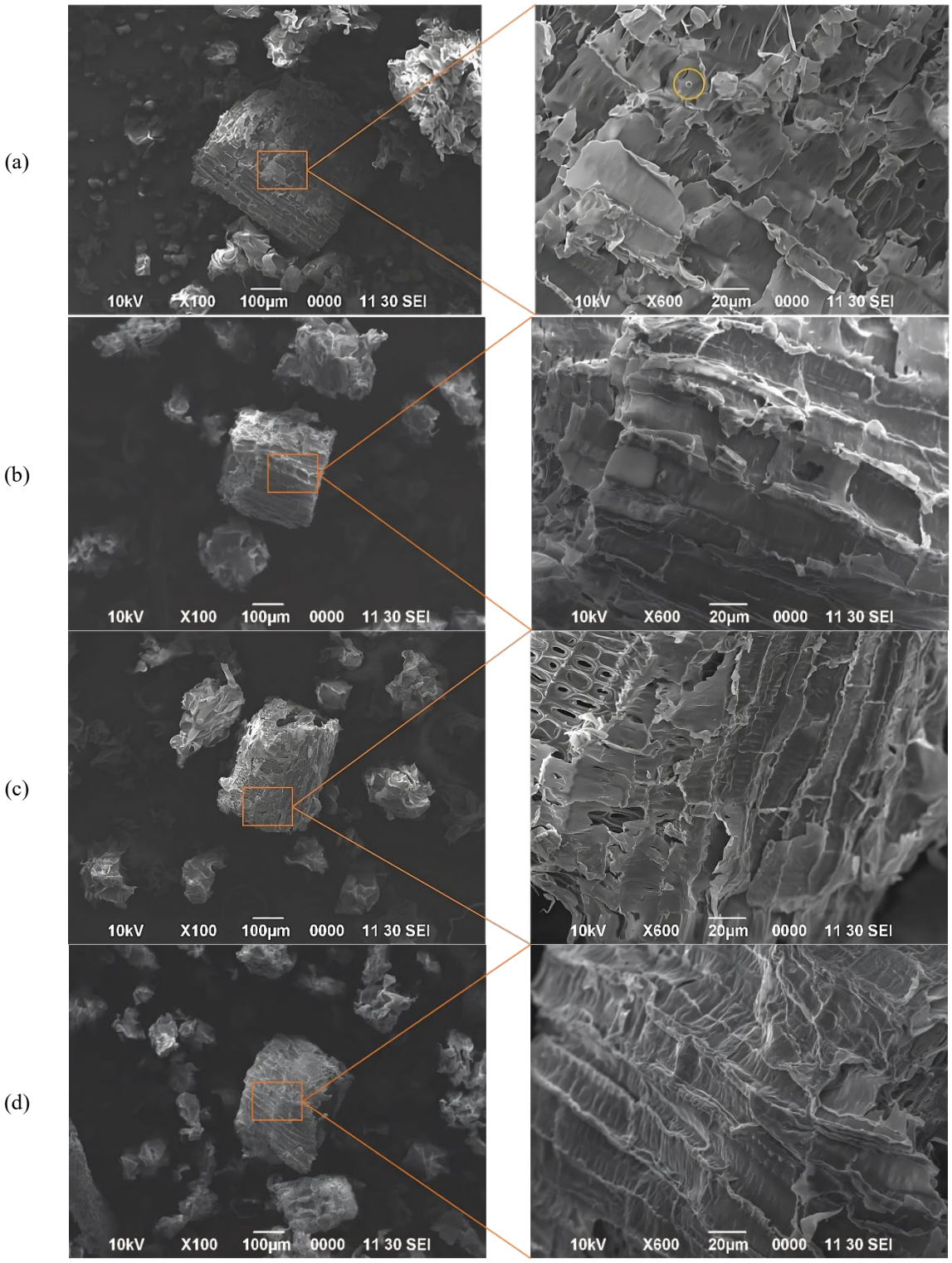


Figure 1: SEM images at 100× and 600× magnification of (a) untreated sago waste, (b) 1 M NaOH-treated, (c) 2 M NaOH-treated, and (d) 3 M NaOH-treated sago waste samples

