Research Article

The Effect Of Different Plasticizers On The Physicochemical Properties Of Starch Film Incorporated With Pomegranate (*Punica Granatum*) Peel Powder

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ABSTRACT

This research studies the effect of different plasticizers on the physicochemical properties of starch film incorporated with pomegranate peel powder (PGP). PGP was selected due to its biodegradable property and potential to enhance the physicochemical properties of biopolymer films, while providing a low-cost use for an agricultural by-product. The objective was to evaluate the effect of various plasticizers on the physical, mechanical, and barrier properties of starch-based films. Film samples were prepared using five plasticizer ratios (4:0, 3:1, 2:2, 1:3, and 0:4 of glycerol: sorbitol) and a fixed concentration of PGP powder. The films were produced using the solution casting method. Evaluations included thickness, opacity, water solubility, colour, mechanical properties, biodegradability, and oil permeability. The film thickness increased progressively with higher sorbitol concentrations. The increasing concentration of sorbitol also increased the transparency and water solubility. The different ratios of glycerol and sorbitol did not have a significant effect on the Hunter L*, a*, and b* values of the films due to the colourless nature of the plasticizers. Mechanical testing revealed that plasticizer ratio did not significantly affect tensile strength or elongation at break but affected Young's modulus. All films degraded by day 9 as well as exhibited excellent oil barrier properties. Overall, the combination of glycerol and sorbitol provides a good balance of mechanical and barrier properties, making it a promising sustainable alternative for biodegradable packaging and for edible films.

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1. Introduction

Plastic films are extensively used in the food industry as packaging to protect food from contamination, preserve food quality, provide physical protection, and facilitate transportation (Wani *et al.,* 2014). Approximately half of global plastic production has occurred within the last 15 years (Allen *et al.*, 2020), and this figure may rise two to three times by 2050 (World Economic Forum, 2016). Despite the various benefits of plastic packaging, its widespread use has a negative environmental impact because polymers take a very long time to degrade (Lim *et al.*, 2020).

This persistence of plastic waste in the environment has driven increasing exploration of biodegradable alternatives to conventional films. Biodegradable films are plastics that break down through the activity of natural microorganisms, such as algae, bacteria, and fungi (Nizamuddin *et al.*, 2024). They can be produced from renewable resources, including starch, protein, chitosan, gelatin, or pectin (Ali *et al.*, 2019). Among these, starch is particularly attractive due to its low cost, non-toxicity, and abundance (Chhatariya *et al.*, 2022), as well as its desirable characteristics of being translucent, colourless, and tasteless (Rahmadi *et al.*, 2023). Furthermore, starches from different botanical sources exhibit varying amylose contents, which influence film properties by enhancing gelation behavior and thereby improving film strength (Zhong *et al.,* 2021). For example, corn starch contains approximately 28–33% amylose, which is associated with stronger film formation, a level that closely compares with wheat starch (30–32%) and exceeds that of potato (18–20%) and cassava starch (16–19%) (Nordin *et al.*, 2020). Owing to its favourable characteristics, corn starch has been extensively used as a matrix in the development of biodegradable film.

However, starch-based films require the use of plasticizers, which are small molecules that disperse and embed within polymer chains to improve film properties (García *et al.*, 2000). Without plasticizers the films typically become rigid, fragile, and brittle (Hazrol *et al.,* 2021). Among the various plasticizers, glycerol and sorbitol are most commonly used in biofilm formulations (Lim *et al.*, 2020). Films plasticized with glycerol have shown improved flexibility Nordin *et al.*, (2020) and compatibility with amylose Vieira *et al.*, (2011), but with reduce tensile strength Sandeep *et al.*, (2023), while sorbitol has shown higher intermolecular forces but lower ability to interact with water (Sanyang *et al.*, 2015).

