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Effect of Glycerol Plasticization on the Mechanical and Physical Properties of PLA/Tapioca Starch Biofilm

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Abstract

Starch-based polymers such as polylactic acid (PLA) are increasingly used in biomedical devices, medical tools and packaging due to their biodegradability, biocompatibility and good processability. In this study, biofilms were prepared using PLA, tapioca starch and glycerol. A PLA-to-starch ratio of 70:30 (w/w) was maintained with glycerol added at concentrations of 4, 8, 12, 16 and 20 ml. Mechanical testing showed that the 4 ml glycerol film had the highest tensile strength, while higher glycerol levels improved flexibility. Soil burial tests revealed that the 12 ml glycerol sample degraded completely within 28 days. Water solubility and swelling increased with glycerol content due to its hydrophilic nature. FTIR analysis confirmed hydrogen bonding between PLA, Tapioca starch and glycerol, with peak shifts in hydroxyl and carbonyl regions. SEM images indicated smoother and more uniform surfaces at higher glycerol concentrations, suggesting improved compatibility. TGA results showed a slight reduction in thermal stability with increased glycerol, attributed to greater chain mobility. These findings demonstrate that glycerol content significantly influences the mechanical, thermal and biodegradation properties of PLA–starch biofilms.

Keywords: Poly Lactic Acid, Tapioca Starch, Biodegradation, Tensile Strength, Medical Gadgets, Biofilm

1. Introduction

The advancement of polymers derived from naturally occurring substances has been centered on the rise in carbon dioxide resulting from the combustion of fossil fuels. Polylactic acid (PLA) is a naturally occurring polymer substance with the potential to replace petroleum-based polymers. PLA is an eco-friendly and biocompatible polymer synthesized from renewable biological sources and it naturally degrades over time [1]. Lactic acid is derived from sugar, starch, cellulose and glycerin can be used to make this substance [2]. Lactic acid stereoisomers L and D can be combined to create PLLA (poly (L-lactic acid)) and PDLA (poly (D-lactic acid)) [3].In addition to its positive qualities, PLA has drawbacks such as excessive hydrophobicity, brittleness and poor ductility [4], [5]. Through mixing techniques with other polymers, PLA can be altered to enhance its mechanical qualities. Without the need to create new polymers, this approach is regarded as efficient, straightforward and adaptable for creating new materials [6]. Many polymers are employed including starch, chitosan, polybutylene adipate coterephthalate (PBAT), polycaprolactone (PCL), polyethylene glycol (PEG) and polyhydroxybutyrate (PHB) [7]. Flour and tapioca root embryo are separated to make tapioca starch. One million tons of tapioca root yields half a ton of tapioca starch. Since tapioca starch is now primarily utilized for animal feed, its application is rated as low value [8]. Dry or wet basis 55 to 60% non-starch carbohydrate, 14 to 25% starch, 13 to 18% protein, 3 to 8% minerals and 3 to 4% fat make up the composition of tapioca starch [9]. 52 to 70% of all non-starch carbohydrates are made up of arabinoxylan. A polysaccharide group called arabinoxylan can be used to create films with excellent lubricating and strength [10]. In addition, arabinoxylan works well as a hemicellulose when plasticizers are added [11]. Biodegradable polymers derived from renewable resources are PLA and tapioca starch. Given that tapioca starch is a readily available and reasonably priced biomaterial, combining PLA with it could be one of the more promising approaches. The PLA/TS blend's tapioca starch is utilized to boost PLA's strength, hydrophilicity and flexibility. Melt-blending and solution-blending are two methods for creating PLA/TS blend polymers [4]. Blending PLA with tapioca starch enhances the mechanical behavior of the material by increasing its elongation at break and reducing tensile strength, which contributes to improved flexibility in the composite [12]. From the literature, it is identified that the PLA, with orange peel, wheat bran, etc., are fabricated and tested but this combination of Tapioca starch, glycerol and fabricated biofilms has not been studied. This study focuses on the effects of the combination of PLA+ Tapioca Starch + Glycerol inclusion is varied to fabricate and test.

2. Process Description

2.1 Materials

The following materials of polylactic acid (PLA), tapioca starch and glycerol were selected as the primary materials for developing biodegradable films. PLA in pellet form was sourced from Kusbutha Plastics Granules Manufacturer, Erode, Tamil Nadu. This biodegradable polymer possesses a density of about 1.24 g/cm^3 and exhibits a melt flow index between 6 and 10 g/10 min at 210 °C under a 2.16 kg load. Food-grade tapioca starch, procured from SVM Tapioca Private Limited, Rasipuram, Tamil Nadu, served as a natural polymeric filler and was characterized by low moisture content, typically below 12% and an amylose content around 17 to 20%. The starch granules measured between 5 and 35 micrometers and appeared as a fine white powder with the general chemical formula $(C_6H_{10}O_5)n$ for its repeating units. Glycerol sourced from Godrej Industries, Salem, Tamil Nadu acted as a plasticizer to enhance flexibility and reduce brittleness in the films. It was used in liquid form, clear and viscous, with a purity exceeding 99%, a density near 1.26 g/cm^3 at room temperature and a chemical formula of $C_3H_8O_3$. Distilled water was utilized throughout the solution casting process as the primary solvent.

