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Performance Analysis of PLA/TS Blends with Glycerol Plasticizers

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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 (TS) 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 films 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, TS and glycerol with peak shifts in the hydroxyl and carbonyl regions. Scanning Electron Microscopy (SEM)images indicated smoother and more uniform surfaces at higher glycerol concentrations, suggesting improved compatibility. Thermogravimetric Analysis (TGA) results showed a slight reduction in thermal stability with increased glycerol, attributed to greater chain mobility. Glycerol content is found to have a considerable effect on biofilm properties.

Keywords: Glycerol, polylactic acid, biodegradation, Tensile strength, biofilm, casting.

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. PLA is a naturally occurring polymer substance with the potential to replace petroleum-based polymers. It is an eco-friendly and biocompatible polymer synthesized from renewable biological sources, which naturally degrades over time [1]. It is made from cellulose, glycerin, sugar, and starch[2]. The obtained products combine with the stereoisomers [3]. Plasticizers such as glycerol are commonly added to enhance flexibility and reduce brittleness.PLA has drawbacks, including high hydrophobicity, brittleness, and low ductility, in addition to its advantages [4,5]. By mixing techniques with other polymers, PLA can be modified to enhance its mechanical properties without the need to create new polymers. This approach is regarded as efficient, straightforward and adaptable for creating new materials [6]. Biodegradable biofilms have been developed using natural polymers such as starch, cellulose, chitosan, alginate, and gelatin, as well as synthetic biodegradable polymers. Polymers are employed, with starch, chitosan, polybutylene adipatecoterephthalate (PBAT), polycaprolactone (PCL), polyethene glycol (PEG), Polybutylene Succinate (PBS) and polyhydroxybutyrate (PHB) [7]. Flour and tapioca root embryo are separated to make TS. One million tons of tapioca root yields half a ton of TS, since TS 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 TS [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 TS, it is readily available and reasonably priced biomaterials; combining PLA with them could be one of the more promising approaches. The PLA/TS blend's TS is utilised 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 TS enhances the behaviour of the material,likeelongation with reducedpulling strength, which contributes to improved flexibility in the composite [12,25]. From the literature, it is identified that the PLA, with orange peel, wheat bran, etc., is fabricated and tested, but this combination of TS, glycerol and fabricated biofilms has not been studied in earlier research. The effects of the PLA+TS+Glycerol

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combination are the main focus of this study; the inclusion is modified for fabrication and testing.The key limitations of existing PLA/starch biofilms are brittleness, poor interfacial compatibility, moisture sensitivity, and limited control over biodegradation. The novelty of this work lies in the systematic investigation of PLA/thermoplastic starch biodegradable films with controlled plasticizer content and the correlation between mechanical performance, structural characteristics, and surface morphology to better understand their suitability for sustainable packaging applications.. The scientific justification for selecting tapioca starch and the plasticization mechanism of glycerol was also not adequately explained. To address these weaknesses, the primary objective of this study is to systematically investigate the effect of varying glycerol concentrations on the mechanical, physical, thermal, morphological, and biodegradation properties of PLA/TS biofilms prepared via solution casting, to enhance their functional performance for biodegradable material applications on PLA/tapioca starch biofilms.

2. Process Description

2.1. Materials

PLA, TS 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. Food-grade TS, 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 of around 17 to 20%. The starch granules measured between 5 and 35 micrometres and appeared as a fine white powder $(C_6H_{10}O_5)n$ 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/cm³ 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/TS biofilms via the solution casting technique, the formulation consisted of 10 grams of TS and 3 grams of PLA, resulting in a polymer blend composition of approximately 76.92% TS and 23.08% PLA by weight. This ratio was selected to balance the biodegradable and film-forming properties of starch, having mechanical strength and hydrophobic behaviour 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 PLA. TS, glycerol, distilled water and vinegar through a solution casting technique. Initially, 10 grams of TS were dispersed with 100 mL of purified 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 gelatinisation 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 film solutions were poured onto clean glass plates and spread evenly to maintain a consistent thickness. The films were then gently peeled off for additional examination and characterisation after being heated to 50°C for 24 hrs. to eliminate any remaining moisture.