Fourier-transform infrared (FTIR) spectroscopy

FTIR spectra of untreated and alkali-treated sago waste samples are presented in Figure 2. All samples exhibited a broad absorption peak around 3337 cm^{-1}, corresponding to the O-H stretching vibration of cellulose associated with intermolecular and intramolecular hydrogen bonding.[7] The untreated sago waste showed characteristic peaks at 1730 cm^{-1} and 1601 cm^{-1}, which are attributed to the carbonyl groups of hemicelluloses and aromatic structures of lignin, respectively.[8] After alkali treatment, the disappearance of these peaks indicated the partial removal of hemicelluloses and lignin from the fiber surface. In addition, the reduction of peaks associated with lignin-related aromatic groups confirmed the delignification effect of NaOH treatment. The appearance of the peak at 1327 cm^{-1} further suggested structural modification of the lignocellulosic components after alkali treatment.[9] Comparable spectral changes have been reported by Ng *et al.* during the isolation of cellulose from orange peel through alkaline treatment followed by bleaching, where FTIR was used to characterise the cellulose-rich material obtained after chemical treatment.[23] These spectral changes demonstrate that alkali treatment effectively enhanced cellulose exposure in the sago waste fibers.

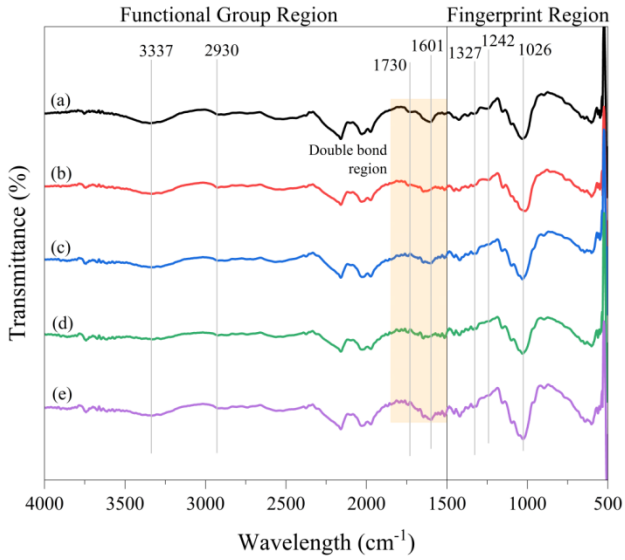


Figure 2: FTIR-ATR spectra of (a) ground sago waste, (b) rinsed sago waste, (c) 1 M NaOH-treated sago waste, (d) 2 M NaOH-treated sago waste, and (e) 3 M NaOH-treated sago waste

Thermogravimetric analysis (TGA)

The thermal stability of the SWC biofilms with different glycerol contents was evaluated using thermogravimetric analysis (TGA), as presented in Figure 3. The biofilms without glycerol exhibited three major stages of thermal degradation. The initial weight loss observed below 200 °C was mainly attributed to moisture evaporation and the removal of physically absorbed water molecules from the hydrophilic cellulose structure.[10] The second degradation stage occurring between 225 and 350 °C corresponded to the decomposition of cellulose, hemicellulose residues, and low molecular weight organic compounds within the biofilm matrix. The final degradation stage above 350 °C was associated with the decomposition of carbonaceous residues and further degradation of the polymeric structure.[11]

In contrast, the biofilms containing glycerol exhibited an additional degradation stage between 125 and 290 °C, which was attributed to glycerol decomposition and volatilization.[12] The incorporation of glycerol reduced the thermal resistance of the biofilms due to its plasticizing effect, which increased the mobility of polymer chains and weakened intermolecular hydrogen bonding within the cellulose-PVA matrix. As the glycerol content increased, the thermal degradation of the biofilms became more pronounced, resulting in greater weight loss at lower temperatures. Similar thermal behavior has been reported for glycerol-plasticized cellulose-based films, where the presence of glycerol accelerated thermal decomposition due to reduced intermolecular interactions between polymer chains.[13] Similar effects of formulation and intermolecular

interactions on the thermal behaviour of cellulose/PVA materials have been reported in the literature. Niu *et al.* demonstrated that changes in cellulose dispersion and crosslinking within a PVA/cellulose system affected its thermal stability, highlighting the importance of interactions between the cellulose and polymer phases in determining the degradation behaviour.[24]

