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PRODUCTION OF BIODEGRADABLE PLASTIC FILM FROM POTATO AND SAGO STARCH

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ABSTRACT

The increasing focus on biodegradable plastic is attributed to their non-hazardous characteristics, distinguishing them from synthetic plastics. The present work focuses on the development of potato and sago starch-based bioplastic film with sorbitol and glycerol as plasticizer glycerol as plasticizers, along with the incorporation of acetic acid and sodium bicarbonate as an antimicrobial agent. In this study, a method was employed involving the mixing of 10 grams each of potato starch and sago starch with varying concentrations of plasticizers (10%, 20%, 30% sorbitol and 20%, 30%, 40% glycerol), along with 10%, 20%, 30% acetic acid and 5%, 10%, 15% sodium bicarbonate relative to the total starch amount. The mixture was then heated with stirring. The resulting products underwent characterization through Fourier Transform Infrared Spectroscopy (FTIR), chemical resistance testing, swelling testing, water solubility testing, solubility testing in chemical solvents, biodegradability testing, and mechanical analysis. The outcomes revealed that the highest tensile strength, reaching 5.81 MPa, was achieved with a combination of 6 mL acetic acid, 6g sorbitol, and 8 mL glycerol. FTIR spectroscopy confirmed the formation of starch bioplastics by identifying functional groups O–H, C–H, and C=O. Potato and sago starch-based bioplastics were found to degrade effectively when buried in soil. Overall, this study underscores the significant potential of the potato and sago starch mixture in bioplastic production, highlighting its favorable miscibility and compatibility.

Keywords: Biodegradability; Bioplastic Film; Potato Starch; Sago Starch; Characterization

INTRODUCTION

In today's consumer-driven society, there is a substantial demand for plastic, which is widely used in the production of various goods. The majority of the plastic in circulation originates from non-renewable hydrocarbons, posing environmental challenges due to its non-renewable nature and the significant solid waste it generates **(Moshood *et al.*, 2022).** Recent advancements have facilitated the creation of biodegradable plastics using natural and renewable resources, capable of decomposing under specific temperature and humidity conditions **(Samir 2022; Ahsan *et al.*, 2023)**. By reducing reliance on fossil fuels, biodegradable plastics contribute to lessening their environmental impact. Consequently, the adoption of biodegradable plastics has the potential to address waste disposal issues. Furthermore, it is economically viable as they are crafted from inexpensive and readily available materials such as starch and cellulose (natural polymers), which can be easily extracted or stored **(Kaewphan & Gheewala, 2013; Orenia *et al.*, 2018; Kumar *et al.*, 2019).**

The production of biodegradable plastics involves utilizing polymers sourced from biological origins, such as vegetable oils, potatoes, potato peels, corn, sugarcane, wheat, rice, and seaweed. Seaweed-based bioplastics, comprising various proportions of agar-agar, seaweed, starch, or cellulose, along with plasticizers, have been studied for their impact on physical properties and bioplastic characteristics. These materials, derived from renewable biomass, possess the ability to undergo organic degradation in nature through the action of microorganisms **(Coppola *et al.*, 2021; Kumar *et al.*, 2020).** While these biodegradable materials remain stable during production and usage, they can be converted into biomass energy, carbon dioxide, and water once discarded within a suitable timeframe. Biodegradable plastics refer to plastic materials that can undergo complete degradation in landfills, composting facilities, or sewage treatment plants, biological activity of natural microorganisms.

Starch is hailed as a promising material for sustainable development due to its complete biodegradability, affordability, and renewable potential. Authentic biodegradable plastics exhibit no toxic, visible, or detectable residue post-decomposition **(Moshood *et al.*, 2022).** Presently, bioplastics constitute approximately one percent of the nearly 300 million tons of plastic produced annually. However, the market is expanding due to the rising demand for biopolymers across various applications and products. The total bioplastic production in 2022 reached around 2.44 million tons **(Marichelvam, 2019).** Approximately 50% of commercially used bioplastics are starch-based, known for their ease of production and widespread application in packaging. The inherent properties of starch are conducive to producing packaged goods, and the addition of glycerin as a thickener enhances these properties. The desired characteristics of bioplastics are attained by adjusting the quantity of additives. In commercial applications, resin-based plastics are frequently blended with natural polyester. Another ancillary challenge that can be mitigated involves minimizing agricultural waste by utilizing it for bioplastic production. This is because a substantial amount of food and agricultural waste, including spoiled fruits and vegetables, is discarded and collected annually **(Thomas *et al.*, 2023).** This waste material is rich in starch and cellulose, the primary components of plants globally. The hydroxyl bonds of cellulose, when in contact with water, create a flexible substance suitable as a substitute for plastic. Moreover, this recycled material can be adapted to achieve fire and water resistance, making it sturdy enough for application as a building material. Starch encompasses two polysaccharides, amylase and amylopectin, both holding potential for bioplastic production **(Nigam *et al.*, 2021).** Furthermore, given its short-term decomposition potential, there is a pressing need to develop plastics that readily break down, thus contributing to environmental protection.