2.2 Biofilm Preparation

In the preparation of PLA/tapioca starch biofilms via the solution casting technique, the formulation consisted of 10 grams of tapioca starch and 3 grams of PLA resulting in a polymer blend composition of approximately 76.92% tapioca starch and 23.08% PLA by weight. This ratio was selected to balance the biodegradable and film-forming properties of starch with the mechanical strength and hydrophobic characteristics of PLA. Adding glycerol as a polyol in varying amounts ranging from 4 mL to 20 mL was employed to enhance flexibility and reduce brittleness in the resulting films. Biofilms were prepared using polylactic acid (PLA), tapioca starch, glycerol, distilled water and vinegar through a solution casting technique. Initially, 10 grams of tapioca starch were dispersed in 100 mL of distilled water and heated to approximately 80°C under continuous stirring for 20 minutes to achieve gelatinization. Separately 3 grams of PLA were dissolved in 50 mL of distilled water containing 2% v/v vinegar at around 60°C with constant stirring to aid dispersion. Once gelatinization was complete, the PLA solution was gradually combined with the starch mixture. Glycerol was then incorporated at different volumes, specifically 4, 8, 12, 16 and 20 mL while a control sample was prepared without glycerol addition. The mixtures were stirred thoroughly at 70–75°C for an additional 20 minutes to ensure uniform blending. The final film-forming solutions were poured onto clean glass plates and spread evenly to maintain consistent thickness. The films were subsequently dried in an oven at 50°C for approximately 24 hours to remove excess moisture and were carefully peeled off for further analysis and characterization.