In addition to plasticizers, reinforcement materials have been shown to enhance the properties of biopolymer-based films (Chen *et al.*, 2019). Such reinforcements can include natural substances, such as pomegranate peel powder. Pomegranate peel offers several advantages, including its biodegradability, abundant availability, low cost as a by-product, non-toxicity, and high strength-to-weight ratio (Xiang *et al.,* 2022). Moreover, pomegranate peel is abundant in polyphenols, including phenolic acids such as ellagic acid, punicalagin, punicalin, and gallic acid, as well as flavonoids such as epicatechin and rutin, which have been reported to contribute antioxidant and antimicrobial activities (Salim *et al.*, 2023; Ciupei *et al.*, 2024), making them potentially valuable in edible films. Previous studies have demonstrated that pomegranate peel can strengthen starch-based films mechanically (Ali *et al.*, 2019; Zhang *et al.*, 2024), polyvinyl alcohol films (Abinaya *et al.*, 2024), and PLA/starch composite films (Yang *et al.*, 2024). It is naturally rich in cellulose, a key structural fibre, and together with other components such as hemicellulose and lignin, it provides strength and stability, making the peel suitable for use in composite materials and biodegradable films (Siddiqui *et al.*, 2024). However, the effect of plasticizer selection on the performance of PPP composites remains unexplored. Hence, this study addresses this gap by specifically evaluating the influence of two distinct plasticizers on the physicochemical and permeability properties of corn starch films reinforced with pomegranate peel powder (PPP).

2. Materials and Methods

2.1 Materials

Pomegranates, Sunflower seed oil, black soil were purchased from a local supermarket in Selangor, Malaysia. Cornstarch was purchased from Thye Huat Chan Sdn Bhd (Selangor, Malaysia), Food-grade Sorbitol powder was purchased from Bischem Technology Sdn Bhd (Selangor, Malaysia) and Glycerol (≥ 99%) from Bio3 Sdn Bhd (Selangor, Malaysia).

2.2 Pomegranate peel powder preparation

The PGP powder was processed as described in the method laid out by Zhang *et al.* (2024). Fresh pomegranates were washed, peeled, cut into fine pieces and then oven-dried (Venticell, MMM, Munchen, Germany) at 50°C for 6h. The dried peels were ground into powder and sieved using a 200-mesh sieve.

2.3 Film preparation

The films were prepared according to the method of Leites *et al.* (2021), with some modifications. Starch suspension was prepared with 8% (w/v based on distilled water) of corn starch and glycerol and sorbitol (40% w/w based on corn starch) using different ratios (4:0, 3:1, 2:2, 1:3, 0:4) and was kept in a water bath (Memmert, Federal Republic of Germany) at 80°C under constant mechanical stirring for 30 minutes. The plasticizer concentration used was based on a preliminary study, in which the likelihood of cracks forming was lower at 40% plasticizer concentration, and the films were easily removed from the petri dish. Starch films incorporated with PGP powder (10% w/w based on corn starch), based on a preliminary study, were added to the corn starch suspension. Later, 10 ml of the suspension was poured into Petri dishes, followed by oven-drying at 30°C for 24 hours. The films were kept in a desiccator at approximately 25 ± 3 °C, with 50% relative humidity, for 24 hours before further analysis.

2.4 Thickness

The film's thickness was determined with callipers at five different locations and the average were calculated (Chhatariya *et al.*, 2022).

2.5 Film opacity

The film opacity was measured using a UV-Visible Spectrophotometer (Hach dr6000, Malaysia), where light absorption is measured at a wavelength of 600 nm. The film was cut into 1 cm x 4 cm strips and placed in quartz cuvettes for transmittance measurements. A clean quartz cuvette was used as a reference blank (Lim *et al.*, 2020). Analysis was triplicated.

2.6 Tensile strength

The mechanical properties of the films were tested following the method described by Esfahani *et al.* (2022), with modifications, using a tensile testing machine (Testometric, England, United Kingdom). The sample dimensions were 70 mm x 20 mm, the crosshead speed used was 30 mm/min the gauge length used was 50 mm and the load cell capacity used was 10 kN. The elongation at break, Young's modulus and tensile strength were measured. Analysis was triplicated.

2.7 Water solubility

Film solubility was measured using the method described by Moghadam *et al.* (2020), with adjustments. 1 cm x 4 cm pieces of the films were dried in an oven (Venticell, MMM, Munchen, Germany) at 110°C for 24h, and then the initial weight was measured. After drying, the samples were submerged in 40 mL of distilled water and left for 24 h at room temperature. The remaining undissolved film was dried at 110°C for 24h to determine the final dry weight. The analysis triplicated.

Water solubility of the films was calculated using the equation below:

$$WS\ (\%) = \frac{W_i - W_f}{W_i} \times 100 \quad (1)$$

Where *Wi* denotes the initial weight and *Wf* represents the final weight of the films

2.8 Colour measurement

Surface colour measurements were performed using a colourimeter (Konica Minolta, Tokyo, Japan). The samples were positioned on a white standard plate for the measurement of Hunter coluor values (L*, a*, and b*). Five measurements were taken at different locations (Li *et al.*, 2015). The analysis was triplicated.