3. Results and Discussion

3.1. Physical Appearance of Prepared Samples

The effect of glycerol content on the appearance and handling properties of PLA/TS biofilms is shown in Table 1. The control film without glycerol exhibited surface cracks and brittle behavior, making it difficult to peel from the casting surface. With the addition of glycerol (4–8 mL), the films became more transparent, flexible, and easier to peel, indicating improved plasticization of the polymer matrix. The increased glycerol content enhanced intermolecular mobility, reducing brittleness and improving film integrity.

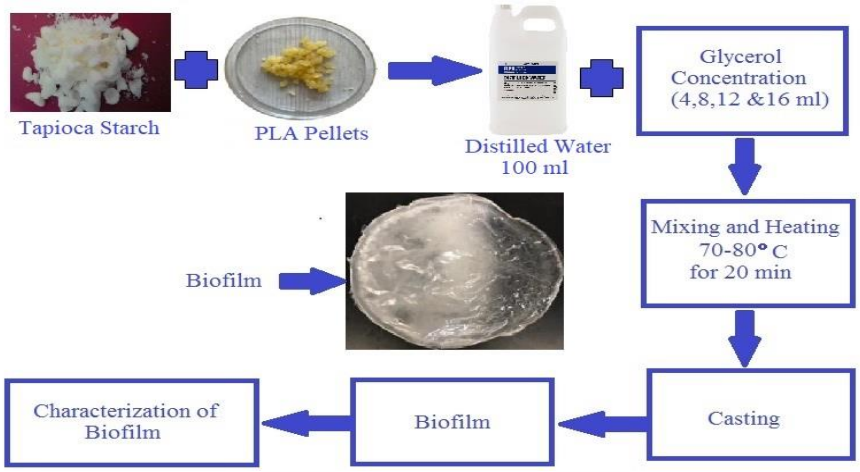
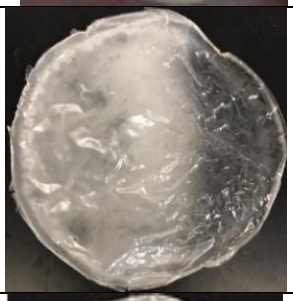


Figure 1. Flow diagram of PLA/TS biofilm

Table 1. Appearance of biofilm

Sample	Plasticizer	Plasticizerin ml	Appearance of biofilm	
Control PLA/TS	No Glycerol	0	Transparent film with cracks on the surface; 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 peel able.	
PLA/TS/G2	Glycerol	8	More transparent; stickier than G1; flexible; not brittle or fragile; easily peel able.	
PLA/TS/G3	Glycerol	12	More transparent; stickier than G2; flexible; not brittle or fragile; easily peel able.	
PLA/TS/G4	Glycerol	16	More transparent; stickier than G3; flexible; not brittle or fragile; easily peelable.	
PLA/TS/G5	Glycerol	20	Clear appearance; rigid; non-sticky; not brittle or fragile; peelable without damage	

3.1.1. Thickness of Biofilm

An increase in plasticizer content from 4 mL to 20 mL resulted in a noticeable rise in the thickness, irrespective of the plasticizer, as shown in Figure 2. It is identified that similar trend was noted by [24], showing that the nature of the plasticizer does not influence this effect. The ability of the plasticizer to alter the intermolecular interactions within the polymer matrix [25]. This modification increases the free volume between polymer chains, thereby contributing to the observed increase in film thickness. The thickness of the biofilms was measured using a digital micrometer with an accuracy of ±0.001 mm. Measurements were taken at five different random positions on each film sample to ensure uniformity, and the average value was reported as the final thickness.