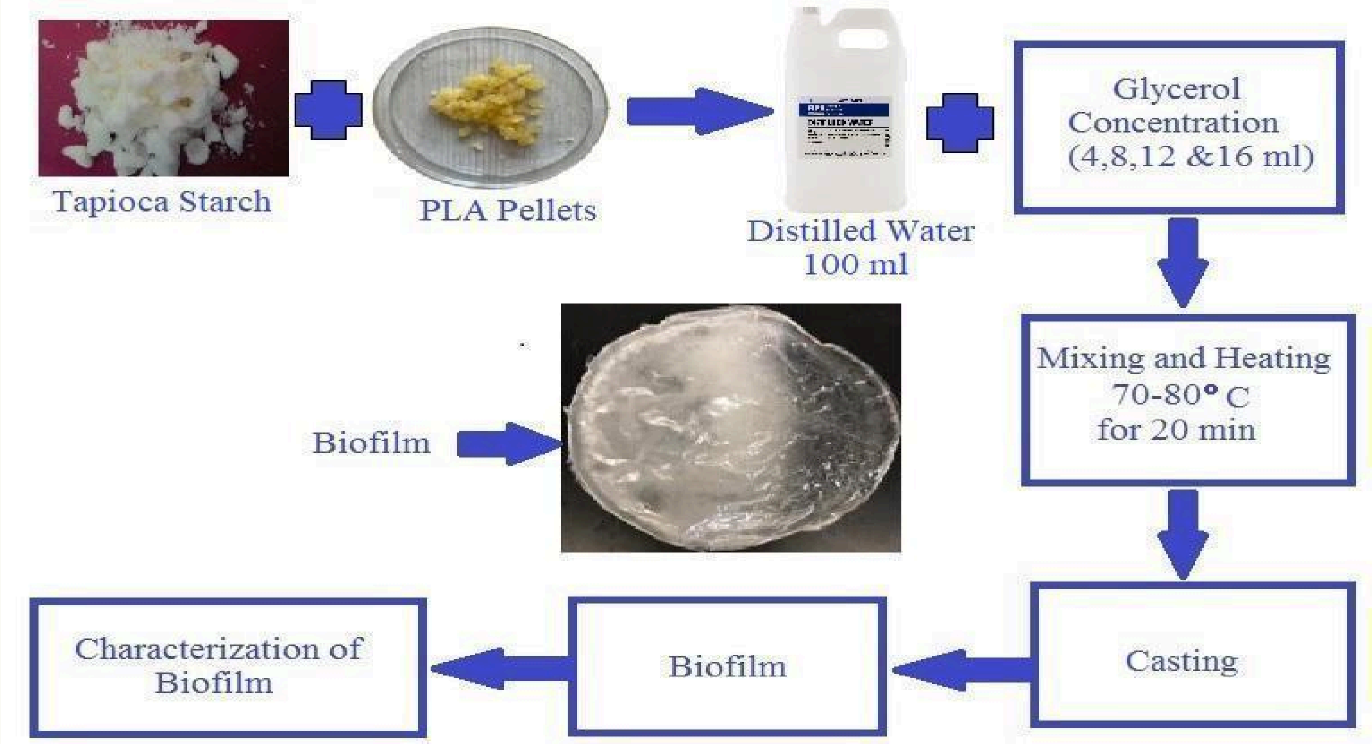


Figure 1. Flow diagram of PLA/TS biofilm

3. Results and Discussion

3.1 Physical characteristics

3.1.1 Appearance of biofilm

Table 1. Appearance of biofilm

Sample	**Plasticizer**	**Plasticizer in ml**	**Appearance of biofilm**	
Control PLA/TS	No Glycerol	0	Transparent film with surface cracks; brittle and fragile; difficult to peel from casting surface.	
PLA/TS/G1	Glycerol	4	More transparent than control; slightly sticky; flexible; not brittle or fragile; easily peelable.	
PLA/TS/G2	Glycerol	8	More transparent; stickier than G1; flexible; not brittle or fragile; easily peelable.	
PLA/TS/G3	Glycerol	12	More transparent; stickier than G2; flexible; not brittle or fragile; easily peelable.	
PLA/TS/G4	Glycerol	16	More transparent; stickier than G3; flexible; not brittle or fragile; easily peelable.	
PLA/TS/G5	Glycerol	20	Crystal clear appearance; rigid; non-sticky; not brittle or fragile; peelable without damage	

3.1.2 Thickness of Biofilm

Figure 2 illustrates the changes in film thickness at varying plasticizer concentrations. An increase in plasticizer content from 4 mL to 20 mL resulted in a noticeable rise in the thickness of the films, irrespective of the plasticizer type. A similar trend was noted by Ibrahim et al., who reported that the nature of the plasticizer did not significantly influence this effect. Likewise, Sanyang et al. (2015) attributed this phenomenon to the plasticizer's ability to alter the intermolecular interactions within the polymer matrix. This modification increases the free volume between polymer chains, thereby contributing to the observed increase in film thickness.

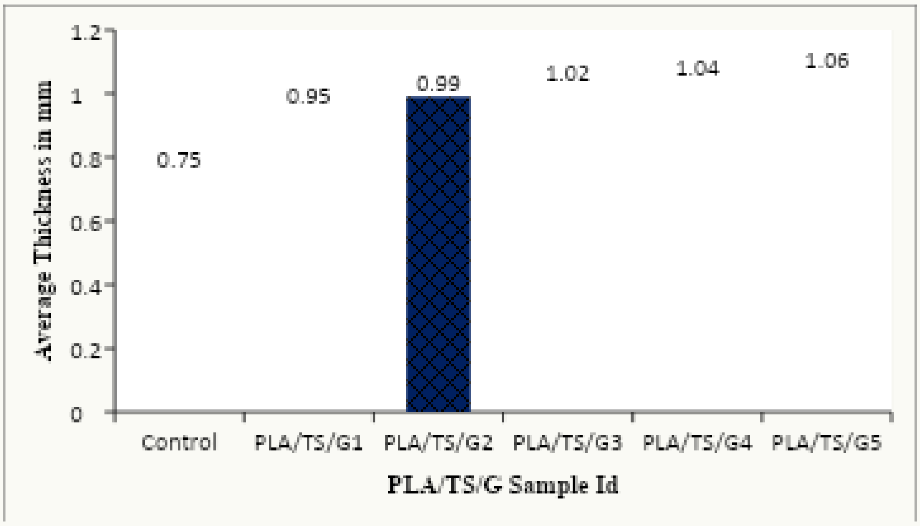


Figure 2. Thickness of PLA/TS/G

3.1.3 Solubility of PLA/TS/G

Figure 3 presents the diverse results obtained from solubility tests conducted on PLA and tapioca starch (TS) films plasticized with glycerol. The data revealed that the PLA/TS/G5 formulation exhibited the highest solubility among the samples. This behavior is linked to the presence of glycerol, which plays a significant role as a plasticizer in polymer processing. Glycerol, an organic compound with a relatively high boiling point, can reduce intermolecular forces between long polymer chains, thereby decreasing rigidity in otherwise stiff or brittle resins, [17]. Consequently, plasticization influences key mechanical properties of the films, including their elasticity, hardness and resistance to water absorption.