Cassava serves as a primary source of starch, offering biodegradability, non-toxicity, cost-effectiveness, renewability, and a carbon-based composition. In recent years, 66% of global biodegradable plastic production has utilized starch as a recyclable raw material **(Moshood *et al.*, 2022).** Potato starch is another example of a starch-derived product employed as a raw material for manufacturing biodegradable plastics. Corn, recognized for its easy availability, nutritional richness, and simple nutrient composition, is also utilized as an optional material in bioplastic production. Banana leaves, readily accessible and cost-effective, emerge as a viable raw material for efficient bioprocesses in bioplastic production **(Jeenusha & Amritkumar, 2020).** Additionally, the agricultural waste of wheat grass can be harnessed by bacteria like *Bacillus licheniformis* and *Bacillus megaterium* to produce polyhydroxybutyrate (PHB), a material utilized in the production of biodegradable plastics **(Krishnan *et al.*, 2021).**

Sago, derived from the Metroxylon sp plant, is a rich source of starch cultivated and harvested in various regions of India. Comprising 88 percent carbohydrates, 0.5 percent protein, and minimal amounts of fat, with almost no B vitamins, sago is predominantly starch. Moreover, a bioplastic developed from sago starch exhibits thermoplastic properties and superior thermal and mechanical characteristics **(Ahmad *et al.*, 2011).** Being a natural raw material, sago starch is non-harmful, as it reduces the chemical components in the mixture by substituting it with a chemical-free sago starch that exhibits no harmful reactions when combined with other biodegradable components. The amylose content in sago starch facilitates easy bonding with other chemical compounds, and its gelatinization occurs at 70°C when heated.

Tabi and Kovacs (2007) asserted that bioplastic incorporates polymers derived from renewable resources, such as thermoplastic starch (TPS) or poly-lactic-acid (PLA). In contrast, **Janssen and Moscicki (2006)** highlighted that TPS, composed of starch and a plasticizer, has emerged as an intriguing and economically viable alternative to traditional plastics. **Dai *et al.* (2009)** contended that various plasticizers have been examined to enhance the processing properties and product performance of TPS. According to most TPS literature, commonly used plasticizers for promoting starch plasticization include glycerol, glycol, xylitol, sorbitol, sugars, and amides like urea, formamide, and ethylene bisformamide. **Yang *et al.* (2006)** discovered that plasticizers play a crucial role during the thermoplastic process. They form hydrogen bonds with starch, substituting the strong interaction between intra- and intermolecular hydrogen bonds in starch, leading to the display of starch plasticization.

Plasticizers, a crucial category of low-molecular-weight, non-volatile compounds, find extensive use in polymer industries as additives. These compounds play a role in decreasing deformation tension, hardness, density, viscosity, and electrostatic charge within a polymer, while simultaneously enhancing polymer chain flexibility, resistance to fracture, and dielectric constant. Another property influenced by plasticizers is the resistance to biological degradation. Glycerol stands out as the most commonly utilized type of plasticizer. However, before producing such materials, it is essential to modify the structure of native starch adequately. This modification is necessary because starch, being a multi-hydroxyl polymer, contains numerous intermolecular and intramolecular hydrogen bonds, rendering native starch unsuitable as a true thermoplastic. Nevertheless, in the presence of a plasticizer at high temperatures and under shear, native starch readily undergoes melting and flowing. The incorporation of plasticizers into edible films becomes crucial to counteract the brittleness caused by extensive intermolecular forces **(Vieira 2011).**

Utilization of organic waste like sago and potato to manufacture starch-based bioplastics holds the potential to alleviate the environmental harm associated with conventional plastics. The objective of this study was to obtain starch from potato and sago starch, and subsequently develop a biodegradable plastics film incorporating various concentrations of sorbitol and glycerol as plasticizers, acetic acid along with the incorporation sodium bicarbonate as an antimicrobial agent. The characterization of films involved assessing its physicochemical, mechanical, and biological properties.