Among the prepared samples, the 2N0.75P0.0G biofilm exhibited the highest residual weight compared to the glycerol-containing samples, indicating relatively better thermal stability in the absence of plasticizer. The residual weights of 2N0.75P0.0G, 2N0.75P1.5G, and 2N0.75P3.0G were 27.2%, 16.7%, and 15.2%, respectively, demonstrating that higher glycerol content decreased the thermal stability of the biofilms. Nevertheless, the overall degradation profile associated with cellulose decomposition remained relatively unchanged, suggesting that glycerol mainly affected the low-temperature degradation behavior without significantly altering the primary cellulose degradation mechanism. Overall, the TGA profiles indicate that glycerol addition modified the thermal behavior of the SWC/PVA biofilms, particularly within the lower-temperature degradation region.

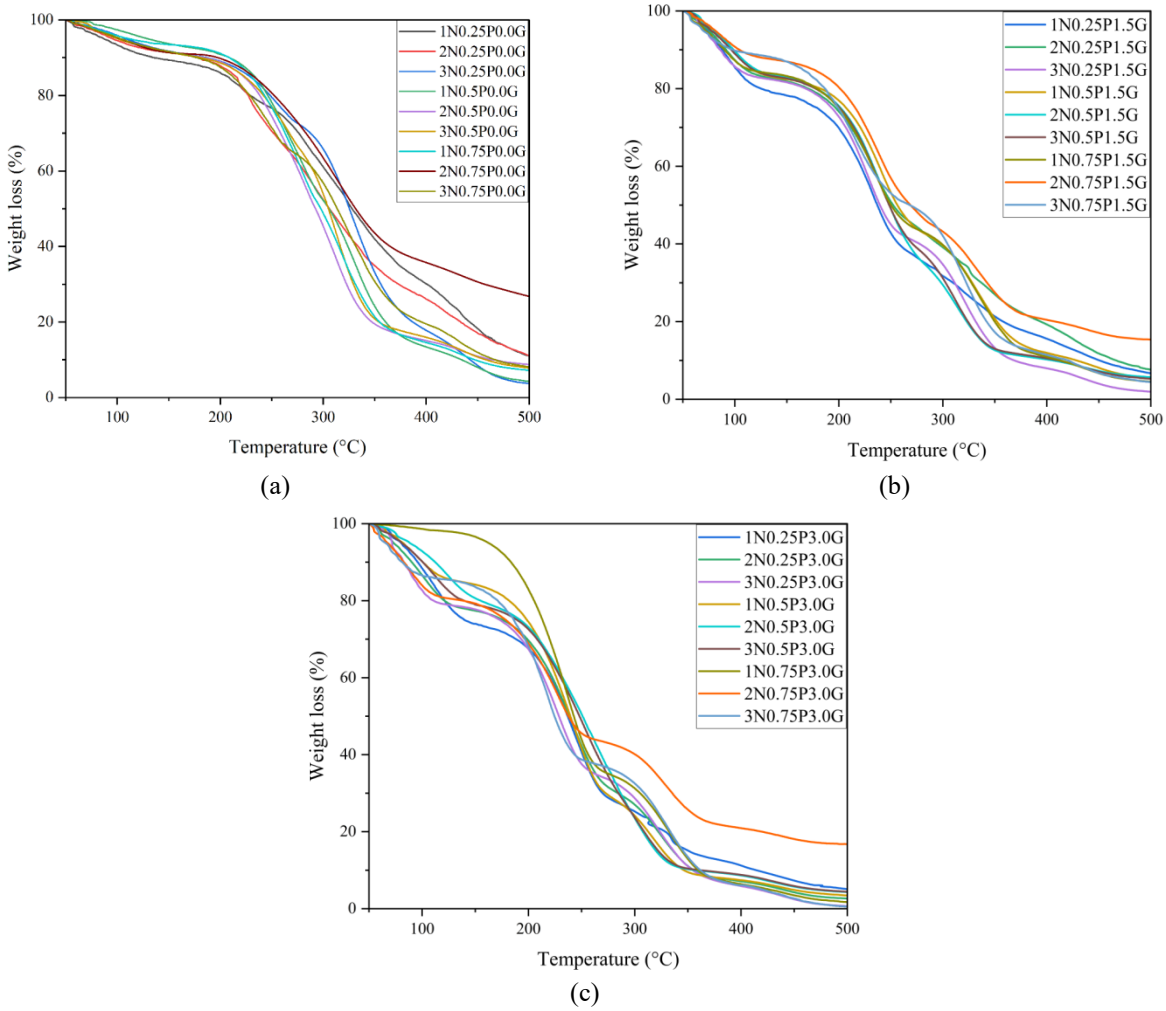


Figure 3: TGA curve comparison of SWC biofilms at (a) 0.0 mL, (b) 1.5 mL, and (c) 3.0 mL glycerol content