2.9 Oil permeability

The samples were cut into 5 cm × 5 cm squares and secured with rubber bands into the openings of test tubes containing 5 ml of edible oil; sunflower seed oil was used. These tubes were then inverted and placed onto filter paper for 48 hours. The filter paper was weighed both prior to and following the test. (Wu *et al.*, 2024). Analysis was triplicated.

$$PO = \frac{W \times FT}{S \times T} \quad (2)$$

Where PO is the oil permeability coefficient, W is the change in mass of filter paper, FT is film thickness, S is the surface area of the film, and T is the duration.

2.10 Biodegradability

Film biodegradability was determined using the soil burial method. The films were cut into 1 x 4 cm pieces. Later, the film pieces were placed on a wire gauze. The film was buried at a depth of 2 cm in a plastic container containing black soil and kept in ambient condition. The soil was watered 10 ml daily to maintain moisture throughout the test. The films were observed and photographed at 3-day intervals over 15 days to monitor degradation (Susmitha *et al.*, 2021).

2.11 Statistical analysis

Data were statistically analyzed using Minitab Statistical Software, version 22. The data is presented as the mean ± standard deviation and was analyzed using one-way ANOVA, followed by Tukey's post-hoc test. The results are considered statistically significant at $p \leq 0.05$.

3. **Results and Discussion**

Table 4.1 shows the thickness of starch film incorporated with PGP powder where there was no significant difference ($P>0.05$) when the plasticizer content was modified. The film formed with the ratio of 0:4 (glycerol: sorbitol) exhibited film thickness of 0.15 mm, while films produced using the ratios of 4:0 and 2:2 exhibited thickness of 0.10 mm. Hence, films plasticized with a higher concentration of sorbitol in proportion produce slightly thicker films, though the difference is statistically insignificant.

This trend could be due to sorbitol has higher molecular weight. Higher molecular weight compounds typically possess larger molecular structures, which results in the intermolecular gap within the film matrix increasing (Farhan & Hani, 2017). Furthermore, sorbitol also tends to crystallize within the films, affecting its mechanical properties, which could lead to increased thickness (Ma *et al.*, 2023). This finding is similar to Hazrol *et al.* (2020), in which corn starch films prepared with a 45% glycerol concentration had lower thickness than those prepared with 45% sorbitol. A study by Müller *et al.* (2008) with cassava starch films also found that films plasticized with sorbitol had greater thickness when compared to glycerol films.

Table 4.1 shows the transmittance at 600nm of starch film incorporated with PGP. Low transmittance through the film could increase product shelf life, while higher transmittance could give the customer a glimpse of the product (Simona *et al.*, 2021). The findings indicate that there is no significant difference ($p > 0.05$) in light transmittance when different ratios of glycerol and sorbitol are used.

Table 4.1. Thickness, transmittance and Hunter _L*, a*_ and _b*_ values of starch film incorporated with PGP powder with different ratios of glycerol and sorbitol

Glycerol:Sorbitol Ratio	**Thickness**	**Transmittance**	**_L*_**	**_a*_**	**_b*_**
4:0	0.10 ± 0.02^{a}	53.13 ± 3.68^{a}	84.90 ± 1.39^{a}	0.96 ± 0.86^{a}	28.16 ± 1.31^{a}
3:1	0.12 ± 0.03^{a}	52.51 ± 2.01^{a}	85.49 ± 0.36^{a}	-0.14 ± 0.09^{b}	29.25 ± 0.89^{a}
2:2	0.10 ± 0.01^{a}	54.02 ± 3.34^{a}	85.68 ± 0.58^{a}	0.01 ± 0.15^{ab}	28.40 ± 1.29^{a}
1:3	0.12 ± 0.02^{a}	53.38 ± 1.12^{a}	85.09 ± 0.05^{a}	0.13 ± 0.1^{ab}	29.75 ± 0.12^{a}
0:4	0.15 ± 0.02^{a}	57.02 ± 0.73^{a}	85.58 ± 0.74^{a}	0.06 ± 0.11^{ab}	29.21 ± 1.62^{a}

Mean values in the same column with different letters are significantly different ($p<0.05$).