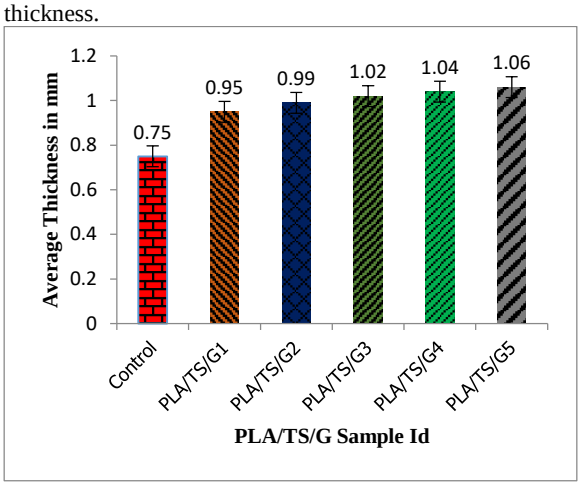


Figure 2. Average thickness of PLA/TS/G biofilms

3.1.2. Solubility of PLA/TS/G

Figure 3 presents the diverse results obtained from solubility tests conducted on PLA and TS films plasticized with glycerol. The data revealed that the PLA/TS/G5 formulation exhibited the highest solubility among the samples. This behaviour 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. The solubility test was conducted by immersing a pre-weighed dry film sample in distilled water for a specified duration. After immersion, the remaining insoluble portion was collected, dried to constant weight, and the percentage solubility was calculated based on the difference between the initial dry weight and the final dried residue.

3.1.3. Swelling ratio of PLA/TS/G samples

Figure 4 illustrates that incorporating glycerol as a plasticizer into PLA and TS (TS) blends reduces the material's tendency to absorb water. The swelling ratio was determined by immersing the dried film samples in distilled water for a specific time period. The swollen films were then removed, gently wiped to remove excess water, and weighed. The swelling ratio was calculated based on the percentage increase in weight relative to the initial dry weight. 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 behaviour, facilitating improved environmental breakdown of the films.

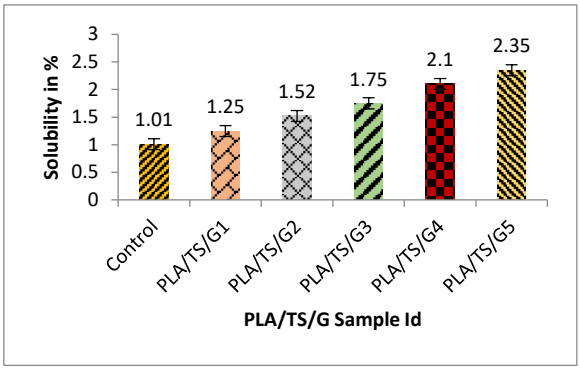


Figure 3. Solubility of PLA/TS/G samples

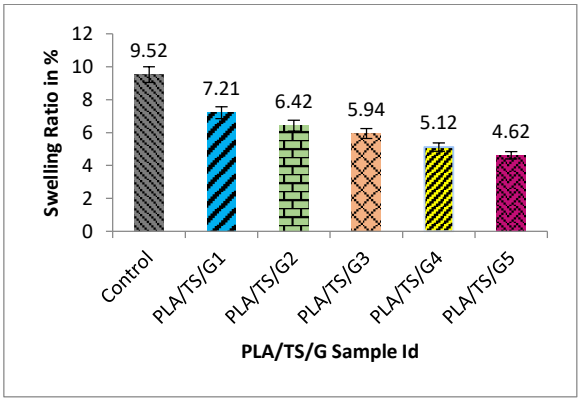


Figure 4. Swelling ratio of the samples

3.1.4. 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. According to the findings, PLA/TS/G1 was dissolvable at all conditions that were tested (acidic, neutral, and basic). PLA/TS/G2 was soluble in basic and acidic medium but was insoluble in neutral medium. PLA/TS/G3 was always insoluble in any case. PLA/TS/G4 was soluble in neutral and basic medium and insoluble in acidic medium, whereas PLA/TS/G5 was soluble in all three pH levels except the neutral medium, in which it was also soluble. The results from the chemical resistance test PLA/TS/G3 revealed the best layer due to TS and glycerol are strongly attached to the PLA, making this combination more resistant to chemicals than other types.