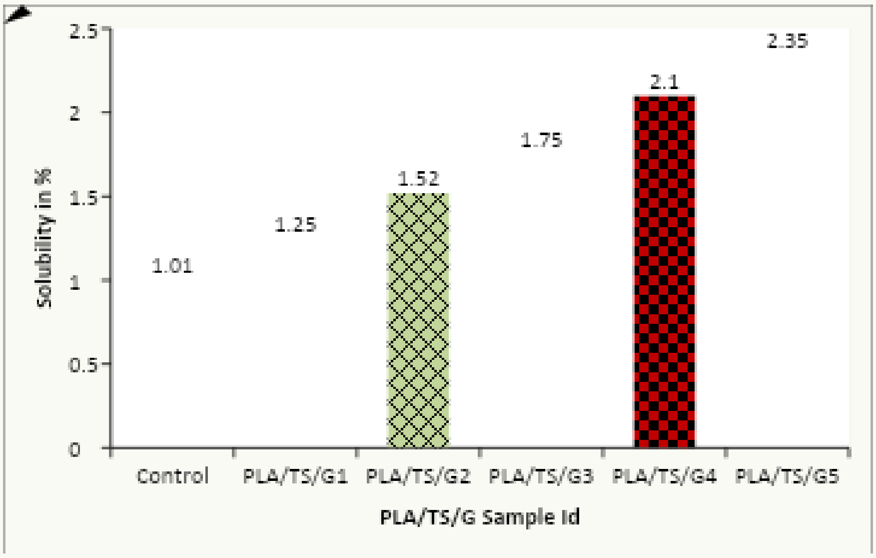


Figure 3. Solubility of PLA/TS/G

3.1.4 Swelling ratio of PLA/TS/G

As illustrated in Figure 4, incorporating glycerol as a plasticizer into PLA and tapioca starch (TS) blends reduces the material's tendency to absorb water. The results indicate a clear inverse relationship between the glycerol content and the swelling ratio, suggesting that higher concentrations of glycerol enhance the water resistance of the polymer films. Glycerol functions effectively as a plasticizer, contributing to increased elongation, reduced stiffness and improved flexibility of the resin [16]. Glycerol contributes to preserving the structural stability of polymer blends, reducing substantial physical alterations during storage. The presence of both hydrophilic and hydrophobic polymers within these blends enhances their degradation behavior, facilitating improved environmental breakdown of the films.

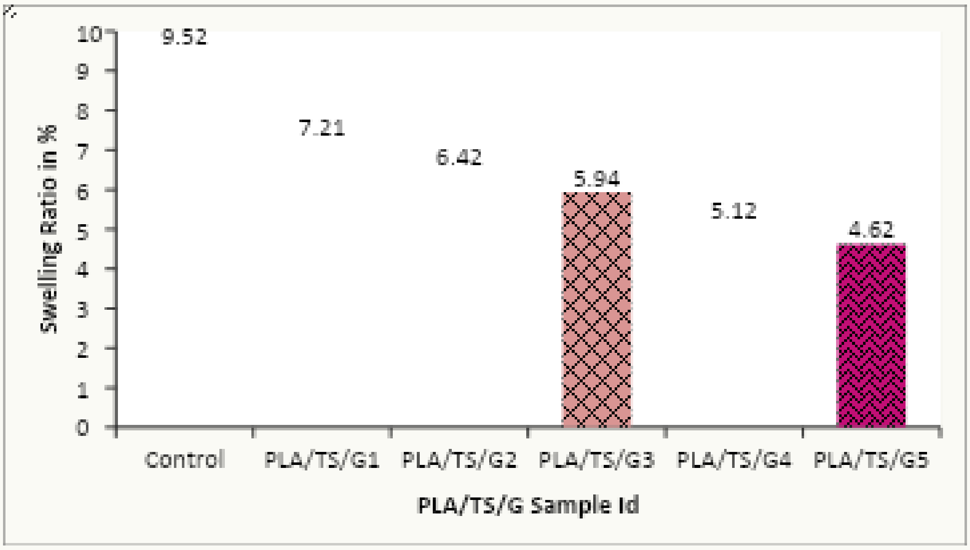


Figure 4. Swelling Ratio of PLA/TS/G

3.1.5 Chemical resistance of PLA/TS/G

One test used to assess a material's resistance is chemical resistance. Testing for chemical resistance is done using ASTM D1647-89. For three days, an acidic (pH of HCl) and alkaline (pH of NaOH) solution served as the test media. The lack of testing suggested that the coated surface was peeling off and no longer adhered to the substrate. Table 2 tabulates the test findings.

Table 2. Solubility results in different PH values

Conditions	PLA/TS/G 1	PLA/TS/G2	PLA/TS/G3	PLA/TS/G4	PLA/TS/G5
Acid (pH 4)	Soluble	Soluble	Insoluble	Insoluble	Soluble
Neutral (pH 7)	Soluble	Insoluble	Insoluble	Soluble	Soluble
Base (pH 10)	Soluble	Soluble	Insoluble	Soluble	Soluble

The findings of the chemical resistance test indicate that PLA/TS/G3 is the optimal layer. This is evident from the fact that TS and glycerol are present and tightly bound to PLA, giving them superior chemical resistance over other forms.

3.1.6 Tensile Strength of PLA/TS/G

Figures 5 & 6 illustrate how glycerol addition to PLA/TS affects tensile strength and elongation. The image demonstrates how tensile strength can be decreased by adding glycerol plasticizer, with 1.32 MPa being the ideal value for PLA/TS/G1. Conversely, the PLA/TS/G4 variable yields the best elongation result, with a value of 0.015. The material is fragile if the elongation value is less than 15% [7]. The properties of the material can be altered by adding tapioca starch (TS), which has been plasticized with glycerol. These findings support a study by Yu et al. (2006) that showed that tensile strength can be increased by combining PLA with products made from Tapioca Starch [12].