Colour analysis is another key parameter in packaging films, as it can enhance product visibility on retail shelves (Spence & Velasco, 2018). The colour analysis in Table 4.1 presents Hunter values, where L denotes lightness, _a_ represents the red–green axis, and b* corresponds to the yellow–blue axis. There is no statistically significant difference ($p>0.05$) between the hunter values _L*_ and _b*_, but there is a significant difference ($p<0.05$) between the a* values of the films. For L* and b* values, this may be due to glycerol and sorbitol are clear, colourless substances (Ballesteros-Mártinez _et al._, 2020). Glycerol and sorbitol are also polyols that function as plasticizers in the same way, which is by decreasing intramolecular hydrogen bonding among polymer chains (Sirbu _et al._, 2024). This leads to similar interactions with the film microstructure, which influence the film's optical properties such as colour. Consistent with this, Farhan & Hani (2017) reported no significant differences in L* and b* values between semi-refined kappa-carrageenan films plasticized with 30% glycerol and those with 30% sorbitol. Similarly, Jost _et al._ (2014) found only slight differences in L* and b* values between alginate films plasticized with the two polyols but observed greater greenness in sorbitol-plasticized films. This aligns with the present study, in which films containing sorbitol exhibited lower a* values than those containing only glycerol, indicating a shift toward greenness with increasing sorbitol content.

Table 4.2 shows the water solubility of starch films, which is crucial for determining the suitability of films for application on high-moisture or low-moisture food products. The ratio of glycerol and sorbitol significantly ($p \leq 0.05$) affected the water solubility of the films. As the ratio of sorbitol in proportion to glycerol increased, the water solubility subsequently increased. The film produced with the plasticiser ratio of 4:0 (glycerol: sorbitol) had the lowest water solubility at 21.56%, while the film produced with the ratio of 0:4 had the highest water solubility at 31.36%. This can be explained by several factors. At higher concentrations, the molecular shape of sorbitol may hinder its proper integration within the starch matrix, making it more prone to leaching or dissolution in water (Ballesteros-Mártinez _et al._, 2020). In contrast, glycerol's linear structure allows better integration with the starch matrix, thereby reducing water solubility. Additionally, Sorbitol contains more hydroxyl groups than glycerol, allowing more hydrogen bonding with starch (Reza _et al._, 2023), which may increase the water solubility of the films. Additionally, sorbitol-plasticized films exhibit higher crystallinity, contributing to a more amorphous structure that enhances water solubility (Hazrol _et al._, 2020). Studies by Abera _et al._ (2020), Ballesteros-Mártinez _et al._ (2020), and Mohammed _et al._ (2023) showed that films made from anchote, sweet potato, and wheat had higher water solubility when plasticized with sorbitol than with glycerol.

Table 4.2. Water solubility and oil permeability of starch film incorporated with PGP powder with different ratios of glycerol and sorbitol

Glycerol:Sorbitol Ratio	Water solubility	Oil permeability
4:0	21.56 ± 2.51^{c}	0.000 ± 0.000^{a}
3:1	21.92 ± 1.12^{c}	0.001 ± 0.002^{a}
2:2	24.65 ± 1.76^{bc}	0.001 ± 0.002^{a}
1:3	26.68 ± 0.94^{b}	0.001 ± 0.001^{a}
0:4	31.36 ± 0.34^{a}	0.000 ± 0.000^{a}

Mean values in the same column with different letters are significantly different ($p<0.05$).

Table 4.2 shows the oil permeability results, indicating that all films exhibited excellent oil barrier properties, with an oil permeability coefficient of 0.00. During the experiment, only minimal oil permeated through the films, with tiny splotches observed. The weight difference was too small to be detected within two decimal places, which explains the zero readings. This may be explained by the fact that starch, the predominant component of the film matrix, is naturally a hydrophilic substance Lee *et al.* (2020), which leads to films that are less permeable to nonpolar substances (Glenn *et al.*, 2006). The semicrystalline nature of starch film due to amylose and amylopectin creates a highly ordered film matrix Singh *et al.* (2022), which limits the diffusion pathway for molecules. This finding is supported by Farhan & Hani (2017), who reported that glycerol and sorbitol at 30% concentration showed low oil permeability.

Table 4.3 shows the tensile strength, Young's modulus, and elongation at break of starch film. The ratio of glycerol and sorbitol did not significantly ($p>0.05$) affect the tensile strength and elongation at break of the films; however, there is a statistically significant difference ($p<0.05$) for Young's modulus.