3.1.5. Tensile Strength of PLA/TS/G

Figures 5 & 6 illustrate that glycerol addition to PLA/TS affects tensile strength and elongation. The image demonstrates that 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 samples changed by adding TS, which has been plasticized with glycerol. The tensile strength can be increased by combining PLA with products made from TS [12].

3.1.6. Biodegradability Test

The soil burial test was carried out over 28 days, with samples retrieved and examined at intervals of seven days. Table 3 provides a comparison of the film's appearance before burial and after each exposure period in soil. Literature reports that PLA degrades primarily through hydrolysis of ester linkages, followed by microbial assimilation, which occurs slowly under ambient soil burial conditions [13,14]. The complete disintegration into small fragments in G4 and G5 at 28 days indicates transition from surface erosion to bulk degradation. Similar fragmentation patterns in PLA/starch/glycerol systems under soil burial have been reported by [15]. The soil burial study clearly demonstrates the progressive degradation behaviour of PLA/TS and PLA/TS/G biofilms as a function of burial duration and glycerol content. Before burial, all samples exhibited a uniform and intact structure, indicating proper film formation and good compatibility among the components. After 7 days of soil exposure, slight discoloration and initial surface deterioration were observed, particularly in glycerol-containing films, suggesting the onset of moisture absorption and microbial interaction. As the burial period increased to 14 and 21 days, the films showed noticeable structural weakening, including increased opacity, surface irregularities, and the formation of cracks, indicating ongoing hydrolytic and microbial degradation. By 28 days, the films with higher glycerol concentrations displayed significant fragmentation and loss of structural integrity, whereas the PLA/TS film without glycerol remained comparatively more stable. This behaviour can be attributed to the hydrophilic nature of starch and glycerol, which facilitates water uptake and enhances microbial accessibility within the polymer matrix. The increased plasticization effect of glycerol also reduces intermolecular interactions, making the polymer network more susceptible to degradation. Overall, the results confirm that glycerol incorporation accelerates the biodegradation rate of PLA/TS biofilms under soil burial conditions.

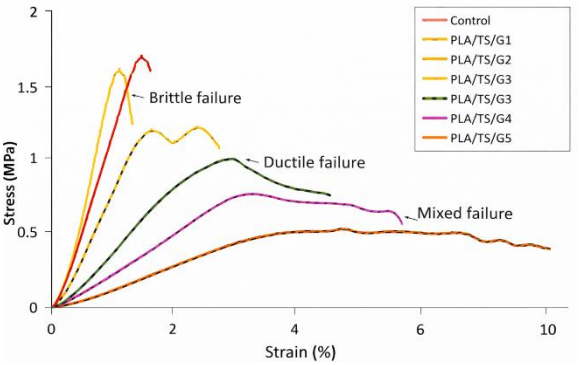


Figure 5. Tensile Strength of PLA/TS/G

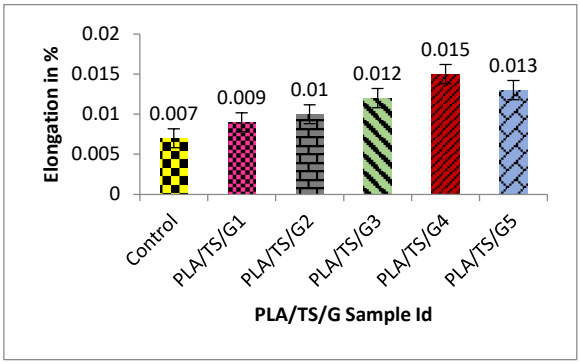
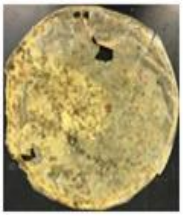
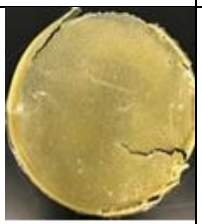


Figure 6. Elongation of PLA/TS/G

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

The SEM images shown in Figure 7 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 [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 [21]. 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 [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 [21].