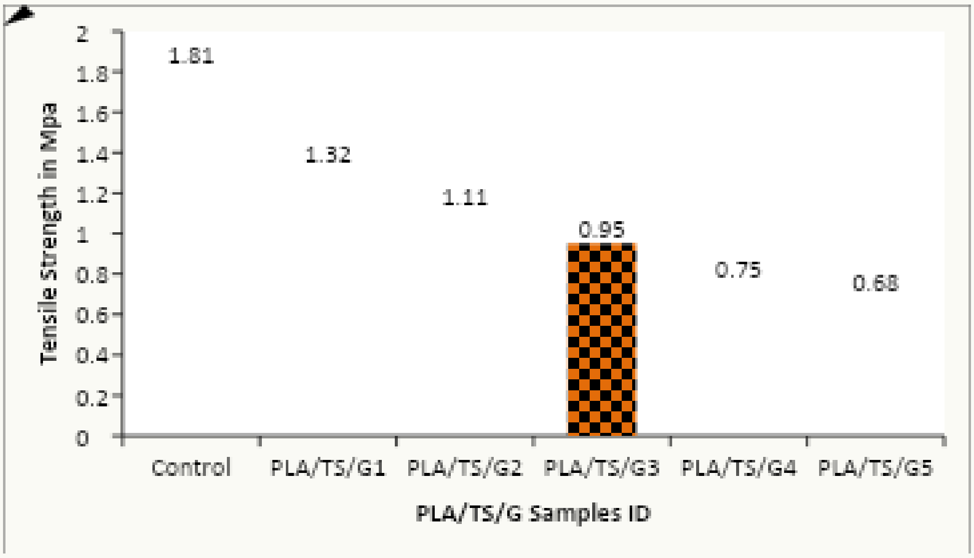


Figure 5. Tensile Strength of PLA/TS/G

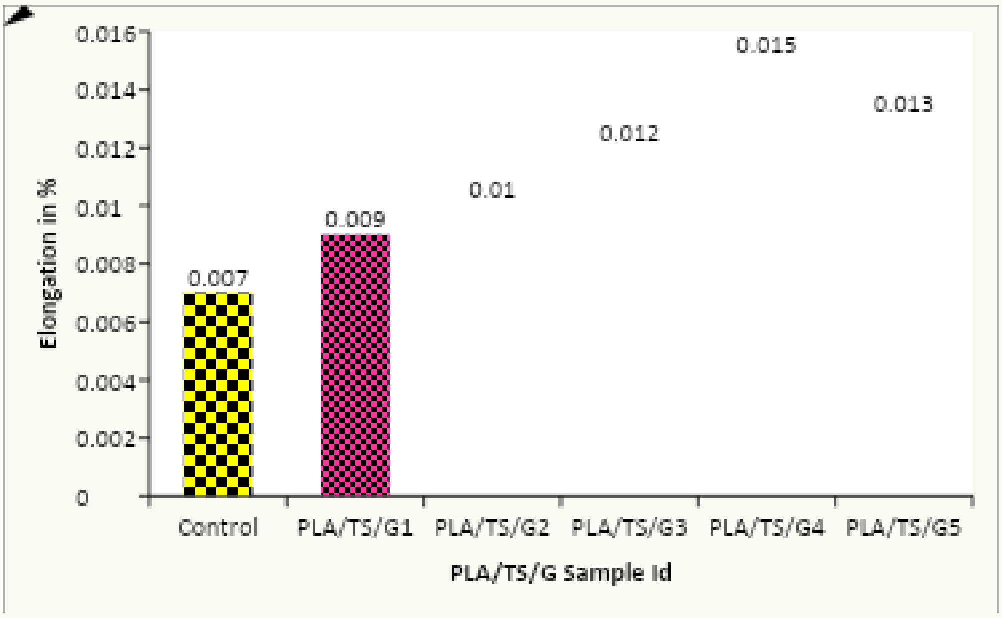
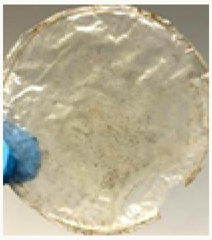
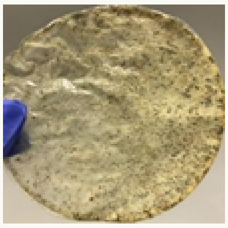
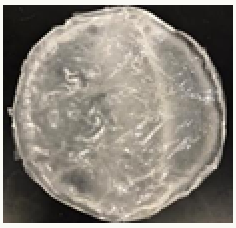
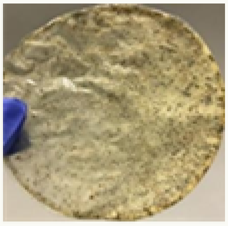
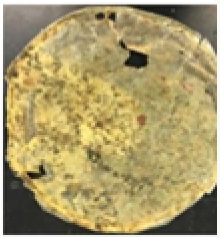
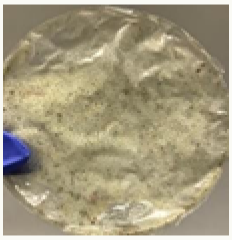


Figure 6. Elongation of PLA/TS/G

3.1.7 Biodegradability Test

The soil burial test was carried out over a 28-day period, with samples retrieved and examined at intervals of seven days. Table 3 provides a comparison of the films' appearance before burial and after each exposure period in soil.