MATERIALS AND METHODS

Materials

The materials employed in this study included potato and sago starch, purchase from a market in Mohali. Glycerol, acetic acid, sorbitol, ammonia, acetic acid, acetone, and sulfuric acid and sodium bicarbonate were purchased from Merck private limited, India. Sodium hydroxide, hydrochloric acid, sodium chloride, and ethanol were utilized for acid testing and were procured from HiMedia laboratories private limited, India. The equipment utilized in this research comprised an analytical balance, FTIR, hot air oven, autoclave, glass beaker, grinder, hotplate, magnetic stirrer, vernier caliper, acrylic plates, digital thickness gauge, petri dish, oven, ultrasonic bath, Whatman filter paper, rotary evaporator, and spectrophotometer. Millipore water was used throughout the experiments.

Extraction of Starch

Five hundred grams of potatoes were meticulously washed, peeled, and diced into small cube-shaped pieces. A quarter of the volume of distilled water was introduced to the chopped potatoes, and the blender was operated at high speed for duration of two minutes. The supernatant was then carefully discarded, resulting in the extraction of white potato starch. Weighing two hundred grams of bulk sago, the sago was blended with 400 mL of water using a blender until achieving a smooth consistency. The resulting blend was then strained through muslin cloth to separate the fine paste of sago starch from the solid mass, collecting it in a steel pan (Figure 1). Subsequently, the collected sago starch paste was subjected to heating at 100°C in a hot air oven for 24 hours to eliminate water content. The final product obtained was dry sago starch.

Preparation of Bioplastics Film

In the formulation of bioplastic using potato and sago starch, acetic acid acts as a modifier, while sorbitol and glycerol serve as plasticizers, chosen for their favorable mechanical properties and water solubility. To create the solution, potato and sago starch were introduced to 100 mL of distilled water in a glass beaker. Subsequently, acetic acid, sorbitol, and glycerol were added to the potato and sago starch solution in varying proportions of 10%, 20%, and 30%, and 20%, 30%, and 40%, respectively (wt. of substrate). The mixture was then heated on a magnetic stirrer hot plate. Initially, the temperature of the magnetic stirrer remained constant, starting at 40°C, while the rpm was set to 150. Gradually, both the temperature and stirring revolution increased from 40°C to 90°C and 150 to 250 rpm. Upon reaching a thicker consistency in the mixture, 2, 4, and 6 grams of sodium bicarbonate ($NaHCO_3$), dissolved in 10 mL of water, were introduced to the solution. The mixing process persisted for an additional 10 minutes, maintaining a constant temperature of 90°C. The application of high temperature was essential to confer thermal stability to the biodegradable plastic. Subsequently, the thickened mixture was poured onto glass plates to form a film. The glass plate, bearing the film, was then placed in an oven set at 70°C for 5 hours to facilitate hardening and drying.

Figure 1 Schematic representation of the development of sago and potato starch-based bioplastic

Fourier Transform Infrared Spectroscopy Analysis (FTIR)

Fourier transform infrared spectroscopy (FTIR) emerges as the most dependable and economical method for discerning the key elements of a plastic material. One of FTIR's notable strengths lies in its ability to identify degradation-oxidation within the plastic. Spectra in the frequency range of 4000 to 600 cm^{-1} were acquired with a resolution of 4 cm^{-1}. The FTIR spectrum software facilitated the data analysis of each film.

Mechanical Properties

The tensile strength of bioplastic films was assessed using the Testometric Machine M350 10CT. Dumbbell-shaped film samples, conforming to the ASTM D882 standard for tensile testing (Marichelvam et al., 2023), were employed. Each film underwent three replications during the testing process. The tensile strength (in MPa) was recorded during the stretching phase.