Moisture absorption test

The moisture absorption behavior of the SWC biofilms with different glycerol contents is presented in Figure 4. The moisture absorption capacity of the biofilms was strongly influenced by the glycerol concentration due to the hydrophilic nature of both cellulose and glycerol. Among the samples without glycerol, the 2N0.75P0.0G biofilm exhibited the highest moisture absorption of 10.63%, which may be attributed to the porous cellulose structure and increased availability of hydroxyl groups capable of interacting with water molecules. The alkali treatment enhanced cellulose exposure by removing hemicelluloses and lignin,

resulting in a rougher and more hydrophilic fiber surface that promoted water uptake.

The incorporation of glycerol increased the moisture absorption of the biofilms. The 2N0.75P1.5G sample exhibited the highest moisture absorption value of 19.20%, representing an increase of approximately 8.57% compared to the non-plasticized sample. This behavior can be attributed to the hygroscopic nature of glycerol, which contains multiple hydroxyl groups capable of forming hydrogen bonds with water molecules.[14] Similar observations have been reported for glycerol-plasticized cellulose-based films, where the presence of glycerol increased water affinity and moisture uptake due to enhanced polymer chain mobility and hydrophilicity.[15]

At higher glycerol content, the 2N0.75P3.0G biofilm exhibited a moisture absorption value of 13.33%. Although glycerol generally promotes water absorption, excessive glycerol incorporation may alter the structural arrangement of the polymer matrix and reduce the availability of free hydrophilic sites for water interaction. Previous studies have also reported that glycerol-plasticized cellulose films exhibit enhanced moisture handling properties due to the high water retention capability of glycerol and the hydrophilic characteristics of cellulose.[16,17] Xie *et al.* evaluated water resistance and barrier-related behaviour in bagasse cellulose/PVA films, demonstrating the importance of controlling interactions between cellulose-based films and moisture when considering packaging applications.[25] Overall, glycerol incorporation increased the hydrophilic behavior and moisture absorption of the SWC/PVA biofilms. Although glycerol contributes to film flexibility, the increased moisture affinity may limit the moisture resistance of the biofilms, particularly for packaging applications requiring protection against environmental moisture.