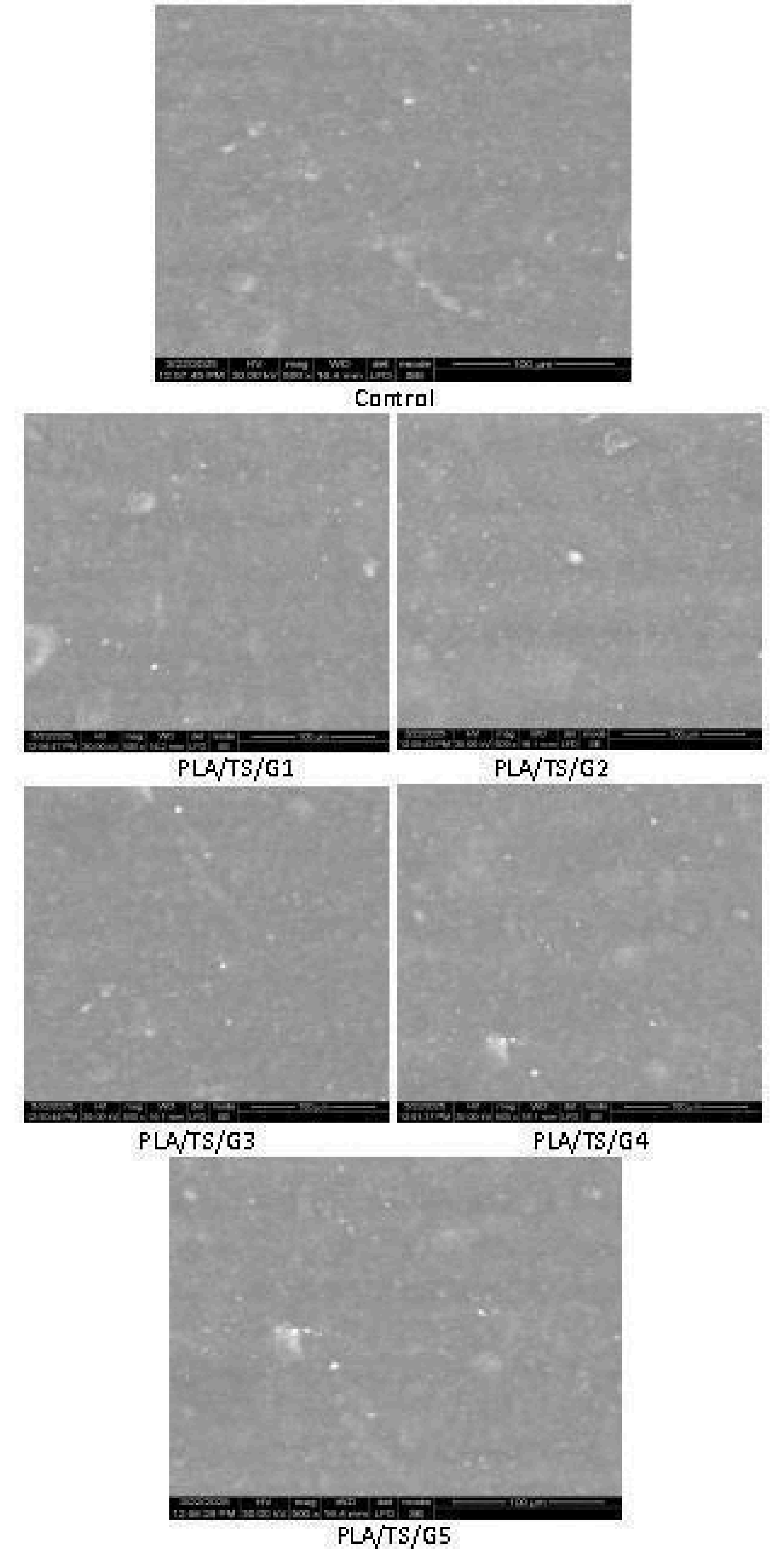
Table 3. Physical appearance of Biofilm before and after Biodegradability test.

Biofilm ID	Before Soil Burial	7 Days After	14 Days After	21 Days After	28 Days After
PLA/TS					
PLA/TS/ G1					
PLA/TS/ G2					
PLA/TS/ G3					
PLA/TS/ G4					
PLA/TS/ G5					

From Table 3, it is evident that the control PLA/TS biofilm exhibits minimal visual changes during the soil burial period, which can be attributed to its hydrophobic characteristics and relatively slow degradation rate. Comparable findings were reported in previous PLA/TS soil burial degradation studies. As the tapioca starch (TS) content in PLA increases, the composites display a progressive rise in both weight loss percentage and overall degradation. Observations from Table 4 indicate that PLA/TS/G2, PLA/TS/G3, PLA/TS/G4 and PLA/TS/G5 bio-composites develop visible cracks, holes and fungal growth on their surfaces and become increasingly brittle after burial. These degradation features intensify over time for all bio-composites, with more pronounced changes in samples containing higher starch loadings.

3.1.8 Scanning Electron Microscopy (SEM) for PLA/TS/G Biofilm

The SEM images revealed noticeable differences in the microstructure depending on glycerol concentration. Films with lower glycerol content displayed relatively rough and irregular surfaces, with visible starch granules and micro-cracks, indicating weaker interactions between the polymer components, consistent with prior observations in PLA/starch composites (Jandas et al., 2013; Ma et al., 2021)[18,19]. Conversely, samples containing higher glycerol content, particularly PLA/TS/G5, displayed smoother and more uniform surface morphologies. This indicates enhanced interfacial compatibility and more effective dispersion of starch particles within the PLA matrix (Wang et al., 2018). This enhanced microstructural uniformity can be linked to the increased solubility observed in the solubility tests, as well as to the plasticizing effect of glycerol, which reduces brittleness and promotes flexibility, as documented in earlier studies on starch-based biofilms (Ma et al., 2021; Jandas et al., 2013)[18,19]. Overall, the SEM observations support the conclusion that glycerol not only modifies the mechanical and water-related properties of the films but also significantly influences their morphological characteristics (Wang et al., 2018)[21]. Figure 2.5 shows the SEM images of PLA/TS/G Biofilm.