Table 3. Physical appearance of Biofilm before and after the 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					

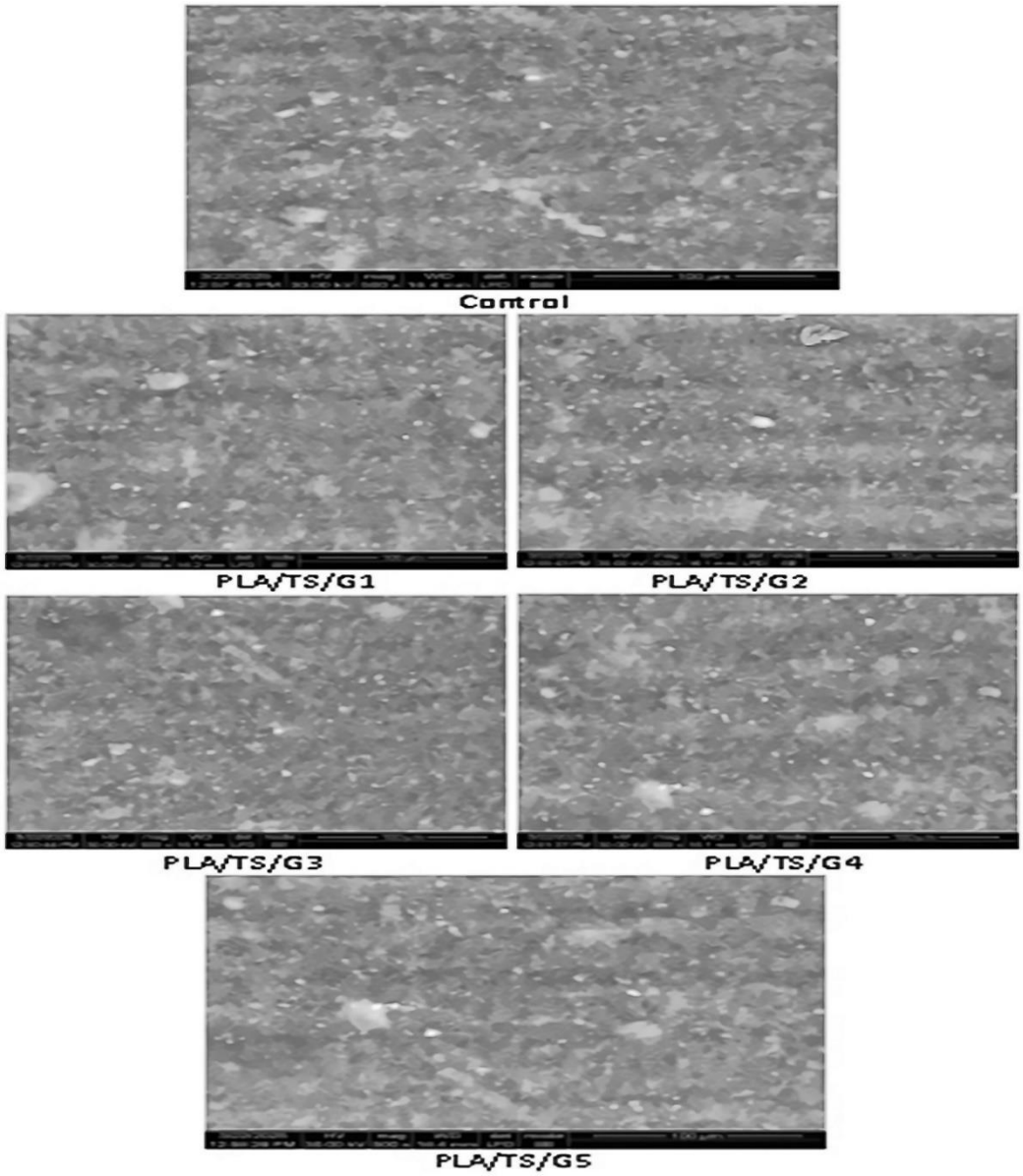


Figure 7. SEM of PLA/TS/G Biofilm

3.1.8. FTIR for PLA/TS/G Biofilm

The structural and chemical modifications in PLA/TS biofilms with varying glycerol concentrations are analyzed through FTIR, as illustrated in Figure 8. The spectra of all samples exhibited characteristic peaks associated with PLA, including absorption bands around 1750 cm^{-1} with C=O stretching ester groups, consistent with previous findings on PLA-based materials [20,21]. Peaks observed near 1180–1080 cm^{-1} attributed to C–O–C vibrations, indicatepolymer 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 widening O–H stretching 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.

3.1.9. TGA for PLA/TS/G Biofilm

Figure 9 reveals the thermal degradation behaviour of PLA/TS biofilms containing various concentrations of glycerol evaluated using TGA.A sample weighing 10 mg was heated in a crucible from 32 °C to600°C,and nitrogen gas was used to stop oxidation and breakdown, following standard methods for polymeric materials [22, 23].