Chemical Resistance Test

Chemical resistance testing is a quick and easy approach to determine whether a protective coating can withstand chemical deterioration before it is put into operation. The bioplastic films were precisely cut into 1 cm x 1 cm dimensions and subsequently dried at 60°C for approximately 15 minutes in a hot air oven. Following this, the bioplastic film samples were immersed in various solvents (0.1 N NaOH, 0.1 N HCl, NaCl solution, and 50% Ethanol) for a duration of 48 hours, and their resistance was evaluated by measuring changes in appearance **(Krishnamurthy and Amritkumar 2019).**

Swelling Test

Swelling tests are commonly employed to assess whether the produced material maintains its original characteristics following its formation in the preparation process. The swelling percentage, reflected in the thickness gain of bioplastics, serves as an indicator of the extent to which water permeates the bioplastic material. All bioplastic films were precisely cut into 1 cm x 1 cm dimensions and subjected to drying at 60°C for approximately 15 minutes in a hot air oven. The initial thickness of the dried film was measured using a vernier caliper. Subsequently, the bioplastic film was immersed in 20 mL of various solvents (water, methanol, chloroform) in a petri plate and left for 24 hours. The thickness of the films after immersion was measured using a vernier caliper.

$$\text{Swelling (\%)} = \frac{(\mathrm{Th}_1 - \mathrm{Th}_\circ)}{\mathrm{Th}_\circ} \times 100$$

Where, Th_o and Th_1 is thickness of sample before and after immersion

Water Solubility Test

One of the critical indicators for assessing the extent of bioplastics deterioration when exposed to water or water vapor is their resilience to water absorption and solubility. All bioplastic film samples were precision-cut into 1 cm x 1 cm dimensions, dried at 70°C for approximately 1 hour in a hot air oven, and the mass of the dry film was meticulously weighed and recorded. The dried sample was then immersed in 100 mL of distilled water in a 250 mL beaker, placed on a magnetic stirrer, and stirred at 150 rpm for 10 hours. Following this period, the distilled water was discarded, and the sample was collected to dry at 80°C for about 15 minutes in a hot air oven. The final readings were obtained by weighing the sample again.

$$\text{Solubility } (\%) = \frac{W_{\circ} - W_1}{W_{\circ}} \times 100$$

Where, W_o is initial dry weight of bioplastic and W_1 is final dry weight of bioplastic after immersing in water

Solubility Test in Chemical Solvents

A solubility test is a method employed to determine a substance's ability to dissolve in a solvent, typically a liquid. The bioplastic samples were precisely cut into 1 cm x 1 cm dimensions and subjected to drying at 60°C for approximately 1 hour in a hot air oven. After drying, the samples were weighed using a weighing machine. Subsequently, the dried samples were immersed in various solvents (ammonia, acetic acid, acetone, sulfuric acid) for 24 hours in separate beakers. Following the 24-hour period, the solvent was discarded, and the samples were collected for drying at 80°C for about 15 minutes in a hot air oven. After the samples were removed from the oven, their final weights were recorded.

$$\text{Solubility } (\%) = \frac{(W_0 - W_1)}{W_0} X100$$

Where, W_o is initial dry weight of bioplastic and W_1 is final dry weight of bioplastic after immersing in solvents

Soil Burial Biodegradation

The bioplastic films were sliced into strips measuring 1 cm x 1 cm in dimensions. These cut samples were then buried in soil, with some taken from a botanical garden and others from the roadside, to evaluate their natural degradation in a landfill setting under atmospheric conditions. After a 15-day period, the samples were retrieved from the soil.

RESULTS AND DISCUSSION

Biodegradability of bioplastics films

All the soil buried bioplastic samples were taken from the soil after 15 days. The first sample disintegrated into pieces, with 60% of the sample undergoing degradation. The sample S2 experienced an 80% degradation, while the sample S3 completely degraded. The biodegradability of bioplastic films appears to escalate with higher concentrations of acetic acid, sorbitol, and glycerol. This phenomenon may be attributed to the presence of O-H and C-O functional groups within the films. This outcome is evident in research conducted by Azevedo, Almeida, and Santos (2017), which explored various natural polymers, including potato starch. Wahyuningtiyas et al. (2017) produced bioplastic using cassava flour with varying proportions of glycerol and assessed its biodegradation through soil burial. The bioplastic's mass decreased within a week, achieving complete degradation within 12 days. This study highlights the rapid degradation capacity of starch-based bioplastics.

Fourier Transform Infrared Spectroscopy Analysis of Bioplastics Films

FTIR results of potato and sago starch-based bioplastics film samples are shown in Figure 2. In sample S1, the initial broad peak at 3292.27 cm−1 signifies the presence of carboxylic acid O–H stretch, confirming the interaction of various –OH groups in the starch and acetic acid blends. Additionally, the peak at 2922.97 cm−1 in both films indicates a robust C–H stretch of glycerol with starch. Other notable peaks at 1150.07 cm−1 and 996.21 cm−1 suggest the existence of C–H wag with halo compounds and (R-CH=CH2), respectively (Krishnamurthy and Amritkumar 2019). Peaks within the 900 to 600 range indicate the presence of alkynes, nitro, and alkyl halide groups.