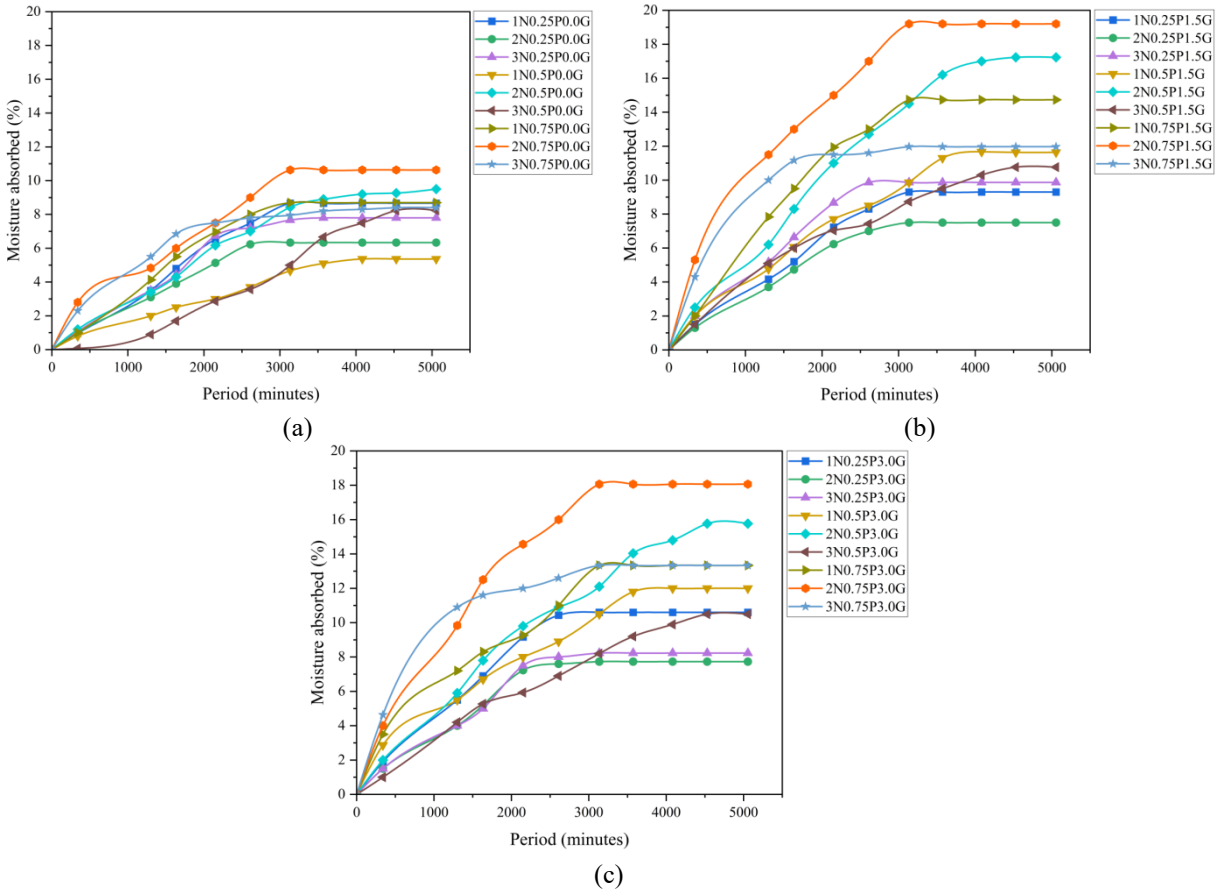


Figure 4: Moisture absorption test data of SWC biofilms at (a) 0.0 mL, (b) 1.5 mL, and (c) 3.0 mL glycerol content

Mechanical properties

The mechanical properties of the SWC biofilms are summarized in Table 2 in terms of ultimate tensile stress, yield strength, elongation at break, and Young's modulus. The mechanical behavior of the biofilms was significantly influenced by both alkali treatment and glycerol content. Among the samples without glycerol, the 2N0.5P0.0G biofilm exhibited the highest tensile strength of 16.66 MPa, together with relatively high Young's modulus and yield strength values of 157.98 MPa and 3.94 MPa, respectively. The improved mechanical performance can be attributed to the effective alkali treatment at 2 M NaOH concentration, which enhanced cellulose exposure and increased surface roughness through the partial removal of lignin and hemicelluloses. The roughened fiber surface promoted stronger interfacial adhesion and better stress transfer between the cellulose fibers and the PVA matrix.[18,19] However, the elongation at break of this sample was relatively low (38.37%), indicating a more rigid and brittle structure due to the absence of glycerol plasticizer.

Table 2
Mechanical properties of SWC biofilm samples

Sample	Ultimate tensile stress (MPa)	Yield strength (MPa)	Elongation at break (%)	Young's modulus (MPa)
1N0.25P0.0G	12.14	3.07	96.32	35.74
2N0.25P0.0G	9.61	4.63	118.15	25.19
3N0.25P0.0G	11.92	3.07	106.16	29.61
1N0.5P0.0G	10.03	2.79	69.63	46.81
2N0.5P0.0G	16.66	3.94	38.37	157.98
3N0.5P0.0G	15.03	3.15	62.17	127.57
1N0.75P0.0G	0.84	0.23	7.74	17.08
2N0.75P0.0G	7.17	1.58	27.40	87.41
3N0.75P0.0G	8.13	1.71	59.78	54.26
1N0.25P1.5G	4.06	1.36	142.99	8.26
2N0.25P1.5G	7.18	2.01	279.25	9.41
3N0.25P1.5G	7.43	1.77	258.98	9.42
1N0.5P1.5G	10.20	1.67	115.14	9.87
2N0.5P1.5G	9.26	2.52	160.62	46.16
3N0.5P1.5G	8.61	1.52	139.82	9.20
1N0.75P1.5G	2.64	0.52	54.85	9.83
2N0.75P1.5G	1.41	0.46	38.47	7.12
3N0.75P1.5G	2.50	0.71	56.94	8.19
1N0.25P3.0G	4.46	1.07	260.59	4.05
2N0.25P3.0G	4.14	1.21	267.24	4.64
3N0.25P3.0G	8.80	1.72	360.68	7.53
1N0.5P3.0G	6.00	1.34	217.83	5.36
2N0.5P3.0G	8.26	1.98	235.12	16.32
3N0.5P3.0G	4.22	1.28	197.12	5.39
1N0.75P3.0G	2.49	0.96	184.20	4.45
2N0.75P3.0G	1.75	0.70	109.19	4.77
3N0.75P3.0G	2.84	0.58	135.72	5.13