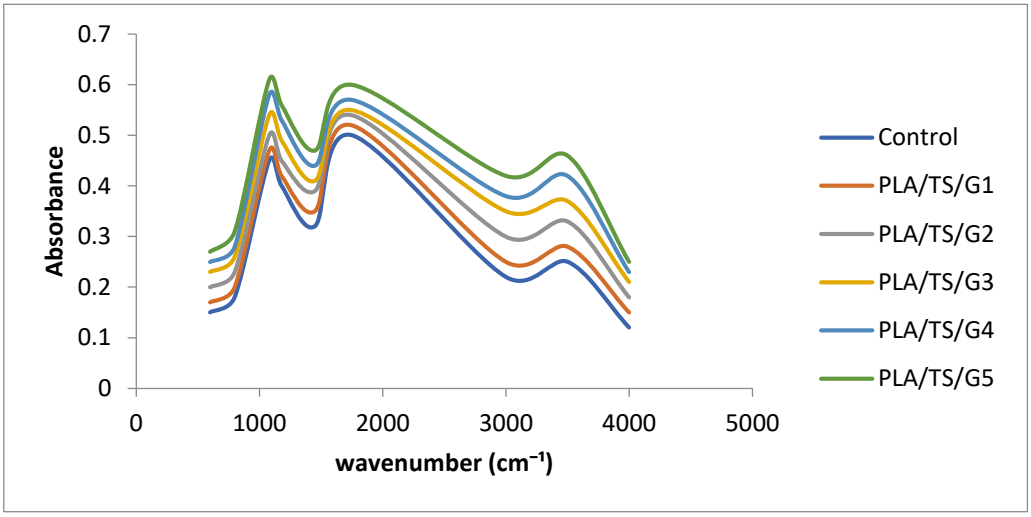


Figure 8. FTIR of PLA/TS/G Biofilm

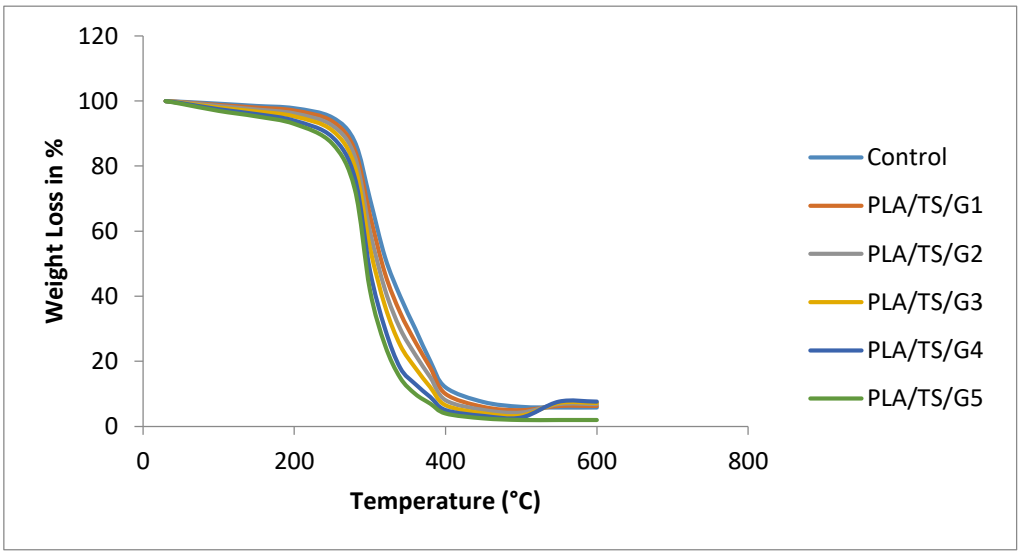


Figure 9. TGA of PLA/TS/G Biofilm

The TGA curves showed a small weight loss under 150°C, which is due to moisture and leftover solvents evaporating, something that has been seen in past research on biodegradable polymers [23]. There was a big drop in weight between about 280°C and 380°C, which matches the breakdown of PLA and starch parts, similar to what's usually seen in these types of 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. There is a strong relationship between glycerol content, mechanical properties, interaction with water, morphology, and biodegradation behaviour. Increasing the content of glycerol increased the biofilms' flexibility and elongation but reduced the tensile strength of the biofilms due to the reduced intermolecular forces. Increasing solubility and swelling behaviour were related to the increased hydrophilicity, which was evident from the FTIR results. This was confirmed by the SEM images, which showed the improvement in dispersion as well as the reduction of microcracks with the addition of the plasticizer. The thermal results showed that the degradation temperature was reduced with the addition of the plasticizer. The biodegradability increased with the starch and glycerol content due to improved accessibility to microorganisms. The results collectively confirm the importance of glycerol content in the modification of the biofilms.The observed results are consistent with previously reported