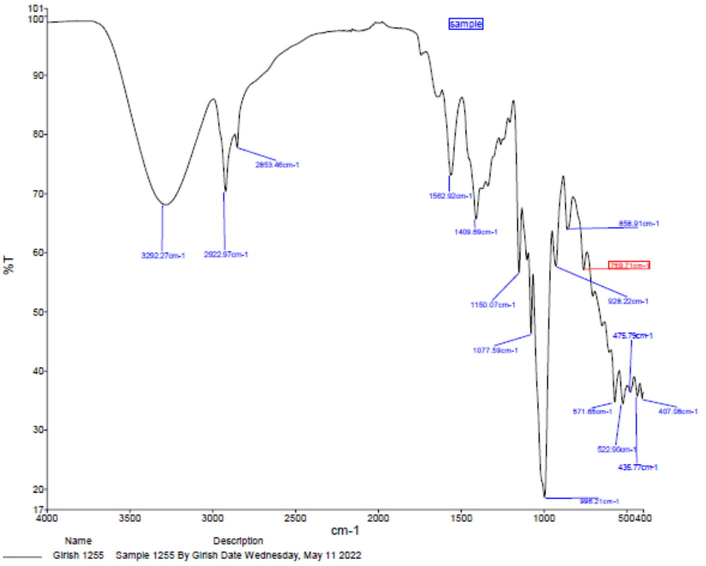

Figure 2 FTIR spectra of bioplastic

Water Solubility

The solubility of the films serves as an indicator of the presence of hydrophilic compounds, contributing to their characterization as effective biodegradable films. A desirable bioplastic is characterized by low water solubility. In the case of the synthesized bioplastic films, sample S1 exhibited a water solubility index of 25.6%, sample 2 had 29.4%, and sample S3 showed 33.3%. The notably superior water solubility of sample S3 compared to samples S1 and S2 is attributed to the higher proportions of glycerol, acetic acid, and sorbitol in its composition. Potato starch and glycerin films demonstrated solubility values of approximately 31.7% (Wang et al., 2007).

Chemical Resistance

The three bioplastic films underwent exposure to various chemical solvents, and their impact on the films was assessed based on the parameters outlined in Table 1. Notably, the films absorbed the solvent and exhibited softening in an alkaline environment, likely due to the presence of acid within the film, engaging in a reaction and subsequent neutralization. The bioplastic film displayed softening in all solvents, with complete dissolution observed in the case of the strong base NaOH. These outcomes align with findings by Jack et al. (2017), who observed similar behaviour in their bioplastic film when exposed to acid and alkali. Ethanol induced minimal swelling, attributed to the reduction of amylopectin molecules to lower molecular weights during acid-alcohol hydrolysis, thereby diminishing swelling power and making the film less susceptible to breakage.

Table 1 Chemical resistance test of bioplastic in different solvents

Chemical name	Properties
0.1N HCl	a, c, d, e
0.1N NaOH	b
NaCl	c, d, f
50% Ethanol	c, g

Where, a is no change in dimension, b is fully dissolve, c is swollen, d is softened, e is discoloured (turned whitish), f is turned translucent, and g is no change in colour

Solubility Test in Chemical Solvents

The results of the study indicated that the substance displayed water insolubility, rendering it well-suited for application as a bioplastic material. Furthermore, it exhibited insolubility in acetone (a polar solvent), ethyl alcohol (a non-polar solvent), acetic acid (a polar solvent), ammonia (a polar solvent), and H_2SO_4 (completely soluble in sulfuric acid, a strongly acidic solvent). Starch, as per O'Neil (2006), is generally insoluble in alcohol at room temperature. However, incorporating a plasticizer into starch for bioplastic production can enhance its solubility in alcohol. In this context, sorbitol and glycerol served as plasticizers. Sorbitol is nearly insoluble in alcohol, while glycerol demonstrates slight solubility.

A substance cannot be categorized as bioplastic if it dissolves in water and other solvents; thus, solubility plays a crucial role in selecting a sustainable biomaterial for bioplastic production. The results of the solubility test presented in Table 2 &

3 demonstrate that the material is largely insoluble in organic solvents, enhancing the cost-effectiveness of bioplastic manufacturing.