Control

PLA/TS/G1

PLA/TS/G2

PLA/TS/G3

PLA/TS/G4

PLA/TS/G5

Figure 2.5. SEM of PLA/TS/G Biofilm

3.1.9 FTIR for PLA/TS/G Biofilm

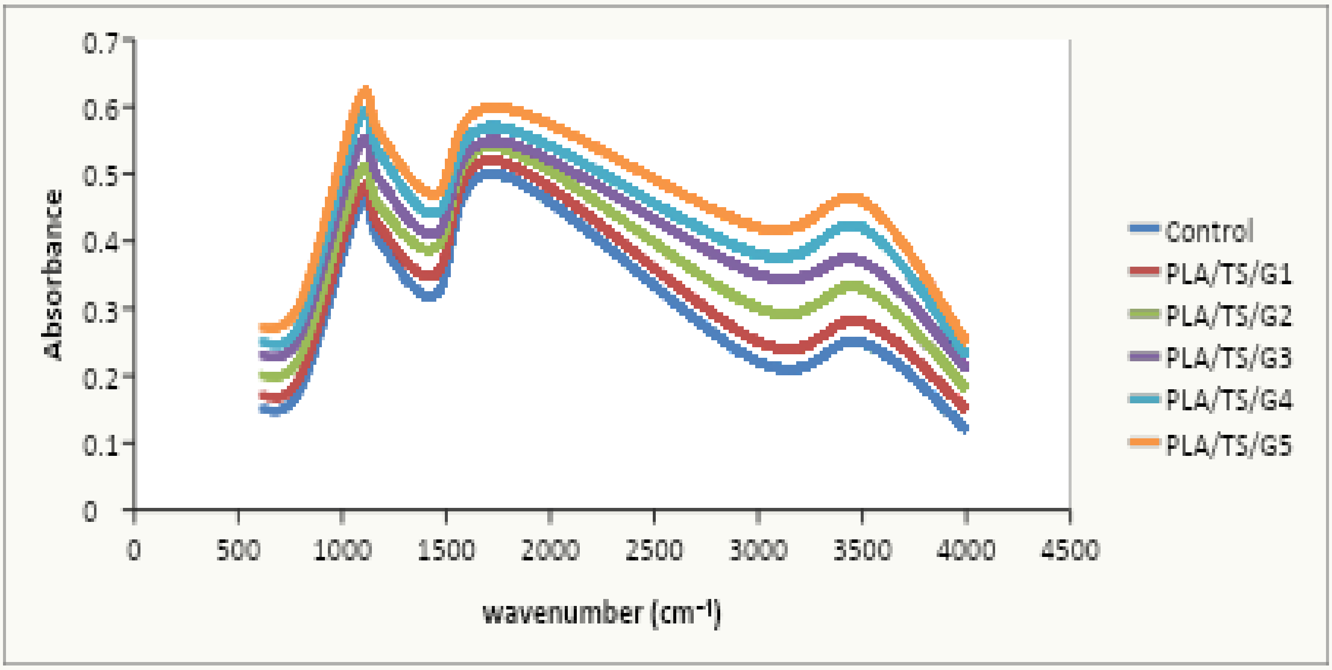


Figure 7. FTIR of PLA/TS/G Biofilm

FTIR analysis was performed to examine the chemical interactions and structural modifications in PLA/TS biofilms with varying glycerol concentrations, as illustrated in Figure 7. The spectra of all samples exhibited characteristic peaks associated with PLA, including prominent absorption bands around 1750 cm^{-1}, corresponding to the C=O stretching vibration of ester groups, consistent with previous findings on PLA-based materials [20,21]. Peaks observed near 1180–1080 cm^{-1} were attributed to C–O–C stretching vibrations, indicative of the polymer backbone structure of PLA [20]. For the starch component, broad absorption bands around 3200–3400 cm^{-1} were noted, corresponding to O–H stretching vibrations, reflecting the hydrophilic nature of starch, as reported in earlier studies on starch-based films 19]. As the glycerol content increased, slight intensification and broadening of the O–H stretching peak were observed, suggesting enhanced hydrogen bonding among glycerol, starch and PLA chains. Additionally, minor shifts in the C=O stretching peak position were detected in films with higher glycerol levels, indicating possible interactions between glycerol and PLA molecules. These spectral changes confirm the effective incorporation of glycerol as a plasticizer, influencing molecular interactions within the bio-composite films and contributing to modifications in mechanical properties and solubility behavior.