Table 4.3. Tensile strength, Elongation at break, and Young's modulus of starch film incorporated with PGP powder with different ratios of glycerol and sorbitol

Glycerol: Sorbitol Ratio	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
4:0	0.30 ± 0.12^{a}	25.87 ± 1.86^{b}	10.57 ± 4.94^{a}
3:1	0.39 ± 0.1^{a}	37.65 ± 8.19^{b}	10.71 ± 3.44^{a}
2:2	0.35 ± 0.06^{a}	41.0 ± 18.1^{b}	20.88 ± 4.61^{a}
1:3	0.25 ± 0.17^{a}	41.0 ± 18.1^{b}	20.88 ± 4.61^{a}
0:4	0.25 ± 0.14^{a}	135.5 ± 72.6^{a}	18.24 ± 4.32^{a}

Mean values in the same column with different letters are significantly different ($p<0.05$).

The Young's modulus (YM) of the films was gradually increased with decreasing of glycerol ratio (or increasing of sorbitol ratio) used in the film and the highest YM was achieved when glycerol was eliminated (0:4 glycerol:sorbitol ratio). This increase in YM with higher sorbitol content can be attributed to stronger hydrogen bonding between sorbitol and starch, which reduces chain mobility. Sorbitol has greater structural similarity to the glucose units in starch compared to glycerol, allowing more extensive interactions with starch molecules (Reza *et al.,* 2023). Similar findings were reported by Mohammed *et al.* (2023), where wheat starch films plasticized with sorbitol exhibited higher YM values than those plasticized with glycerol.

Meanwhile, for elongation at break, no notable differences were observed between the films. Similarly, tensile strength also exhibited insignificant difference between the samples although slight decreasing trend was observed as sorbitol concentration increased. Generally, plasticizers are known to reduce intermolecular bonds between starch molecules and replacing them with hydrogen bonds formed between the plasticizer and starch, thereby increasing chain mobility and enhancing the flexibility of the film (Sanyang *et al.*, 2015).

Table 4.4 shows the biodegradability of starch films in soil at ambient temperature. The findings show that under these conditions, there is no perceivable difference in the degradation rate of the films. By Day 3, all films showed a similar degree of degradation. Further breakdown was observed by Day 6, with films plasticized at a 2:2 glycerol-to-sorbitol ratio nearly fully degraded. By Day 9, all films had completely degraded. Both glycerol and sorbitol bring many –OH groups into the polymer matrix, providing flexible active sites that polymer-degrading enzymes target more effectively (Ehman *et al.*, 2022). The presence of –OH groups associated with flexible active sites has been directly linked to faster enzymatic binding and subsequent degradation (Ehman *et al.*, 2022).

This can be seen in the study by Mohammed *et al.* (2023), where control films without plasticizers only degraded slightly faster when compared to films plasticised with glycerol and sorbitol. The research done by Mohammed *et al.* (2023) also showed that wheat starch films plasticized with 35% glycerol degraded at the same rate as films plasticized with sorbitol at the same concentration over 15 days. Furthermore, Alqahtani *et al.* (2021) and Silva *et al.* (2020) also reported corn starch films did not have a significant difference in biodegradability rate. This shows that biodegradable films are environmentally friendly, which could degrade within 12-24 months compared to conventional plastics (Phillips, 2021).

Table 4.4. Biodegradability of starch film incorporated with PGP powder with different ratios of glycerol and sorbitol.

Glycerol: Sorbitol Ratio	**Observation**		
	Day 3	**Day 6**	**Day 9**
4:0			
3:1			
2:2			
1:3			

0:4			

4. Conclusion

In conclusion, this study examined the effects of different plasticizers on corn starch films. It was evident that film thickness, opacity, and oil barrier properties remained largely unaffected by the type or concentration of plasticizer. Furthermore, all films biodegraded at a comparable rate, showing no perceivable difference by Day 9. The choice of plasticizer did, however, significantly influence other properties. Films plasticized with sorbitol exhibited a distinct green hue, though overall film colour was unaffected by the glycerol-to-sorbitol ratios. Sorbitol also resulted in higher water solubility, while the glycerol plasticized film showed the lowest solubility, underscoring the role of plasticizer selection in tailoring film functionality. Mechanical properties were partially affected; a significant difference in Young's Modulus (YM) was observed with a high sorbitol content (0:4 ratio), while tensile strength and elongation at break remained unchanged. For future research, experiments should include a control formulation without PGP powder, using the same plasticizer ratios, to provide a direct comparison for these results.

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