Table 2 Solubility of bioplastic in different chemical solvents

Solvents	Solubility test		
	Insoluble	**Partially soluble**	**Completely soluble**
Ammonia	-	+	-
Acetic acid	+	-	-
Acetone	-	+	-
Sulphuric acid	-	-	+
Ethyl alcohol	-	+	-

*where, + = positive, - = negative

Table 3 Thickness and solubility percentage of bioplastic films

Solvent	W_0 (g)	W_1(g)	Solubility (%)
Ammonia	0.05	0.04	20
Acetic acid	0.06	0.05	16.6
Acetone	0.05	0.03	40
Sulphuric acid	0.06	-	-
Ethyl alcohol	0.05	0.04	20

Swelling

The absorption of solvents in starch-based bioplastics plays a pivotal role in shaping their properties and characteristics. Among the solvents tested, the bioplastic exhibited higher water absorption compared to substances like methanol and chloroform. The melting process disrupted the crystalline structure of starch, influenced by both heat and excess water. This disruption led to the separation of amylose and branched chains of amylopectin, resulting in the formation of hydrogen bonds. Additionally, some exposed hydroxyl groups of amylose and amylopectin, not involved in cross-linking with plasticizers, reacted with water upon exposure, causing the film to undergo swelling. As per Sasaki and Matsuki's findings, the swelling volume of starch is influenced by the concentration of amylose and the structure of amylopectin. The commencement of swelling and gelatinization has been observed to be dictated by the molecular weight, form of the amylopectin molecule, and the crystallites within it. The interaction between glycerol and starch (AV) is noted for its strength, limiting the film's water uptake and resulting in minimal swelling. Table 4 provides details on the thickness and swelling percentage of the three films.

A reduction in swelling percentage was observed with methanol due to acid-alcohol hydrolysis, causing the degradation of amylopectin molecules into smaller molecular weight counterparts. This degradation resulted in a diminished swelling power, as noted by Tester and Morrison (1990). Additionally, chloroform reported the lowest swelling percentage.

Table 4 Swelling percentage of bioplastic in different solvents

Component	Th_0	Th_1	Swelling%
Water	0.4	1.0	150%
Methanol	0.4	0.6	50%
Chloroform	0.4	0.5	25%

Tensile Test

The biodegradable plastic films comprised of 6 mL acetic acid, 6g sorbitol, and 8 mL glycerol in sample S3, exhibits a tensile strength of 5.81 MPa. The tensile strength of the biodegradable plastic increased from 2 mL to 6 mL as the concentration of acetic acid was increased. This rise in tensile strength is attributed to the augmented acetic acid concentration, leading to an increased production of substrate constituent polymers and enhanced attraction between starch and acetic acid molecules. With increasing pressure, the potato and sago starch-based films with sorbitol/glycerol plasticizers exhibit a rise in elongation at break. This indicates that sorbitol plasticizers yield higher elongation at break, whereas glycerol plasticizers result in lower elongation at break (Hazrol et al., 2021).

CONCLUSION

In this study, bioplastic film was successfully developed from mixture of potato and sago starch. The aim is to attain an optimal formulation for the development of biodegradable plastics. The FTIR analysis revealed absorption bands of carboxylic acid O–H and C–H at 3292.27 cm^{-1} and 2922.97 cm^{-1}, confirming interactions among different –OH groups in the starch and acetic acid blends, as well as the C–H stretch of glycerol with starch. The sample mixture containing 6 mL of acetic acid, 6g of sorbitol, and 8 mL of glycerol achieved the highest tensile strength value at 5.81 MPa. Notably, the results from this study indicate that sample S3 exhibits superior values in terms of biodegradation, solubility in chemical solvents and water, chemical resistance, and swelling testing compared to the other samples. The soil burial biodegradation test demonstrates that potato and sago starch-based bioplastics exhibit high biodegradability. The superior biodegradability values observed in the third sample suggest its suitability for packaging applications and easy disposal. The successful preparation of potato and sago starch-based bioplastics with enhanced mechanical and chemical properties represents a significant accomplishment. Biodegradable plastic films provide a sustainable solution to replace traditional petroleum-based plastics, finding applications in various industries, including food packaging, consumer goods, agriculture, medicine, and healthcare. These bioplastics can serve as a viable alternative to conventional plastics due to their notable biodegradable characteristics, favorable biodegradability, mechanical properties, and cost-effectiveness, making them easy to modify.

Conflict of Interest: The authors declare that they have no conflict of interest.

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