The incorporation of glycerol significantly affected the flexibility of the biofilms. At 1.5 mL glycerol content, the 2N0.25P1.5G biofilm exhibited the highest elongation at break of 279.25%, indicating improved flexibility and ductility of the polymer matrix. Nevertheless, the tensile strength, yield strength, and Young's modulus decreased to 7.18 MPa, 2.01 MPa, and 9.41 MPa, respectively. This reduction in mechanical strength may be attributed to the plasticizing effect of glycerol, which disrupted intermolecular hydrogen bonding within the cellulose-PVA matrix and increased polymer chain mobility.[20] Similar behavior has been reported in glycerol-plasticized cellulose films, where increased flexibility was accompanied by reduced tensile strength and stiffness.[21] Niu *et al*., for example, reported tensile strength and elongation

at break values of up to 18.2 MPa and 361.7%, respectively, for modified PVA/cellulose-fibre biocomposites.[24] Although these values are within a broadly comparable range to some formulations in the present study, direct comparison should be made cautiously because their cellulose fibres underwent mechano-chemical treatment and the composite system involved succinic-anhydride crosslinking.

A similar trend was observed for the biofilms containing 3.0 mL glycerol. The 3N0.25P3.0G sample exhibited the highest elongation at break of 360.68%, demonstrating that increasing glycerol concentration further enhanced the flexibility of the biofilms. The presence of additional glycerol molecules reduced intermolecular interactions between polymer chains and increased free volume within the matrix, resulting in a more flexible and less ordered structure.[22] However, excessive glycerol incorporation reduced the tensile strength, yield strength, and Young's modulus to 8.80 MPa, 1.72 MPa, and 7.53 MPa, respectively, indicating lower resistance toward mechanical deformation. Similar observations have been reported for glycerol-plasticized bacterial cellulose films, where increasing glycerol concentration improved elongation properties but reduced tensile strength due to weakened hydrogen bonding interactions within the polymer network.[22] Overall, the results demonstrate that glycerol improved the flexibility and elongation behavior of the SWC biofilms at the expense of tensile strength and rigidity.

CONCLUSION

In this study, sago waste-derived cellulose (SWC)/PVA biofilms were successfully prepared using the solution casting method for potential biodegradable packaging applications. SEM analysis revealed that alkali treatment improved the surface morphology of the fibers through partial removal of lignin and hemicelluloses, resulting in rougher and more organized cellulose structures that enhanced fiber–matrix interaction. FTIR analysis further confirmed the effectiveness of alkali treatment through the disappearance of characteristic lignin and hemicellulose peaks. Thermal analysis demonstrated that glycerol incorporation reduced the thermal stability of the biofilms by increasing mass loss during degradation, while moisture absorption tests showed that higher glycerol content increased the hydrophilic behavior of the films. Mechanical analysis indicated that biofilms without glycerol exhibited higher tensile strength and rigidity, whereas glycerol-plasticized biofilms showed significantly improved flexibility and elongation at break due to the plasticizing effect of glycerol. Among the prepared samples, the 2 M NaOH-treated SWC biofilms exhibited balanced structural and mechanical properties, indicating effective alkali treatment without severe fiber damage. Overall, the obtained results demonstrate that SWC/PVA biofilms possess promising potential as sustainable and biodegradable materials for food packaging applications through the valorization of agricultural biomass waste. For future work, further optimization of the SWC biofilms should be conducted by investigating the effects of SWC loading, glycerol concentration, and alkali treatment conditions on the mechanical, thermal, moisture barrier, and biodegradation properties of the biofilms.

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