studies on PLA/starch biodegradable films. The incorporation of thermoplastic starch into the PLA matrix generally improves film flexibility while slightly reducing tensile strength due to phase heterogeneity between the two components. Similar behavior has been reported in PLA/starch composite systems where increasing starch content enhanced toughness but reduced strength.[26] Furthermore, the addition of glycerol as a plasticizer

increases polymer chain mobility and promotes higher elongation and flexibility in biodegradable films. Previous investigations on PLA/starch composites also confirmed that starch addition improves biodegradability and modifies the mechanical performance of the films depending on the composition of the polymer blend. Furthermore, the addition of glycerol as a plasticizer increases polymer chain mobility and promotes higher elongation and flexibility in biodegradable films [27].

Table 4. Thermal Degradation of PLA/TS/G Biofilm

Sample	Min. degradation (°C)	Max.Degradation Temp (°C)	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. Conclusions

The properties of the PLA/TS blend improved through 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.

Abbreviations

TGA Thermogravimetric Analysis
PLA Polylactic acid
FTIR Fourier-Transform Infrared spectroscopy
PBAT Polybutylene adipate co terephthalate
PCL Polycaprolactone
PEG Polyethene glycol
PHB Polyhydroxybutyrate
TS Tapioca starch

Conflicts of interest: The authors declare that they have no conflicts of interest.

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