3.1.10 Thermogravimetric Analysis (TGA) for PLA/TS/G Biofilm

Figure 8 shows the thermal degradation behavior of PLA/tapioca starch biofilms containing various concentrations of glycerol was evaluated using Thermogravimetric Analysis (TGA). Approximately 5–10 mg of each film sample was placed in a platinum crucible and heated from room temperature up to 600°C at a heating rate of 10°C per minute under a constant flow of nitrogen gas to prevent oxidative degradation, following standard procedures for polymeric materials [22,23].

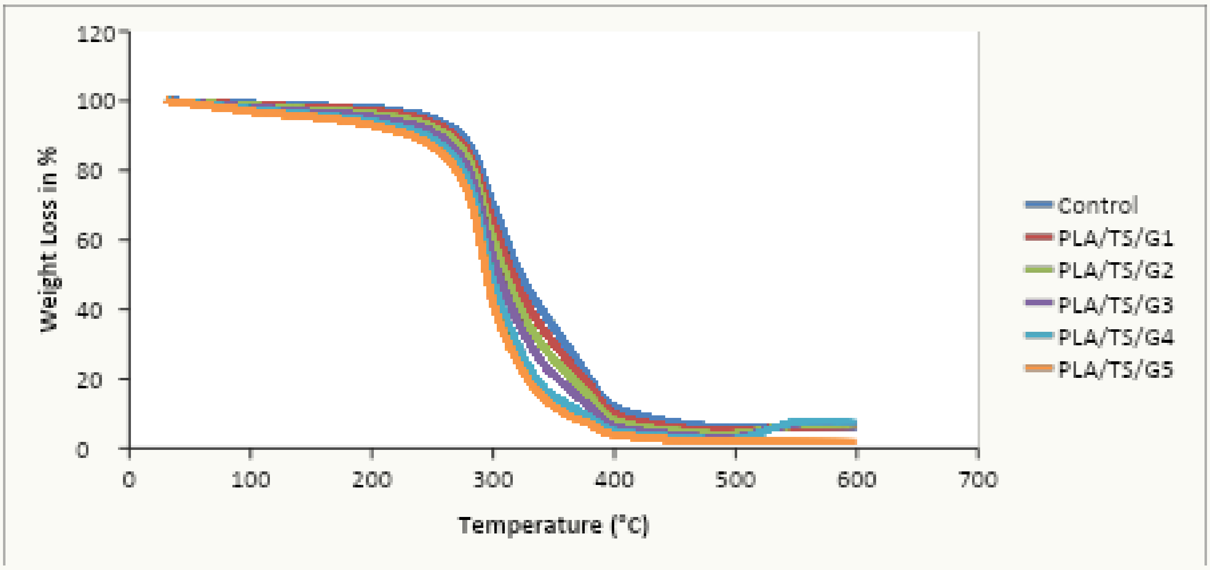


Figure 8. TGA of PLA/TS/G Biofilm

The TGA curves revealed an initial weight loss below 150°C, attributed to the evaporation of moisture and residual solvents, which is consistent with previous studies on biodegradable polymers [23]. A significant degradation step was observed between approximately 280°C and 380°C, corresponding to the thermal decomposition of PLA and starch components, in agreement with typical degradation profiles for such bio-composites. Films with higher glycerol content exhibited slightly earlier onset temperatures of degradation, indicating reduced thermal stability due to the plasticizing effect of glycerol, which decreases intermolecular forces within the polymer matrix. Despite this reduction in thermal stability, the addition of glycerol improved film flexibility, balancing mechanical properties with thermal performance. These thermal results confirm that the formulation of PLA/starch biofilms can be tailored to achieve desired thermal and mechanical characteristics, making them suitable for biodegradable applications. Table 4 shows the degradation temperature for PLA/TS/G Biofilm.

Table 4 Thermal Degradation of PLA/TS/G Biofilm

Sample	**Onset Degradation Temp (°C)**	**Maximum Degradation Temp (°C)**	**Residual Mass at 600°C (%)**
Control	295	340	5.8
PLA/TS/G1	290	335	6.3
PLA/TS/G2	285	330	6.8
PLA/TS/G3	280	325	7.2
PLA/TS/G4	275	320	7.6
PLA/TS/G5	270	315	8.1

4. Conclusion

The mechanical characteristics of the PLA/TS blend were improved through the incorporation of glycerol, which acted as an effective plasticizer. Among the samples, PLA/TS/G1 exhibited the highest tensile strength of 1.32 MPa, while PLA/TS/G3 demonstrated the greatest elongation at break, indicating enhanced flexibility. Regarding solubility and water resistance, PLA/TS/G5 showed the lowest swelling ratio yet the highest solubility, suggesting a balanced interplay between hydrophilic and hydrophobic interactions in the polymer matrix. FTIR analysis confirmed successful integration of glycerol, evidenced by shifts in characteristic O–H and C=O absorption bands, indicating hydrogen bonding between PLA, starch and glycerol. SEM observations revealed smoother and more homogeneous surfaces in films with higher glycerol content, correlating with improved film uniformity and reduced surface defects. Thermal degradation analysis showed that while higher glycerol concentrations lowered the onset degradation temperatures slightly, they also increased residual mass at elevated temperatures, reflecting changes in thermal stability due to plasticization. The development of eco-friendly biomaterials suitable for biomedical applications or sustainable polymer packaging offers a promising alternative to conventional plastics.

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